Appendix B

Quality Assurance Project Plan

Appendix B

FINAL Quality Assurance Project Plan Revision 1

CONTRACT NO.: W912DQ-18-D-3008
DELIVERY ORDER NO.: W912DQ19F3048

Phase 2 Remedial Investigation Work Plan
Operable Unit 1
700 South 1600 East PCE Plume Site
Salt Lake City, Utah

U.S. Army Corps of Engineers
Kansas City District

Department of Veterans Affairs
Veterans Health Administration Salt Lake City Health Care System

December 2020

CDM Smith

Title and Approval Sheet (A1)

Quality Assurance Project Plan (QAPP) Version 1

CDM Smith Project Manager:		Date:	
	CDM Smith		
CDM Smith Technical Reviewer:		Date:	
	CDM Smith		
CDM Smith QA Reviewer:		Date:	
	CDM Smith		
USACE Project Manager:			
	USACE	Date:	
VA CERCLA Program Manager/Remedial Program Manager:		Date:	
	Department of Veteran's Affairs		
EPA Project Manager:		Date:	
	EPA Region 8		
EPA Quality Assurance Manager:		Date:	
	EPA Region 8		
UDEQ Project Manager:			
	Utah Department of Environmental Quality	Date:	

Table of Contents

Table of Contents ii

Section 1 Introduction 1

Section 2 Project Management (EPA Group A) 2

2.1 Project/Task Organization (A4) 2

2.1.1 Veterans Health Administration Program Manager 2

2.1.2 U.S. Army Corps of Engineers Project Manager 2

2.1.3 CDM Smith Project Manager 2

2.1.4 CDM Smith Quality Assurance Manager 2

2.1.5 CDM Smith Subject Matter Expert 3

2.1.6 CDM Smith Safety and Health Manager 3

2.1.7 CDM Smith Data Manager 3

2.1.8 CDM Smith Project Chemist 3

2.1.9 CDM Smith Project Engineer 3

2.1.10 Laboratory Project Managers 3

2.2 Problem Definition/Background 6

2.2.1 Background 6

2.2.2 Problem Definition 6

2.3 Project Description 6

2.3.1 Description of Work Tasks 6

2.3.2 Project Schedule 7

2.3.3 Resources and Constraints 7

2.4 Quality Objectives and Criteria (A7) 7

2.4.1 Project Quality Objectives 7

2.4.2 Data Quality Indicators and Quality Control Sample/Measurement Performance 27

2.5 Special Training/Certification (A8) 28

2.6 Documents and Records (A9) 31

2.6.1 Analytical Data Package 32

2.6.2 Electronic Data Deliverable Format 34

2.6.3 Project Information/Records Storage and Retention 34

Section 3 Data Generation and Acquisition (EPA Group B) 35

3.1 Sampling Design (Experimental Design) (B1) 35

3.2 Sampling Methods (B2) 37

3.3 Sample Handling and Custody (B3) 37

3.3.1 Sample Labeling 38

3.3.2 Packaging and Shipping 38

3.3.3 Chain of Custody 42

3.3.3.1 Laboratory Custody Procedures 42

3.3.4 Custody Seals 43

3.3.5 Field Notebooks and Field Documentation 43

3.3.6 Corrections to Documentation 44
3.4 Analytical Methods (B4) 44
3.4.1 Field Analytical Methods 44
3.4.1.1 Specific Conductance 44
3.4.1.2 Turbidity 45
3.4.1.3 pH 45
3.4.1.4 Dissolved Oxygen 45
3.4.1.5 Oxidation-Reduction Potential 45
3.4.1.6 Temperature 45
3.4.1.7 Field Screening PID Method 45
3.4.1.8 Field Screening ColorTec Method 45
3.4.1.9 Field Screening Portable GC/MS Method 45
3.4.2 Laboratory Analytical Methods 46
3.5 Quality Control (B5) 48
3.5.1 Field Quality Control Procedures 48
3.5.1.1 Field Blanks 49
3.5.1.2 Equipment Rinsate Blanks 49
3.5.1.3 Trip Blanks 49
3.5.1.4 Field Duplicates 49
3.5.1.5 Temperature Blank 49
3.5.2 Laboratory Quality Control Procedures 50
3.5.2.1 Method Blanks 50
3.5.2.2 Matrix Spikes/Matrix Spike Duplicates 50
3.5.2.3 Laboratory Duplicates 50
3.5.2.4 Postdigestion Spike 50
3.5.2.5 Serial Dilution 51
3.5.2.6 Interference Check Sample 51
3.5.2.7 Laboratory Control Samples 51
3.5.2.8 Surrogates 51
3.5.2.9 Internal Standards 51
3.6 Instrument/Equipment Testing, Inspection, Maintenance (B6) and Calibration (B7) 71
3.6.1 Field Equipment 71
3.6.2 Laboratory Equipment 73
3.7 Inspection/Acceptance of Supplies and Consumables (B8) 74
3.8 Data Acquisition Requirements – Nondirect Measurements (B9) 74
3.9 Data Management (B10) 74

Section 4 Assessment and Oversight (EPA Group C) 76

4.1 Assessments and Response Actions (C1) 76
4.1.1 Laboratory Audits 76
4.1.2 Corrective Action Procedures 76
4.1.2.1 Field Corrective Action Procedures 76
4.1.2.2 Laboratory Corrective Action Procedures 77
4.2 Reports to Management (C2) 78
4.2.1 Report Type and Frequency 78

Section 5 Data Validation and Usability (EPA Group D) 81

5.1 Data Review, Verification, and Validation (D1) 81
5.1.1 Field Data Verification 81
5.1.2 Laboratory Data Reduction, Review, and Approval 84
5.2 Verification and Validation Methods (D2) 87
5.3 Reconciliation with User Requirements (D3) 96
5.3.1 Quality Control Summary Report 96
5.3.2 Project-specific Measurement Performance Criteria 96

Section 6 References 97

List of Figures

Figure 2-1. Project Organization Chart 5

List of Tables

Table 2-1. Project Team Members 4
Table 2-2. Project Standard Operating Procedures 9
Table 2-3. Project Laboratory (EMAX Laboratories, Inc.) – Target Analytes and Reporting Limits – Volatile Organic Compounds and 1,4-Dioxane in Soil 12
Table 2-4. Project Laboratory (EMAX Laboratories, Inc.) – Target Analytes and Reporting Limits – Metals in Soil 15
Table 2-5. Project Laboratory (IGES, Microbial Insights, and McCampbell Analytical, Inc.) – Target Analytes and Reporting Limits – Geotechnical and Geochemical Parameters in Soil 16
Table 2-6. Project Field Screening Method (HAPSITE) – Target Analytes and Reporting Limits – Volatile Organic Compounds in Air and Water 17
Table 2-7. Project Laboratory (Eurofins Air Toxics, LLC) – Target Analytes and Reporting Limits – Volatile Organic Compounds in Air 18
Table 2-8. Project Laboratory (EMAX Laboratories, Inc.)– Target Analytes and Reporting Limits – Volatile Organic Compounds in Water (Groundwater/Surface Water) 21
Table 2-9. Project Laboratory (EMAX Laboratories, Inc.) – Target Analytes and Reporting Limits – 1,4-Dioxane in Water (Groundwater/Surface Water) 23
Table 2-10. Project Laboratory (EMAX Laboratories, Inc.) – Target Analytes and Reporting Limits – Metals in Water (Groundwater/Surface Water) 24
Table 2-11. Project Laboratory (EMAX Laboratory, Inc.) – Target Analytes and Reporting Limits – General Water Quality/Natural Attenuation Parameters in Water (Groundwater/Surface Water) 25
Table 2-12. Specialized Training Requirements 30
Table 3-1. Recommended Sample Container, Preservation, and Holding Times 40
Table 3-2. Quality Control Samples for Field Screening of Volatile Organic Compounds in Air and Groundwater (HAPSITE) 52
Table 3-3. Quality Control Samples (Field and Laboratory) for Volatile Organic Compounds in Soil and Water 53
Table 3-4. Precision and Accuracy for Volatile Organic Compounds in Soil and Water 55
Table 3-5. Quality Control Samples (Field and Laboratory) for 1,4-Dioxane in Soil and Water 57
Table 3-6. Precision and Accuracy for 1,4-Dioxane in Soil and Water 59
Table 3-7. Quality Control Samples (Field and Laboratory) for Metals in Soil and Water 60
Table 3-8. Precision and Accuracy for Metals in Soil and Water 62
Table 3-9. Quality Control Samples (Field and Laboratory) for Volatile Organic Compounds in Air (Modified TO-15 SIM) 63
Table 3-10. Quality Control Samples (Field and Laboratory) for Volatile Organic Compounds in Air (Modified TO-17) 65

Table 3-11. Quality Control Samples (Field and Laboratory) for General Chemistry Parameters in Water 66
Table 3-12. Precision and Accuracy for General Chemistry Parameters in Water 67
Table 3-13. Quality Control Samples (Field and Laboratory) for Oxygen and Hydrogen Stable Isotope Parameters in Water 68
Table 3-14. Quality Control Samples (Field and Laboratory) for Carbon Stable Isotope Parameters in Water 69
Table 3-15. Quality Control Samples (Field and Laboratory) for DHC and Functional Genes in Water 70
Table 3-16. Field Equipment Calibration, Maintenance, Testing, and Inspection 72
Table 4-1. Summary of Reports to Management 80
Table 5-1. Data Verification Processes for Field and Laboratory Results 82
Table 5-2. Laboratory Data Validation Qualifiers 86
Table 5-3. Data Validation Process Summary for Field and Laboratory Results 88
Table 5-4. Data Validation Flagging Criteria for Organic Methods 89
Table 5-5. Data Validation Flagging Criteria for Inorganic Methods 92

Appendices

Appendix A Field Standard Operating Procedures
Appendix B Project Data Quality Objectives
Appendix C Laboratory Quality Assurance Manuals
Appendix D Laboratory Standard Operating Procedures

Acronyms and Abbreviations

µg/L	micrograms per liter
µg/m^3	micrograms per cubic meter
°C	degrees Celsius
>	greater than
<	less than
≤	less than or equal to
%D	percent difference
%R	percent recovery
3D	3-dimensional
aq	aqueous
ASQ	American Society of Quality
ASTM	ASTM International
bvcA	vinyl chloride reductase
CA	California
CAR	corrective action report
CAS	Chemical Abstracts Service
CCV	continuing calibration verification
CDM Smith	CDM Smith Federal Programs Corporation
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CH	Certified Hydrogeologist
CIH	Certified Industrial Hygienist
CLP	Contract Laboratory Program
CMQ/OE	Certified Manager of Quality/Organizational Excellence
CO	Colorado
COPC	constituent of potential concern
CQA	Certified Quality Auditor
CSM	conceptual site model
CSIA	compound-specific isotopic analysis
CSM	conceptual site model
CSP	Certified Safety Professional
DAF	dilution attenuation factor
DCV	daily calibration verification
DMP	data management plan
DO	dissolved oxygen
DoD	U.S. Department of Defense
DQO	data quality objective
EB	equipment blank
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
FAR	Federal Acquisition Regulation
FD	field duplicate
FOC	fraction of organic carbon
FSP	field sampling plan
FTL	field team leader
GA	Georgia
gal	gallon
GC/MS	gas chromatograph/mass spectrometer

GIS	geographic information system
GISP	Geographic Information Systems Professional
GPS	Global Positioning System
H_2SO_4	sulfuric acid
HAZWOPER	Hazardous Waste Operations and Emergency Response
HCl	hydrochloric acid
HNO_3	nitric acid
ICAL	initial calibration
ICS	interference check sample
ID	identification
IS	internal standard
L	liter
LCL	lower control limit
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
m^3/kg	cubic meters per kilogram
MB	method blank
MCL	maximum contaminant level
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mL	milliliters
MO	Missouri
MPC	measurement performance criteria
MS	matrix spike
MT	Montana
MSA	method of standard addition
MSD	matrix spike duplicate
NA	not applicable
NaOH	sodium hydroxide
NCR	nonconformance report
NFGs	National Functional Guidelines
ORP	oxidation-reduction potential
PARCCS	precision, accuracy, representativeness, comparability, completeness, and sensitivity
PB	performance based
PCE	tetrachloroethene
PDS	postdigestion spike
PE	Professional Engineer
PG	Professional Geologist
PID	photoionization detector
PM	project manager
PMP	Project Management Professional
ppbV	parts per billion by volume
QA	quality assurance
QAM	quality assurance manager
QAPP	quality assurance project plan
QAS	quality assurance specialist
QC	quality control
qPCR	quantitative polymerase chain reaction

QSM	quality systems manual
RDL	representative detection limit
RI	remedial investigation
RIWP	remedial investigation work plan
RL	reporting limit
RPD	relative percent difference
RPM	Remedial Project Manager
RRF	relative response factor
rRNA	ribosomal ribonucleic acid
RSD	relative standard deviation
RSL	regional screening level
RT	retention time
SDG	sample delivery group
SOP	standard operating procedure
SRM	standard reference material
SSL	soil screening level
tceA	TCE reductase
TDS	total dissolved solids
TOC	total organic carbon
TSA	technical system audit
UCL	upper control limit
UDEQ	Utah Department of Environmental Quality
USACE	U.S. Army Corps of Engineers
USACE-KC	U.S. Army Corps of Engineers, Kansas City District
USCS	Unified Soil Classification System
UT	Utah
vcrA	vinyl chloride reductase
VHA	Veterans Health Administration
VISL	vapor intrusion screening level
VOA	volatile organic analyte
VOC	volatile organic compound
ZnAc	zinc acetate

Section 1
Introduction

This quality assurance project plan (QAPP) presents the policies, organizations, objectives, and functional activities/procedures for the Phase 2 remedial investigation (RI) being conducted at the 700 South 1600 East PCE Plume Superfund site in Salt Lake City, Utah. Phase 1 identified and collected preliminary data to support the RI, including subsurface soil and groundwater sampling at suspected source areas, groundwater delineation activities in the plume, and installation of 12 new single and multiport monitoring wells across the site. Phase 2 is follow-on work to fill identified data gaps, focusing on further delineation of groundwater contamination, primarily shallow groundwater in the area west of 1300 East. Additional details regarding the specific activities included in Phase 1 and Phase 2 of the RI are presented in the Phase 2 Remedial Investigation Work plan (RIWP) (RIWP). The QAPP was developed to document the planning, implementation, and assessment procedures for the type and quality of data needed for environmental decisions.

The QAPP follows U.S. Environmental Protection Agency (EPA) guidelines contained in *Guidance for Quality Assurance Project Plans* (EPA 2002a), and *EPA Requirements for Quality Assurance Project Plans* (EPA 2001). The development, review, approval, and implementation of the QAPP is part of the EPA mandatory quality system, which requires all organizations to develop and operate management structures and processes to verify that data used in agency decisions are of the type and quality needed for their intended use. This document structure correlates with the subtitles found in EPA guidelines (EPA 2002a, 2001). This document is organized as follows:

- **Section 1 – Introduction.** Provides the report purpose and organization.
- **Section 2 – Project Management (EPA Group A).** Provides a summary-level description of the project and task organization, background and problem definition, work tasks and project schedule, quality and objectives criteria, special training and certifications, and documents and records.
- **Section 3 – Data Generation and Acquisition (EPA Group B).** Describes the sampling design; sampling methods; sample handling and custody; analytical methods; quality control; instrument and equipment testing, inspection, and maintenance; instrument/ equipment calibration and frequency; inspection/acceptance of supplies and consumables; nondirect measurements; and data management.
- **Section 4 – Assessment and Oversight (EPA Group C).** Describes assessment, oversight, and reports to management.
- **Section 5 – Data Validation and Usability (EPA Group D).** Introduces the concepts of data review, verification, and validation; describes verification and validation methods; and explains reconciliation with user requirements.
- **Section 6 – References.** Provides a list of references used in this document.

Section 2

Project Management (EPA Group A)

2.1 Project/Task Organization (A4)

Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) investigations at the site will be conducted by CDM Smith Federal Programs Corporation (CDM Smith). The investigations will be staffed with experienced personnel with proper training to provide consistent technical support for activities defined in this QAPP and the RIWP.

The project organization and lines of authority for CDM Smith staff are illustrated on **Figure 2-1**. The organizational functions and responsibilities are consistent with the overall CERCLA guidance for RI/feasibility study projects. Project team members, respective titles, and contact information are presented in **Table 2-1**. The team members listed in **Table 2-1** will receive the approved QAPP and revisions, if any. Specific responsibilities are discussed in the following subsections.

2.1.1 Veterans Health Administration Program Manager

Ms. Shannon Smith is the Veterans Health Administration (VHA) Program Manager and will serve as the environmental restoration manager for the RI. The VHA will be involved in day-to-day project delivery. Where quality assurance (QA) problems or deficiencies in work performed under this QAPP are identified, the CDM Smith project manager (PM), quality assurance manager (QAM), and/or the quality assurance specialist (QAS), in conjunction with the VHA Program Manager, will identify the appropriate corrective action to be initiated by the field team leaders (FTLs) or the laboratory, and confirm the QAPP is amended as necessary.

2.1.2 U.S. Army Corps of Engineers Project Manager

Ms. Josephine Newton-Lund is the U.S. Army Corps of Engineers (USACE) Project Manager and will provide overall project management for the RI. Ms. Newton-Lund will coordinate all project matters with VHA and CDM Smith. A multidisciplinary team will review and provide comment on written deliverables through the USACE PM.

2.1.3 CDM Smith Project Manager

Mr. Nathan Smith will coordinate project responsibilities and financial, schedule, quality, and technical aspects of the project for CDM Smith. The CDM Smith PM, in consultation with the CDM Smith QAM, will maintain and revise the official approved field sampling plan (FSP), RIWP, and QAPP documents, and confirm distribution of revisions to project members.

2.1.4 CDM Smith Quality Assurance Manager

As QAM, Ms. Jo Nell Mullins develops the CDM Smith quality assurance (QA) program for federal government projects and assesses its implementation on the projects. Ms. Mullins plans for assessing the program via QA audits, and approves corrective actions for any deficiencies. The CDM Smith QAS and QA reviewers report to the QAM.

2.1.5 CDM Smith Subject Matter Expert

Dr. Kent Sorenson, an engineer with more than 25 years of experience, will serve as the subject matter expert for groundwater characterization, identify other subject matter experts that may be required, and verify the project's compliance with current state of the science. Dr. Sorenson will serve as a technical reviewer on CDM Smith deliverables. Dr. Sorenson will also provide technical oversight and direction to the project engineer.

2.1.6 CDM Smith Safety and Health Manager

The corporate safety and health manager, Mr. Shawn Oliveira will be responsible for implementing and maintaining the health and safety program for federal government projects at CDM Smith, and for reviewing and approving the accident prevention plan governing field activities. Mr. Oliveira will be the point of contact for safety and health issues or concerns that may arise during performance of the work.

2.1.7 CDM Smith Data Manager

Ms. Catherine Love will verify that field and laboratory data are managed in accordance with the planning documents, including maintaining the field and analytical data in EQuIS, and archive data as required by the contract. She will communicate with the CDM Smith PM, QAS, project chemist, and field staff about any issues with field or laboratory data.

2.1.8 CDM Smith Project Chemist

Ms. Cherie Zakowski will manage, coordinate, and oversee the analytical laboratories. She will also verify that laboratory data are delivered and validated in compliance with the planning documents.

2.1.9 CDM Smith Project Engineer

Mr. Neil Smith will provide oversight to verify all field investigation activities performed by CDM Smith and its subcontractors comply with the planning documents. Mr. Smith will also verify that field activities meet the quality metrics outlined in the planning documents, and that appropriate quality control (QC) inspections are performed and properly documented.

2.1.10 Laboratory Project Managers

Analytical laboratories will be subcontracted to CDM Smith. Ms. Raman Singh (EMAX Laboratories) will act as the laboratory PM for aqueous and soil samples. Mr. Brian Whittaker (Eurofins Air Toxics) will be the laboratory PM for air samples. In their roles, Ms. Singh and Mr. Whittaker will oversee laboratory analytical performance and confirm all laboratory protocols are followed to verify analytical results meet QA requirements. Ms. Singh and Mr. Whittaker will manage any lower-tier subcontract laboratories, as applicable.

Other laboratories, such as stable isotope or geotechnical, will be identified as needed for the collection of screening-level data.

The CDM Smith PM and project chemist will communicate with the laboratory PMs to resolve any issues related to laboratory analyses.

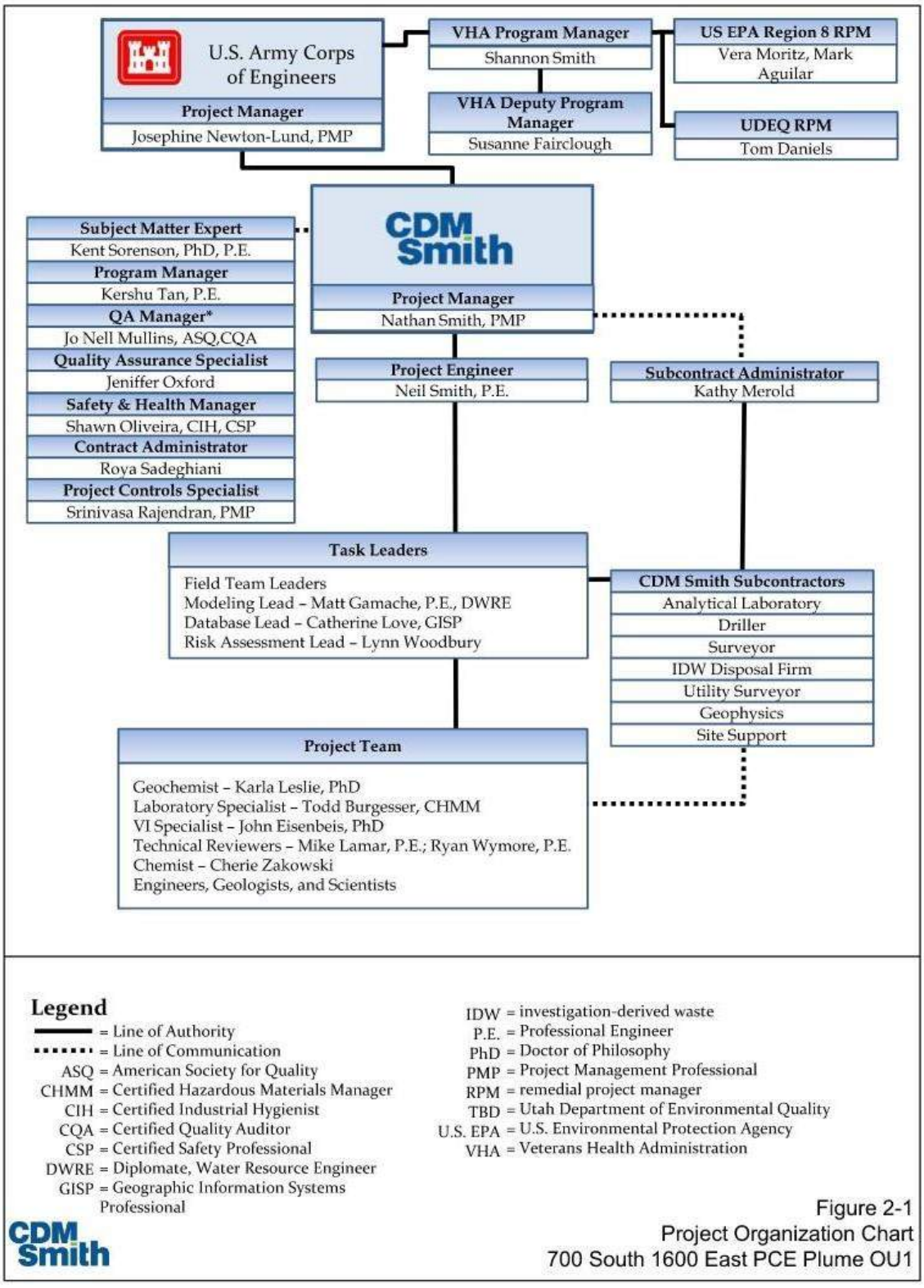
Table 2-1. Project Team Members

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Name	Title	Location	Telephone Number
Shannon Smith, PE	VHA Program Manager/Remedial Project Manager (RPM)	VHA, Salt Lake City, UT	801-582-1565, ext. 2021
Susanne Fairclough	VHA Contracts Manager	VHA, Salt Lake City, UT	801-582-1565, ext. 1952
Josephine Newton-Lund, PMP	USACE PM	USACE-KC, Kansas City, MO	816-289-3912
Nathan Smith, PMP	CDM Smith PM	CDM Smith, Denver, CO	720-264-1124
Jo Nell Mullins, ASQ CMQ/OE, CQA	CDM Smith QAM	CDM Smith, Knoxville, TN	865-963-4318
Kent Sorenson, PhD, PE	Subject Matter Expert	CDM Smith, Denver, CO	303-383-2430
Shawn Oliveira, CIH, CSP	CDM Smith Safety and Health Manager	CDM Smith, Libby, MT	406-293-2672
Catherine Love, GISP	CDM Smith Data Manager	CDM Smith, Helena, MT	406-441-1445
Cherie Zakowski	CDM Smith Project Chemist	CDM Smith, Denver, CO	720-264-1109
Neil Smith, PE	CDM Smith Project Engineer	CDM Smith, Denver, CO	303-383-2447
Todd Burgesser	CDM Smith HAPSITE Operator	CDM Smith, Denver, CO	303-383-2476
Raman Singh	Analytical Laboratory PM	EMAX Laboratories, Inc., Torrance, CA	310-618-8869, ext. 130
Brian Whittaker	Analytical Laboratory PM	Eurofins Air Toxics, LLC, Folsom, CA	916-605-3355

Notes:
ASQ = American Society of Quality
CA = California
CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act
CIH = Certified Industrial Hygienist
CMQ/OE = Certified Manager of Quality/Organizational Excellence
CO = Colorado
CQA = Certified Quality Auditor
CSP = Certified Safety Professional
GISP = Geographic Information Systems Professional
MO = Missouri
MT = Montana
NY = New York
PE = Professional Engineer
PhD = Doctor of Philosophy
PM = project manager
PMP = Project Management Professional
USACE = U.S. Army Corps of Engineers
USACE-KC = U.S. Army Corps of Engineers, Kansas City District
UT = Utah
VHA = Veterans Health Administration

Figure 2-1. Project Organization Chart



2.2 Problem Definition/Background

2.2.1 Background

Detailed information regarding the site background can be found in Section 2 of the RIWP.

2.2.2 Problem Definition

Section 4 of the RIWP provides an evaluation of the data quality objectives (DQOs) process. The outcome is presented in Table 4-1 of the RIWP, starting with the problem definition provided below.

- Tetrachloroethene (PCE)-contaminated groundwater is present beneath VHA property and is present in areas hydraulically downgradient of VHA property. A sewer line originating at the VHA property may have contributed to the release of PCE due to physical defects along the line. PCE-contaminated groundwater was also drawn toward SLC-18 when the well was in operation. Exposure pathways include:
    - Existing or new water-supply wells, and
    - Vapor intrusion and direct-contact pathways associated with shallow groundwater and springs in the East Side Springs area.

Additional data are needed to characterize the hydrogeology and nature and extent of PCE contamination, assess potential exposure pathways and risks, and allow development and detailed analysis of remedial alternatives during the subsequent feasibility study.Table 4-1 Data Quality Objectives from the RIWP is included as Appendix B.

2.3 Project Description

The tasks described in the RIWP and FSP reflect work that will be completed as part of the Phase 2 RI. The study area and site features are presented on Figure 2-1 and Figure 2-2 of the RIWP, respectively. This QAPP addresses analyses that could be performed during Phase 2 fieldwork. Applicable standard operating procedures (SOPs) are listed in **Table 2-2** and presented in **Appendix A**.

2.3.1 Description of Work Tasks

The potential activities to be completed as part of the Phase 2 RI include the following:

- Collection and off-site analysis of soil samples
- Installation of shallow and deep groundwater wells and soil vapor probes
- Push-ahead groundwater sampling during monitoring well drilling
- Collection of soil samples during well installation for geotechnical testing at an off-site laboratory
- Aquifer slug testing and aquifer pumping tests on monitoring wells
- Geophysical and groundwater flow logging within borings or wells

- Groundwater monitoring of new and existing monitoring wells, with collection of field parameters and analysis at off-site laboratories
- Surface water sampling, with collection of field parameters and analysis at off-site laboratories
- Vapor intrusion assessments including the collection of indoor air and/or subslab/near slab soil gas samples at structures within the groundwater plume boundary
- Ecological site reconnaissance
- Well maintenance as necessary

2.3.2 Project Schedule

The proposed schedule for the RI field activities and associated deliverables is summarized in Section 6, Table 6-1 of the RIWP. The CDM Smith PM will update the project schedule on a monthly basis, or more frequently if needed. Concurrent with the Phase 2 fieldwork, the groundwater modeling approach will be developed and presented in a groundwater modeling QAPP. The QAPP will also outline the communications planning, model construction methodology, calibration approach, and describe the level of detail needed in the groundwater model to meet the RI project objectives. A detailed groundwater flow model and solute transport model will be developed using data from the Phase 1 and 2 fieldwork, supplemented with historical data as appropriate. Visualization output will be generated to capture the results of these models.

2.3.3 Resources and Constraints

Available resources for this project include the planning team entities and subcontractors listed in **Figure 2-1** and equipment (e.g., drill rigs) to perform the activities required for this study. Investigation activities are planned for 2020 to allow for completion of the draft RI report in 2021. The schedule for Phase 2 activities is presented in Table 6-1 of the RIWP.

2.4 Quality Objectives and Criteria (A7)

2.4.1 Project Quality Objectives

The project DQOs were considered independently through the EPA seven-step DQO process (EPA 2006) to meet data user needs for each activity. The principle questions that this study will address are:

- What hydrogeologic features control volatile organic compound (VOC) fate and transport?
- What is the lateral and vertical extent of PCE and degradation products in groundwater downgradient from the source area?
- What is the mass discharge of PCE in groundwater at the source area and in the downgradient groundwater plume (i.e., mid plume and toe of plume)?
- How does natural attenuation change the concentrations of PCE and degradation products in the source area vadose zone and downgradient groundwater plume?

- Is there sufficient mass of PCE in the vadose zone in the source area to act as an ongoing source of PCE in groundwater?
- Would human exposure to site-related VOCs in the source area vadose zone via VI result in unacceptable risks?
- Would human exposures to site-related VOCs in groundwater within the plume area result in unacceptable risks?
- Would human and ecological exposures to site-related VOCs in surface water (i.e., springs, creeks, ponds, irrigation water) within the groundwater plume area result in unacceptable risks?

Additional discussion of project DQOs is included in Section 4.1 of the RIWP. **Appendix B** contains the outputs of the DQO process for the Phase 2 RI.

Reporting detection limits, regulatory limits, and other uses of the data were taken into consideration in selecting appropriate methods and laboratory reporting limits (RLs). VOCs, specifically PCE, trichloroethene, cis-1,2-dichloroethene, and vinyl chloride, are the currently known constituents of potential concern (COPCs) for the site. The stabilizer 1,4-dioxane (requested by EPA because of its use as stabilizer in solvents such as 1,1,1-trichloroethane) will also be monitored. Metals and various general water quality and natural attenuation parameters are included in this QAPP to provide additional data for the RI. The preliminary COPCs and characterization constituents determined from the DQO process, and their RL requirements, are listed in **Tables 2-3** through **2-11**. The selected methods are current and appropriate for this study. For most analytes, laboratory-specific RLs are expected to be below the project detection levels listed in **Tables 2-3** through **2-11**. Where sample-specific RLs are higher than project limits, the project team will use method detection limits (MDLs), as needed and available, for project decisions.

Validated laboratory chemical data will be used. This applies to any laboratory contracted by CDM Smith to perform analyses. Laboratories must have an established program for data reduction, review, approval, and reporting, as discussed following subsections.

Table 2-2. Project Standard Operating Procedures

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

SOP # or Reference[a]	Title, Revision, Date	Originating Organization	SOP Option or Equipment Type	Modified for Project? No/Yes (N/Y)	Comments
1-1	Surface Water Sampling, Rev. 10, February 2015	CDM Smith	Discrete sample, peristaltic pump or grab	N	
1-2	Sample Custody, Rev. 8, February 2015	CDM Smith	None	N	
1-3	Surface Soil Sampling, Rev. 9, February 2015	CDM Smith	Encore sampling for VOCs	N	
1-4	Subsurface Soil Sampling, Rev. 8, February 2015	CDM Smith	Sonic drilling, encore sampling for VOCs	N	
1-5	Groundwater Sampling Using Bailers, Rev. 9, February 2015	CDM Smith	None	N	Use Teflon bailer if needed
1-6	Groundwater Water Level Measurement, Rev. 9, February 2015	CDM Smith	Electrical tape, pressure gauge, and transducer	N	
1-8	Vapor Sampling Using a SUMMA Canister, Rev. 8, February 2015	CDM Smith	Time-integrated	N	
1-9	Tap Water Sampling, Rev. 7, February 2015	CDM Smith	None	N	
1-10	Field Measurement of Total Organic Vapors, Rev. 7, February 2015	CDM Smith	Photoionization detector, HAPSITE	N	Direct-reading measurement and headspace measurement with Tedlar bags
1-11	Sediment and/or Sludge Sampling, Rev. 10, February 2015	CDM Smith	None	N	
1-13	Drum Sampling, Rev. 2, February 2015	CDM Smith	None	N	
1-14	Lagoon Sampling, Rev. 2, February 2015	CDM Smith	None	N	
2-1	Packaging and Shipping Environmental Samples, Rev. 6, February 2015	CDM Smith	None	N	
2-2	Guide to Handling of Investigation-Derived Waste, Rev. 8, February 2015	CDM Smith	None	N	
3-1	Geoprobe Sampling, Rev. 7, February 2015	CDM Smith	None	N	
3-2	Topographic Survey, Rev. 8, February 2015	CDM Smith	None	N	
3-4	Geophysical Logging, Calibration, and Quality Control, Rev. 7, February 2015	CDM Smith	None	N	

Table 2-2. Project Standard Operating Procedures

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

SOP # or Reference[a]	Title, Revision, Date	Originating Organization	SOP Option or Equipment Type	Modified for Project? No/Yes (N/Y)	Comments
3-5	Lithologic Logging, Rev. 9, February 2015	CDM Smith	None	N	
3-6	Underground Facility Location, Rev. 2, February 2015	CDM Smith	None	N	
4-1	Field Logbook Content and Control, Rev. 8, February 2015	CDM Smith	None	N	
4-2	Photographic Documentation of Field Activities, Rev. 9, February 2015	CDM Smith	None	N	
4-3	Well Development and Purging, Rev. 7, February 2015	CDM Smith	None	N	
4-4	Design and Installation of Monitoring Wells in Aquifers, Rev. 8, February 2015	CDM Smith	None	N	
4-5	Field Equipment Decontamination at Nonradioactive Sites, Rev. 10, February 2015	CDM Smith	None	Y	Acids and solvents will not be used
4-6	Hydraulic Conductivity Testing, Rev. 5, February 2015	CDM Smith	None	N	
4-9	Aquifer Performance Tests, Rev. 2, February 2015	CDM Smith	None	N	
4-10	Borehole and Well Decommissioning, Rev. 2, February 2015	CDM Smith	None	N	
5-1	Control of Measurement and Test Equipment, Rev. 10, February 2015	CDM Smith	None	N	
6-1	VOC Analysis by HAPSITE GCMS - Water and Vapor	CDM Smith	None	Y	
6-2	Low-Stress (Low-Flow) Groundwater Sampling	CDM Smith	Dedicated bladder pumps and compressed gas lift pumps	Y	Preferred method of groundwater sampling
	HACH Method 8146 Iron, Ferrous (current edition)	HACH	None	N	
	How to Use Radiello	Supelco	None	N	
	AQR Color-Tec Method Procedures Manual	AQR Color-Tec	None	N	
	KT-10 Magnetic Susceptibility Meter	Terraplus	KT-10 v2 Plus	N	

Notes:

[a] All listed SOPs may not be used during the Phase 2 RI.

± = plus or minus

< = less than

Table 2-2. Project Standard Operating Procedures

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

SOP # or Reference[a]	Title, Revision, Date	Originating Organization	SOP Option or Equipment Type	Modified for Project? No/Yes (N/Y)	Comments

% = percent
GC/MS = gas chromatograph mass spectrometer
NTU = nephelometric turbidity unit
rev = revision
SOP = standard operating procedure
VOC = volatile organic compound

Table 2-3. Project Laboratory (EMAX Laboratories, Inc.) – Target Analytes and Reporting Limits – Volatile Organic Compounds in Soil

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Lowest Screening Level Value (mg/kg)[a]	Laboratory RL (mg/kg)	Laboratory MDL (mg/kg)
1,1,1-Trichloroethane	71-55-6	SW8260C	Protection of Groundwater MCL-SSL (DAF=20)	1.4	0.005	0.001
1,1,2,2-Tetrachloroethane	79-34-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0006[b]	0.005	0.001
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	SW8260C	Protection of Groundwater SSL (DAF=20)	510	0.005	0.001
1,1,2-Trichloroethane	79-00-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0018[b]	0.005	0.001
1,1-Dichloroethane	75-34-3	SW8260C	Protection of Groundwater SSL (DAF=20)	0.016	0.005	0.001
1,1-Dichloroethene	75-35-4	SW8260C	Protection of Groundwater MCL-SSL (DAF=20)	0.05	0.005	0.001
1,2,3-Trichlorobenzene	87-61-6	SW8260C	Protection of Groundwater SSL (DAF=20)	0.42	0.005	0.001
1,2,4-Trichlorobenzene	120-82-1	SW8260C	Protection of Groundwater SSL (DAF=20)	0.674	0.005	0.001
1,2,4-Trimethylbenzene	95-63-6	SW8260C	Protection of Groundwater SSL (DAF=20)	1.6	0.005	0.001
1,3,5-Trimethylbenzene	108-67-8	SW8260C	Protection of Groundwater SSL (DAF=20)	1.7	0.005	0.001
1,2-Dibromo-3-Chloropropane	96-12-8	SW8260C	Protection of Groundwater SSL (DAF=20)	0.000029[b]	0.005	0.001
1,2-Dibromoethane (EDB)	106-93-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.000042[b]	0.005	0.001
1,2-Dichlorobenzene	95-50-1	SW8260C	Protection of Groundwater SSL (DAF=20)	5.9	0.005	0.001
1,2-Dichloroethane	107-06-2	SW8260C	Protection of Groundwater SSL (DAF=20)	0.00097[b]	0.005	0.001
1,2-Dichloropropane	78-87-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0056	0.005	0.001
1,3-Dichlorobenzene	541-73-1	SW8260C	NA	NA	0.005	0.001
1,4-Dichlorobenzene	106-46-7	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0092	0.005	0.001
2-Butanone (Methyl Ethyl Ketone)	78-93-3	SW8260C	Protection of Groundwater SSL (DAF=20)	23	0.02	0.005
2-Hexanone	591-78-6	SW8260C	Protection of Groundwater SSL (DAF=20)	0.18	0.02	0.005
4-Methyl-2-pentanone	108-10-1	SW8260C	Protection of Groundwater SSL (DAF=20)	28	0.02	0.005
Acetone	67-64-1	SW8260C	Protection of Groundwater SSL (DAF=20)	58	0.02	0.005

Table 2-3. Project Laboratory (EMAX Laboratories, Inc.) – Target Analytes and Reporting Limits – Volatile Organic Compounds in Soil

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Lowest Screening Level Value (mg/kg)[a]	Laboratory RL (mg/kg)	Laboratory MDL (mg/kg)
Benzene	71-43-2	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0047[b]	0.005	0.001
Bromochloromethane	74-97-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.42	0.005	0.001
Bromodichloromethane	75-27-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.00073[b]	0.005	0.001
Bromoform	75-25-2	SW8260C	Protection of Groundwater SSL (DAF=20)	0.018	0.005	0.001
Bromomethane	74-83-9	SW8260C	Protection of Groundwater SSL (DAF=20)	0.038	0.01	0.002
Carbon Disulfide	75-15-0	SW8260C	Protection of Groundwater SSL (DAF=20)	4.8	0.005	0.001
Carbon Tetrachloride	56-23-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0035[b]	0.005	0.001
Chlorobenzene	108-90-7	SW8260C	Protection of Groundwater SSL (DAF=20)	1.1	0.005	0.001
Chloroethane	75-00-3	SW8260C	Protection of Groundwater SSL (DAF=20)	120	0.005	0.001
Chloroform	67-66-3	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0012[b]	0.005	0.001
Chloromethane	74-87-3	SW8260C	Protection of Groundwater SSL (DAF=20)	0.097	0.005	0.001
cis-1,2-Dichloroethene	156-59-2	SW8260C	Protection of Groundwater SSL (DAF=20)	0.021	0.005	0.001
cis-1,3-Dichloropropene	10061-01-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0034[b]	0.005	0.001
Dibromochloromethane	124-48-1	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0046[b]	0.005	0.001
Dichlorodifluoromethane (Freon 12)	75-71-8	SW8260C	Protection of Groundwater SSL (DAF=20)	6.1	0.005	0.001
Ethylbenzene	100-41-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.034	0.005	0.001
Isopropylbenzene	98-82-8	SW8260C	Protection of Groundwater SSL (DAF=20)	15	0.005	0.001
Methyl Acetate	79-20-9	SW8260C	Protection of Groundwater SSL (DAF=20)	82	0.005	0.0015
Methyl Tert-Butyl Ether	1634-04-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.064	0.005	0.001
Methylene Chloride	75-09-2	SW8260C	Protection of Groundwater MCL-SSL (DAF=20)	0.026	0.01	0.0025

Table 2-3. Project Laboratory (EMAX Laboratories, Inc.) – Target Analytes and Reporting Limits – Volatile Organic Compounds in Soil

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Lowest Screening Level Value (mg/kg)[a]	Laboratory RL (mg/kg)	Laboratory MDL (mg/kg)
m,p-Xylene	108-38-3 and 106-42-3	SW8260C	Protection of Groundwater SSL (DAF=20)	3.8	0.01	0.0025
o-Xylene	95-47-6	SW8260C	Protection of Groundwater SSL (DAF=20)	3.8	0.005	0.001
Styrene	100-42-5	SW8260C	Protection of Groundwater MCL-SSL (DAF=20)	2.2	0.005	0.001
Tetrachloroethene	127-18-4	SW8260C	Protection of Groundwater MCL-SSL (DAF=20)	0.046[b]	0.005	0.001
Toluene	108-88-3	SW8260C	Protection of Groundwater SSL (DAF=20)	14	0.005	0.001
trans-1,3-Dichloropropene	10061-02-6	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0034[b]	0.005	0.001
trans-1,2-Dichloroethene	156-60-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.62	0.005	0.001
Trichloroethene	79-01-6	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0035[b]	0.005	0.001
Trichlorofluoromethane (Freon 11)	75-69-4	SW8260C	Protection of Groundwater SSL (DAF=20)	66	0.005	0.0011
Vinyl Acetate	108-05-4	SW8260C	Protection of Groundwater SSL (DAF=20)	1.7	0.005	0.0013
Vinyl Chloride	75-01-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.00013 [b]	0.005	0.0014

[a] Lowest of: (1) RSLs for residential exposure or (2) SSLs for groundwater protection using a DAF of 20 and soil saturation level. RSLs corresponding to an excess lifetime cancer risk of 1×10^{-6} and a hazard quotient of 1 were used (EPA November 2019).

[b] Because of the low screening level for this analyte, the RL is greater than the screening level. However, soil screening would be used in a source investigation in which the RL would be an acceptable limit.

DAF References: *Soil Screening Guidance: User's Guide* (EPA 1996) and *Supplemental Guidance for Developing Soil Screening Levels for Superfund Sites* (EPA 2002b)

Notes:
CAS = Chemical Abstracts Service
DAF = dilution attenuation factor
MCL = maximum contaminant level
MDL = method detection limit
mg/kg = milligrams per kilogram
NA = not applicable
RL = reporting limit
RSL = regional screening level
SSL = soil screening level

Table 2-4. Project Laboratory (EMAX Laboratories, Inc.) – Target Analytes and Reporting Limits – Metals in Soil
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Lowest Screening Level Value (mg/kg)[a]	Laboratory RL (mg/kg)	Laboratory MDL (mg/kg)
Aluminum	7429-90-5	SW6020A	EPA RSL	77,000	100	20
Antimony	7440-36-0	SW6020A	Protection of Groundwater SSL (DAF=20)	5.4	0.5	0.1
Arsenic	7440-38-2	SW6020A	Protection of Groundwater SSL (DAF=20)	0.03[b]	0.5	0.1
Barium	7440-39-3	SW6020A	EPA RSL	1,600	0.5	0.1
Beryllium	7440-41-7	SW6020A	EPA RSL	64	0.5	0.05
Cadmium	7440-43-9	SW6020A	Protection of Groundwater SSL (DAF=20)	14	0.5	0.05
Calcium	7440-70-2	SW6020A	NA	NA	200	50
Chromium	7440-47-3	SW6020A	Protection of Groundwater MCL-SSL (DAF=20)	3,600,000	1	0.25
Cobalt	7440-48-4	SW6020A	EPA RSL	5.4	0.5	0.05
Copper	7440-50-8	SW6020A	Protection of Groundwater SSL (DAF=20)	560	1	0.25
Iron	7439-89-6	SW6020A	Protection of Groundwater SSL (DAF=20)	7,000	100	25
Lead	7439-92-1	SW6020A	Protection of Groundwater MCL-SSL (DAF=20)	280	0.5	0.05
Magnesium	7439-95-4	SW6020A	NA	NA	100	25
Manganese	7439-96-5	SW6020A	Protection of Groundwater SSL (DAF=20)	570	2	0.5
Mercury	7487-94-7	SW7471A	Protection of Groundwater SSL (DAF=20)	23	0.1	0.033
Nickel	7440-02-0	SW6020A	Protection of Groundwater SSL (DAF=20)	510	1	0.15
Potassium	7440-09-7	SW6020A	NA	NA	100	25
Selenium	7782-49-2	SW6020A	Protection of Groundwater MCL-SSL (DAF=20)	5.2	0.5	0.05
Silver	7440-22-4	SW6020A	Protection of Groundwater SSL (DAF=20)	16	0.5	0.05
Sodium	7440-23-5	SW6020A	NA	NA	250	25
Thallium	7440-28-0	SW6020A	Protection of Groundwater SSL (DAF=20)	0.28[b]	0.5	0.05
Vanadium	7440-62-2	SW6020A	EPA RSL	390	0.5	0.15
Zinc	7440-66-6	SW6020A	Protection of Groundwater SSL (DAF=20)	7,500	10	2.5

[a] Lowest of: (1) RSLs for residential exposure or (2) SSLs for groundwater protection using DAF of 20 and soil saturation level. RSLs corresponding to an excess lifetime cancer risk of 1×10^{-6} and a hazard quotient of 1 were used (EPA November 2019).
[b] Because of the low screening level for this analyte, the RL is greater than the screening level. However, this analyte is not a known constituent of potential concern for the site.

Notes:
CAS = Chemical Abstracts Service
DAF = dilution attenuation factor
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level NA = not applicable
MDL = method detection limit RL = reporting limit
mg/kg = milligrams per kilogram RSL = regional screening level
SSL = soil screening level

Table 2-5. Project Laboratory (Intermountain GeoEnvironmental Services, Inc., Microbial Insights, and McCampbell Analytical, Inc.) – Target Analytes and Reporting Limits – Geotechnical and Geochemical Parameters in Soil

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Lowest Screening Level Value (mg/kg)	Laboratory RL
FOC	NA	ASTM D2974	NA	NA	0.1 %
Magnetic Susceptibility	NA	Bartington System	NA	NA	1×10^{-9} m^3/kg
Ferrous Iron Minerals	NA	SM3500-Fe B4c	NA	NA	1 mg/kg
Sieve Analysis	NA	ASTM D6913	NA	NA	NA
Dry Bulk Soil Density	NA	ASTM D2937	NA	NA	NA
Hydrometer	NA	ASTM D7928	NA	NA	NA
USCS Soil Classification	NA	ASTM D2487	NA	NA	NA
Atterberg Limits	NA	ASTM D4318	NA	NA	NA
Gradation	NA	ASTM D1140	NA	NA	NA
Vertical Permeability	NA	ASTM D2434	NA	NA	NA
Moisture Content	NA	ASTM D2216	NA	NA	NA

Notes:
ASTM = ASTM International
CAS = Chemical Abstracts Service
FOC = fraction of organic carbon
m^3/kg = cubic meters per kilogram
mg/kg = milligrams per kilogram
NA = not applicable
RL = reporting limit
USCS = Unified Soil Classification System

Table 2-6. Project Field Screening Method (HAPSITE) – Target Analytes and Reporting Limits – Volatile Organic Compounds in Air and Water

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level[a]	Lowest Screening Level Value (µg/m³)[a]	Method RL (µg/m³)
Air					
cis-1,2-Dichloroethene	156-59-2	HAPSITE	EPA RSL	NA	1
Tetrachloroethene	127-18-4	HAPSITE	EPA RSL	11	1
Trichloroethene	79-01-6	HAPSITE	EPA RSL	0.48[b]	1
Analyte	**CAS Number**	**Method**	**Screening Level**	**Screening Level Value (µg/L)[b]**	**Method RL (µg/m³)**
Water					
cis-1,2-Dichloroethene	156-59-2	HAPSITE Headspace Analyzer	EPA MCL	70	5
Tetrachloroethene	127-18-4	HAPSITE Headspace Analyzer	EPA MCL	5	5
Trichloroethene	79-01-6	HAPSITE Headspace Analyzer	EPA MCL	5	5

[a] EPA RSL, Resident Air, November 2019, screening levels were based on a target excess lifetime cancer risk of 1×10^{-6} and a hazard quotient of 1.

[b] Because of the low screening level for this analyte, the RL is greater than the screening level. However, the HAPSITE provides screening level data only, and measurements will be confirmed by definitive analysis.

Notes:
µg/m³ = micrograms per cubic meter
µg/L = micrograms per liter
CAS = Chemical Abstracts Service
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level
RL= reporting limit
RSL = regional screening level

Table 2-7. Project Laboratory (Eurofins Air Toxics, LLC) – Target Analytes and Reporting Limits – Volatile Organic Compounds in Air

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Air Method	Screening Level[a]	Lowest Screening Level Value (µg/m³)[a]	Laboratory RL (µg/m³)	Laboratory MDL (µg/m³)
Laboratory Analytical Parameters (SUMMA®)						
1,1,1-Trichloroethane	71-55-6	Modified TO-15	EPA RSL	5,200	0.11	0.033
1,1,2,2-Tetrachloroethane	79-34-5	Modified TO-15	EPA RSL	0.048[b]	0.14	0.045
1,1,2-Trichloroethane	79-00-5	Modified TO-15	EPA RSL	0.18	0.11	0.033
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	Modified TO-15	EPA RSL	5,200	0.77	0.15
1,1-Dichloroethane	75-34-3	Modified TO-15	EPA RSL	1.8	0.081	0.027
1,1-Dichloroethene	75-35-4	Modified TO-15	EPA RSL	210	0.040	0.032
1,2,4-Trichlorobenzene	120-82-1	Modified TO-15	EPA RSL	2.1[b]	3.7	1.3
1,2,4-Trimethylbenzene	95-63-6	Modified TO-15	EPA RSL	63	0.49	0.11
1,2-Dibromoethane	106-93-4	Modified TO-15	EPA RSL	0.0047[b]	0.15	0.024
1,2-Dichlorobenzene	95-50-1	Modified TO-15	EPA RSL	210	0.60	0.11
1,2-Dichloroethane	107-06-2	Modified TO-15	EPA RSL	0.11	0.081	0.015
1,2-Dichloropropane	78-87-5	Modified TO-15	EPA RSL	0.76	0.46	0.11
1,2-Dichlorotetrafluoroethane (Freon 114)	76-14-2	Modified TO-15	NA	NA	0.14	0.041
1,3,5-Trimethylbenzene	108-67-8	Modified TO-15	EPA RSL	63	0.49	0.098
1,3-Butadiene	106-99-0	Modified TO-15	EPA RSL	0.094[b]	0.22	0.048
1,3-Dichlorobenzene	541-73-1	Modified TO-15	NA	NA	0.60	0.21
1,4-Dichlorobenzene	106-46-7	Modified TO-15	EPA RSL	0.26	0.12	0.068
1,4-Dioxane	123-91-1	Modified TO-15	EPA RSL	0.56	0.36	0.19
2-Butanone (Methyl Ethyl Ketone)	78-93-3	Modified TO-15	EPA RSL	5,200	1.5	0.30
2-Hexanone	591-78-6	Modified TO-15	EPA RSL	31	2.0	0.52
2-Propanol (Isopropyl alcohol)	67-63-0	Modified TO-15	EPA RSL	210	1.2	0.14
3-Chloropropene (Ally chloride)	107-05-1	Modified TO-15	EPA RSL	0.47[b]	1.6	0.57
4-Ethyltoluene	622-96-8	Modified TO-15	EPA RSL	NA	0.49	0.11
4-Methyl-2-pentanone	108-10-1	Modified TO-15	EPA RSL	3,100	0.41	0.1
Acetone	67-64-1	Modified TO-15	EPA RSL	32,000	2.4	0.4
Alpha-Chlorotoluene (Benzyl chloride)	100-44-7	Modified TO-15	EPA RSL	0.057[b]	0.52	0.1
Benzene	71-43-2	Modified TO-15	EPA RSL	0.36	0.16	0.094
Bromodichloromethane	75-27-4	Modified TO-15	EPA RSL	0.076[b]	0.67	0.24
Bromoform	75-25-2	Modified TO-15	EPA RSL	2.6	1.0	0.24
Bromomethane	74-83-9	Modified TO-15	EPA RSL	5.2	1.9	0.34

Table 2-7. Project Laboratory (Eurofins Air Toxics, LLC) – Target Analytes and Reporting Limits – Volatile Organic Compounds in Air

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Air Method	Screening Level[a]	Lowest Screening Level Value (µg/m³)[a]	Laboratory RL (µg/m³)	Laboratory MDL (µg/m³)
Carbon disulfide	75-15-0	Modified TO-15	EPA RSL	730	1.6	0.27
Carbon tetrachloride	56-23-5	Modified TO-15	EPA RSL	0.47	0.12	0.058
Chlorobenzene	108-90-7	Modified TO-15	EPA RSL	52	0.46	0.12
Chloroethane	75-00-3	Modified TO-15	EPA RSL	10,000	0.13	0.02
Chloroform	67-66-3	Modified TO-15	EPA RSL	0.12	0.098	0.028
Chloromethane	74-87-3	Modified TO-15	EPA RSL	94	1.0	0.025
cis-1,2-Dichloroethene	156-59-2	Modified TO-15	EPA RSL	NA	0.079	0.028
cis-1,3-Dichloropropene	10061-01-5	Modified TO-15	EPA RSL	0.70	0.45	0.091
Cumene (isopropylbenzene)	98-82-8	Modified TO-15	EPA RSL	420	0.49	0.076
Cyclohexane	110-82-7	Modified TO-15	EPA RSL	6,300	0.34	0.093
Dibromochloromethane	124-48-1	Modified TO-15	EPA RSL	NA	0.85	0.26
Dichlorodifluoromethane (Freon 12)	75-71-8	Modified TO-15	EPA RSL	100	0.099	0.025
Ethylbenzene	100-41-4	Modified TO-15	EPA RSL	1.1	0.087	0.057
Ethanol	64-17-5	Modified TO-15	EPA RSL	NA	0.94	0.2
n-Heptane	142-82-5	Modified TO-15	EPA RSL	420	0.41	0.14
Hexachlorobutadiene	87-68-3	Modified TO-15	EPA RSL	0.13[b]	5.3	1.8
n-Hexane	110-54-3	Modified TO-15	EPA RSL	730	1.8	0.2
m,p-Xylene	108-38-3 and 106-42-3	Modified TO-15	EPA RSL	100	0.17	0.1
Methylene Chloride	75-09-2	Modified TO-15	EPA RSL	100	0.69	0.1
Methyl tert-butyl ether	1634-04-4	Modified TO-15	EPA RSL	11	0.36	0.031
o-Xylene	95-47-6	Modified TO-15	EPA RSL	100	0.087	0.019
Propylbenzene	103-65-1	Modified TO-15	EPA RSL	1,000	0.49	0.12
Styrene	100-42-5	Modified TO-15	EPA RSL	1,000	0.42	0.046
Tetrachloroethene	127-18-4	Modified TO-15	EPA RSL	11	0.14	0.026
Tetrahydrofuran	109-99-9	Modified TO-15	EPA RSL	2,100	1.5	0.59
Toluene	108-88-3	Modified TO-15	EPA RSL	5,200	0.19	0.057
trans-1,2-Dichloroethene	156-60-5	Modified TO-15	EPA RSL	NA	0.40	0.03
trans-1,3-Dichloropropene	10061-02-6	Modified TO-15	EPA RSL	0.70	0.45	0.11
Trichloroethene	79-01-6	Modified TO-15	EPA RSL	0.48	0.11	0.073
Trichlorofluoromethane (Freon 11)	75-69-4	Modified TO-15	EPA RSL	NA	0.56	0.12
Vinyl Chloride	75-01-4	Modified TO-15	EPA RSL	0.17	0.026	0.02

Table 2-7. Project Laboratory (Eurofins Air Toxics, LLC) – Target Analytes and Reporting Limits – Volatile Organic Compounds in Air

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Air Method	Screening Level[a]	Lowest Screening Level Value (µg/m³)[a]	Laboratory RL (µg/m³)	Laboratory MDL (µg/m³)
Laboratory Analytical Parameters (Passive Sampler Radiello 130)						
1,1,1-Trichloroethane	71-55-6	Modified TO-17	EPA RSL	5,200	0.053	0.014
1,2-Dichloroethane	107-06-2	Modified TO-17	EPA RSL	0.011[b]	0.043	0.014
1,4-Dichlorobenzene	106-46-7	Modified TO-17	EPA RSL	0.26	0.065	0.024
2-Butanone (Methyl Ethyl Ketone)	78-93-3	Modified TO-17	EPA RSL	5,200	0.042	0.017
4-Methyl-2-pentanone	108-10-1	Modified TO-17	EPA RSL	3,100	0.099	0.032
Benzene	71-43-2	Modified TO-17	EPA RSL	0.36	0.17	0.12
Carbon tetrachloride	56-23-5	Modified TO-17	EPA RSL	0.47	0.049	0.015
Chlorobenzene	108-90-7	Modified TO-17	EPA RSL	52	0.049	0.017
Chloroform	67-66-3	Modified TO-17	EPA RSL	0.12	0.044	0.017
Cyclohexane	110-82-7	Modified TO-17	EPA RSL	6,300	0.061	0.02
Ethanol	64-17-5	Modified TO-17	EPA RSL	NA	0.32	0.094
Ethyl Acetate	141-78-6	Modified TO-17	EPA RSL	73	0.17	0.11
Ethylbenzene	100-41-4	Modified TO-17	EPA RSL	1.1	0.049	0.019
n-Heptane	142-82-5	Modified TO-17	EPA RSL	420	0.057	0.02
n-Hexane	110-54-3	Modified TO-17	EPA RSL	730	0.050	0.02
m,p-Xylene	108-38-3	Modified TO-17	EPA RSL	100	0.047	0.034
Methyl tert-butyl ether	1634-04-4	Modified TO-17	EPA RSL	11	0.051	0.02
Naphthalene	91-20-3	Modified TO-17	EPA RSL	0.083[b]	0.13	0.051
o-Xylene	95-47-6	Modified TO-17	EPA RSL	100	0.051	0.018
Propylbenzene	103-65-1	Modified TO-17	EPA RSL	1,000	0.058	0.021
Styrene	100-42-5	Modified TO-17	EPA RSL	1,000	0.054	0.031
Tetrachloroethene	127-18-4	Modified TO-17	EPA RSL	11	0.056	0.014
Toluene	108-88-3	Modified TO-17	EPA RSL	5,200	0.045	0.016
Trichloroethene	79-01-6	Modified TO-17	EPA RSL	0.48	0.048	0.02

[a] EPA RSL, Resident Air, November 2019, screening levels were based on a target excess lifetime cancer risk of 1×10^{-6} and a hazard quotient of 1.

[b] Because of the low screening level for this analyte, the RL is greater than the screening level. However, this analyte is not a known COPC for the site.

Notes:
µg/m³ = micrograms per cubic meter
COPC = constituent of potential concern
EPA = Environmental Protection Agency
MDL = method detection limit
RL= reporting limit
RSL = regional screening level

Table 2-8. Project Laboratory (EMAX Laboratories, Inc.)– Target Analytes and Reporting Limits – Volatile Organic Compounds in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (µg/L)[a]	Laboratory RL (µg/L)	Laboratory MDL (µg/L)
Laboratory Analytical Parameters						
1,1,1-Trichloroethane	71-55-6	SW8260C	EPA MCL	200	1	0.1
1,1,2,2-Tetrachloroethane	79-34-5	SW8260C	EPA RSL	0.076[b]	1	0.11
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	SW8260C	EPA RSL	10,000	1	0.15
1,1,2-Trichloroethane	79-00-5	SW8260C	EPA MCL	5	1	0.1
1,1-Dichloroethane	75-34-3	SW8260C	EPA RSL	2.8	1	0.1
1,1-Dichloroethene	75-35-4	SW8260C	EPA MCL	7	1	0.1
1,2,3-Trichlorobenzene	87-61-6	SW8260C	EPA RSL	0.7[b]	1	0.15
1,2,4-Trichlorobenzene	120-82-1	SW8260C	EPA MCL	70	1	0.15
1,2,4-Trimethylbenzene	95-63-6	SW8260C	EPA RSL	56	1	0.11
1,3,5-Trimethylbenzene	108-67-8	SW8260C	EPA RSL	60	1	0.12
1,2-Dibromo-3-Chloropropane	96-12-8	SW8260C	EPA MCL	0.2[b]	2	0.25
1,2-Dibromoethane	106-93-4	SW8260C	EPA MCL	0.05[b]	1	0.103
1,2-Dichlorobenzene	95-50-1	SW8260C	EPA MCL	600	1	0.1
1,2-Dichloroethane	107-06-2	SW8260C	EPA MCL	5	1	0.1
1,2-Dichloropropane	78-87-5	SW8260C	EPA MCL	5	1	0.1
1,3-Dichlorobenzene	541-73-1	SW8260C	NA	NA	1	0.11
1,4-Dichlorobenzene	106-46-7	SW8260C	EPA MCL	75	1	0.1
2-Butanone (Methyl Ethyl Ketone)	78-93-3	SW8260C	EPA RSL	5,600	20	2.5
2-Hexanone	591-78-6	SW8260C	EPA RSL	38	20	2.5
4-Methyl-2-pentanone	108-10-1	SW8260C	EPA RSL	6,300	20	2.5
Acetone	67-64-1	SW8260C	EPA RSL	14,000	20	2.5
Benzene	71-43-2	SW8260C	EPA MCL	5	1	0.1
Bromochloromethane	74-97-5	SW8260C	EPA RSL	83	1	0.11
Bromodichloromethane	75-27-4	SW8260C	EPA MCL	80	1	0.1
Bromoform	75-25-2	SW8260C	EPA MCL	80	1	0.15
Bromomethane	74-83-9	SW8260C	EPA RSL	7.5	1	0.16
Carbon Disulfide	75-15-0	SW8260C	EPA RSL	810	1	0.25
Carbon Tetrachloride	56-23-5	SW8260C	EPA MCL	5	1	0.1
Chlorobenzene	108-90-7	SW8260C	EPA MCL	100	1	0.1
Chloroethane	75-00-3	SW8260C	EPA RSL	21,000	1	0.27
Chloroform	67-66-3	SW8260C	EPA MCL	80	1	0.1
Chloromethane	74-87-3	SW8260C	EPA RSL	190	1	0.15
cis-1,2-Dichloroethene	156-59-2	SW8260C	EPA MCL	70	1	0.1
cis-1,3-Dichloropropene	10061-01-5	SW8260C	EPA RSL	0.47[b]	1	0.1

Table 2-8. Project Laboratory (EMAX Laboratories, Inc.)– Target Analytes and Reporting Limits – Volatile Organic Compounds in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (µg/L)[a]	Laboratory RL (µg/L)	Laboratory MDL (µg/L)
Dibromochloromethane	124-48-1	SW8260C	EPA MCL	80	1	0.1
Dichlorodifluoromethane (Freon 12)	75-71-8	SW8260C	EPA RSL	200	1	0.15
Ethylbenzene	100-41-4	SW8260C	EPA MCL	700	1	0.1
Isopropylbenzene (Cumene)	98-82-8	SW8260C	EPA RSL	450	1	0.1
Methyl acetate	79-20-9	SW8260C	EPA RSL	20,000	2	0.25
Methyl Tert-Butyl Ether	1634-04-4	SW8260C	EPA RSL	14	1	0.13
Methylene chloride	75-09-2	SW8260C	EPA MCL	5	2	0.5
m,p-Xylene	108-38-3 and 106-42-3	SW8260C	EPA RSL	190	2	0.21
o-Xylene	95-47-6	SW8260C	EPA RSL	190	1	0.1
Styrene	100-42-5	SW8260C	EPA MCL	100	1	0.25
Tetrachloroethene (PCE)	127-18-4	SW8260C	EPA MCL	5	1	0.15
Toluene	108-88-3	SW8260C	EPA MCL	1,000	1	0.1
trans-1,3-Dichloropropene	10061-02-6	SW8260C	EPA RSL	0.47[b]	1	0.11
trans-1,2-Dichloroethene	156-60-5	SW8260C	EPA MCL	100	1	0.1
Trichloroethene	79-01-6	SW8260C	EPA MCL	5	1	0.1
Trichlorofluoromethane (Freon 11)	75-69-4	SW8260C	EPA RSL	5,200	1	0.15
Vinyl Acetate	108-05-4	SW8260C	EPA RSL	410	2	0.25
Vinyl Chloride	75-01-4	SW8260C	EPA MCL	2	1	0.12

[a] If an MCL is set for the analyte, the screening level is the MCL. Otherwise, the screening level is the RSL for tap water. RSLs corresponding to an excessive lifetime cancer risk of 1×10^{-6} and a hazard quotient of 1 were used (EPA November 2019).

[b] Because of the low screening level for this analyte, the RL is greater than the screening level. However, this analyte is not a known constituent of potential concern for the site.

Notes:
µg/L = micrograms per liter
CAS = Chemical Abstracts Service
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level
MDL = method detection limit
RL = reporting limit
RSL = regional screening level

Table 2-9. Project Laboratory (EMAX Laboratories, Inc.) – Target Analytes and Reporting Limits – 1,4-Dioxane in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (µg/L)[a]	Laboratory RL (µg/L)	Laboratory MDL (µg/L)
1,4-Dioxane	123-91-1	SW8270SIM-low	EPA RSL	0.46	0.4	0.21

[a] If an MCL is set for the analyte, the screening level is the MCL. Otherwise, the screening level is the RSL for tap water. RSLs corresponding to an excessive lifetime cancer risk of 1×10^{-6} and a hazard quotient of 1 were used (EPA November 2019).

Notes:
µg/L = micrograms per liter
CAS = Chemical Abstracts Service
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level
MDL = method detection limit
RL = reporting limit
RSL = regional screening level

Table 2-10. Project Laboratory (EMAX Laboratories, Inc.) – Target Analytes and Reporting Limits – Metals in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (µg/L)[a]	Laboratory RL (µg/L)	Laboratory MDL (µg/L)
Aluminum	7429-90-5	SW60620A	EPA RSL	20,000	100	25
Antimony	7440-36-0	SW60620A	EPA MCL	6	1	0.25
Arsenic	7440-38-2	SW60620A	EPA MCL	10	1	0.125
Barium	7440-39-3	SW60620A	EPA MCL	2,000	1	0.25
Beryllium	7440-41-7	SW60620A	EPA MCL	4	1	0.1
Cadmium	7440-43-9	SW60620A	EPA MCL	5	1	0.1
Calcium	7440-70-2	SW60620A	NA	NA	100	25
Chromium	7440-47-3	SW60620A	EPA MCL	100	1	0.1
Cobalt	7440-48-4	SW60620A	EPA RSL	6	1	0.1
Copper	7440-50-8	SW60620A	EPA MCL	1,300	2	0.5
Iron	7439-89-6	SW60620A	EPA RSL	14,000	100	25
Lead	7439-92-1	SW60620A	EPA MCL	15	1	0.05
Magnesium	7439-95-4	SW60620A	NA	NA	100	25
Manganese	7439-96-5	SW60620A	EPA RSL	430	1	0.25
Mercury	7487-94-7	SW7470A	EPA MCL	2	0.5	0.1
Nickel	7440-02-0	SW60620A	EPA RSL	390	1	0.25
Potassium	7440-09-7	SW60620A	NA	NA	100	25
Selenium	7782-49-2	SW60620A	EPA MCL	50	1	0.15
Silver	7440-22-4	SW60620A	EPA RSL	94	1	0.1
Sodium	7440-23-5	SW60620A	NA	NA	100	25
Thallium	7440-28-0	SW60620A	EPA MCL	2	1	0.1
Vanadium	7440-62-2	SW60620A	EPA RSL	86	1	0.25
Zinc	7440-66-6	SW60620A	EPA RSL	6,000	20	5

[a] If an MCL is set for the analyte, the screening level is the MCL. Otherwise, the screening level is the RSL for tap water. RSLs corresponding to an excessive lifetime cancer risk of 1×10^{-6} and a hazard quotient of 1 were used (EPA November 2019).

Notes:
µg/L = micrograms per liter
CAS = Chemical Abstracts Service
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level
MDL = method detection limit
NA = not applicable
RL = reporting limit
RSL = regional screening level

Table 2-11. Project Laboratory (EMAX Laboratory, Inc.) – Target Analytes and Reporting Limits – General Water Quality/Natural Attenuation Parameters in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

General Water Quality

Analyte	CAS Number	Method	Screening Level	Screening Level Value (mg/L)[a]	Laboratory RL (mg/L)	Laboratory MDL (mg/L)
TOC	TOC	SW9060	NA	NA	1	0.25
TDS	TDS	SM2540C	NA	NA	10	10
Ethane (Dissolved Gas)	74-84-0	RSK-175	NA	NA	0.002	0.002
Ethene (Dissolved Gas)	74-85-1	RSK-175	NA	NA	0.002	0.002
Methane (Dissolved Gas)	74-82-8	RSK-175	NA	NA	0.002	0.002
Chloride	16887-00-6	E300.0	NA	NA	0.2	0.05
Nitrate/Nitrite	14797-55-8/ 14797-65-0		EPA MCL	10/1.0	0.05	0.025
Sulfate	14808-79-8		NA	NA	0.5	0.13
Nitrate/Nitrite-N	14797-55-8/14797-65-0	SM4500-NO3E	EPA MCL	10/1.0	0.05	0.05
Sulfide	18496-25-8	SM4500S2-CF	NA	NA	1	1
Alkalinity	ALK	SM2320B	NA	NA	5	5
Stable Oxygen and Hydrogen Isotopes	NA	Picarro Cavity Ring-Down Spectroscopy	NA	NA	NA	NA

Stable Isotopes

Analyte	CAS Number	Method	Laboratory RL (µg/L)
Tetrachloroethene ($\delta^{13}C$)	127-18-4	Gas Chromatogram Isotope-Ratio Mass Spectrometry	1
cis-1,2-Dichloroethylene ($\delta^{13}C$)	156-59-2		1
trans-1,2-Dichloroethylene ($\delta^{13}C$)	156-60-5		1
Trichloroethylene ($\delta^{13}C$)	79-01-6		1
Vinyl chloride ($\delta^{13}C$)	75-01-4		2
Ethene ($\delta^{13}C$)	74-85-1		2
Groundwater ($\delta^{18}O$ and $\delta^{2}H$)	NA	Picarro Cavity Ring-Down Spectroscopy	NA

Molecular Biological Diagnostic Tools				
Analyte	**CAS Number**		**Method**	**Laboratory RL**
DHC 16S rRNA gene and functional genes tceA, bvcA, and vcrA	NA	NA	QuantArray and/or qPCR	500 cells per sample

[a] If an MCL is set for the analyte, the screening level is the MCL.

Notes:
bvcA = vinyl chloride reductase
CAS = Chemical Abstracts Service
DHC = Dehalococcoides spp.
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level
MDL = method detection limit
mg/L = milligrams per liter
NA = not applicable
qPCR = quantitative polymerase chain reaction
RL = reporting limit
rRNA = ribosomal ribonucleic acid
RSL = regional screening level
tceA = TCE reductase
TDS = total dissolved solids
TOC = total organic carbon
vcrA = vinyl chloride reductase

2.4.2 Data Quality Indicators and Quality Control Sample/Measurement Performance

This plan identifies procedures and criteria that will provide data of known and appropriate quality for the project objectives, specifically regulatory and screening levels detailed in **Tables 2-3** through **2-11**. Data quality is assessed using data quality indicators, specifically, parameters for precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS). These parameters, applicable procedures, and measurement performance criteria are described in the following paragraphs.

QC procedures, quantitative target limits, and other quality measures are dictated by the intended use of the data and nature of the analytical methods. Analytical precision, accuracy, and completeness requirements discussed herein align with the project quality objectives identified in Section 2.4.1.

To assess the quality of the data generated during this site investigation, data quality indicators (PARCCS) will be evaluated against project-specific measurement performance criteria (MPC).

Precision is the measure of variability among individual sample measurements under prescribed conditions. The relative percent difference (RPD) between primary and field duplicate (FD) samples, laboratory sample duplicate pairs, and matrix spike (MS)/matrix spike duplicate (MSD) sample results demonstrate the precision of the sample matrix. The level of effort for precision measurements will be a minimum of 1 in 10 samples for FDs and a minimum of 1 in 20 samples for MS/MSDs. Precision will be calculated from duplicate measurements as follows:

$$RPD = \frac{(C1-C2)}{(C1+C2) \div 2} \times 100\% \qquad (1)$$

where:

RPD	=	relative percent difference
C_1	=	larger of the two observed values
C_2	=	smaller of the two observed values

Accuracy is a measure of the closeness of a reported concentration to the true value. Accuracy is expressed as a bias (high or low) and is determined by calculating percent recovery (%R) from MS/MSDs and laboratory control samples (LCSs). MS/MSD recoveries (from field samples) indicate accuracy relevant to a unique sample matrix. LCS recoveries (including surrogate spikes) indicate accuracy relevant to an analytical batch lot and are strictly a measure of analytical accuracy conditions independent of samples and matrices. The %R of an analyte, and the resulting degree of accuracy expected for the analysis of QC spike samples, are dependent upon the sample matrix, method of analysis, and the compound or element being measured. For measurements where MSs are used:

$$R = 100\% \times \left[\frac{S-U}{Csa}\right] \qquad (2)$$

where:

%R	=	percent recovery
S	=	measured concentration in spiked aliquot

U	=	measured concentration in unspiked aliquot
C_{sa}	=	actual concentration of spike added

For situations where a standard reference material (SRM) is used instead of or in addition to MSs:

$$R = 100\% \times \left[\frac{Cm}{Csm}\right] \quad (3)$$

where:

%R	=	percent recovery
C_m	=	measured concentration of SRM
C_{sm}	=	actual concentration of SRM

Representativeness is a measure of how closely the results reflect the actual concentration or distribution of the chemical compounds in the matrix samples. Sampling plan design, sampling techniques, and sample-handing protocols (e.g., storage, preservation, transportation) were developed and are discussed in Section 3. Field inspections/oversight and field and laboratory records will document that protocols were followed, and sample identification and integrity were maintained.

Comparability expresses the confidence with which one data set can be compared to another. Data comparability will be maintained using defined procedures and the use of consistent methods and consistent units of measurement. Actual detection limits will depend on the sample matrix and will be reported as defined for the specific samples.

Completeness is a measure of the amount of valid data obtained from the analytical measurement system and the complete implementation of defined field procedures. The target completeness objective will be 90 percent; the actual completeness may vary depending on the intrinsic nature of the samples. Completeness is defined as follows for all measurements:

$$C = 100\% \times \left[\frac{V}{T}\right] \quad (4)$$

where:

%C	=	percent completeness
V	=	number of measurements judged valid
T	=	total number of measurements

Sensitivity is the capability of the method or instrument to discriminate between measurement responses representing different levels of the variable of interest. The lowest calibration standard can be used to evaluate the reported laboratory limits. The reported laboratory limits are compared to the project action limits and screening criteria to assess whether sensitivity is adequate for project needs.

2.5 Special Training/Certification (A8)

All project staff (including subcontractors) working on-site must be trained in health and safety, including 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) Training and annual 8-hour refresher training, and must follow requirements specified in the health and safety plan. Required training will be administered by the employing company of the personnel

working on-site and training documentation will be provided to the FTL on request. In addition, the FTL will have completed the 8-hour HAZWOPER Supervisor Training, and the HAPSITE operator will have completed the KD Analytical HAPSITE Basic and Advanced Operator Training. The health and safety plan describes the specialized training, including medical tracking and documentation, required for personnel on the project. The CDM Smith PM will confirm that all field staff have the appropriate training and certifications (if required) for their assigned tasks. Training records are maintained on-site and in CDM Smith's health and safety database. Certifications are verified as part of a readiness review before initiation of work. **Table 2-12** provides a summary of the training requirements for the project. If a designee is appointed for any project function, that designee will meet the training and certification requirements. The project staff is summarized in **Table 2-1**.

Table 2-12. Specialized Training Requirements

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Project Function	Specialized Training – Title or Description of Course	Training Date	Personnel/ Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
FTL	• 40-Hour HAZWOPER • 8-Hour HAZWOPER Supervisor Training • 8-Hour HAZWOPER Refresher	• 2005 • 2008 • Current	Neil Smith or designee	FTL	Certificates are available upon request; documentation files are maintained on-site and in CDM Smith's Health and Safety Database.
Field Staff (including subcontractors)	• 40-Hour HAZWOPER • 8-Hour HAZWOPER Refresher	• Current	All field staff	Field technicians, geologist, equipment operators, drillers, various	Certificates are available upon request; documentation files are maintained on-site and in CDM Smith's Health and Safety Database.
HAPSITE GC/MS Operator	• KD Analytical Training Certificate • 40-Hour HAZWOPER • 8-Hour HAZWOPER Refresher	• 2017 • 1991 • Current	Todd Burgesser or designee	HAPSITE Operator	Certificates are available upon request; documentation files are maintained on-site and in CDM Smith's Health and Safety Database.

FTL = field team leader

GC/MS = gas chromatograph/mass spectrometer

2.6 Documents and Records (A9)

Field documentation and records are described in SOPs 4-1, *Field Logbook Content and Control*, 4-2, *Photographic Documentation of Field Activities*, and 1-2, *Sample Custody* (**Appendix A**). Field documentation records and electronic files that will be generated during the RI include the following:

- Field logbooks
- Field forms
- Calibration records
- Photographs (electronic files)
- Chain-of-custody forms
- Global Positioning System (GPS) data (electronic files)

Field logbooks, field forms, calibration records, and chain-of-custody forms will be handwritten in the field, reviewed by the CDM Smith FTL or designee within 1 week of the field task completion, and scanned. Scanned field forms will be submitted to VHA, uploaded to the project file on Microsoft SharePoint, and included in technical memorandums/final reports that summarize field activities. Field logbooks, field forms, and photographs are discussed in SOP 4-1, *Field Documentation* and SOP 4-2, *Photographic Documentation of Field Activities;* chain-of-custody documentation is discussed in SOP 1-2, *Sample Custody* (**Appendix A**).

Laboratory documentation will follow method requirements and QA protocols listed in Section 3. Laboratory documentation will be retained for a minimum of 10 years after contract closeout. A complete list of the documents, records, and electronic files provided in the laboratory data package will include the following:

- Case narrative
- Summary results
- Chain of custody
- QC results
- Method summary
- Chromatograms

A detailed listing of the various required elements that will be included in the laboratory data package is provided in Section 2.6.1.

2.6.1 Analytical Data Package

An EPA Level IV Contract Laboratory Program (CLP)-equivalent data package will be provided by the project laboratories (except for laboratories providing stable isotope and geotechnical testing), with the following information (when applicable) for each data deliverable:

- Cover letter, complete with the following information:
  - Title of report and laboratory unique report identification
  - Project name and site location
  - Name and location of laboratory and second-site or subcontracted laboratory
  - Client name and address
  - Statement of authenticity and official signature and title of person authorizing report release
- Table of contents (paginated pages)
- Summary of samples received that correlates field sample identification (ID) with the laboratory ID
- Laboratory qualifier flags and definitions
- Field ID number
- Date received
- Date prepared
- Date analyzed (and time of analysis if the holding time is less than or equal to 48 hours)
- Preparation and run logs
- Analytical methods/SOPs used
- Result for each analyte (dry-weight basis for soils)
- Percent solids results for soil samples
- Dilution factor (both diluted and undiluted results, when available)
- Sample-specific RL adjusted for sample size, dilution/concentration
- Sample-specific MDL adjusted for sample size and dilution/concentration (project objectives require reporting to the MDL)
- Units
- Case narrative that addresses the following information, at a minimum:

  - Sample receipt discrepancies, such as bubbles in VOC samples and temperature exceedances
  - Descriptions of all nonconformances in the sample receipt, handling, preparation, analytical and reporting processes, and the corrective action taken in each occurrence
  - Identification and justification for sample dilution
- Surrogate percent recoveries
- MS/MSD and LCS spike concentrations, native sample results, spiked sample results, percent recoveries, and RPDs between the MS and MSD results; associated QC limits must also be provided
- Laboratory duplicate results
- Method blank (MB) results
- Analytical batch reference number that cross references samples to QC sample analyses
- Chain-of-custody form and sample receipt checklist
- Analytical sequence or laboratory run log that contains sufficient information to correlate samples reported in the summary results to the associated method QC information, such as initial and continuing calibration analyses
- The measured final residual vacuum of each sample canister measured with a digital meter immediately prior to analysis
- Confirmation results
- Calibration blank results for inorganic analyses
- Inductively Coupled Plasma Serial Dilution percent difference results
- Interference check sample true and measured concentrations and percent recoveries
- Method of standard addition results (if applicable)
- Postdigestion spike (PDS) recoveries (if applicable)
- Internal standard recovery and retention time information, as applicable
- Initial calibration (ICAL) summary, including standard concentrations, response factors, average response factors, RSDs or correlation coefficients, and calibration plots or equations, if applicable
- Continuing calibration verification (CCV) summary, including expected and recovered concentrations and percent differences
- Instrument tuning and mass calibration information, as applicable

- Any other method-specific QC sample results plus full supporting raw instrument data, records of standard preparation, manual integrations, example calculations, spectra, chromatograms, sample preparation logs (full, EPA Level IV data deliverable-equivalent)

2.6.2 Electronic Data Deliverable Format

Electronic data deliverables (EDDs) are required for all laboratory analytical data. An automated laboratory information management system must be used to produce the EDD from the same source as the data; manual creation of the deliverable (data entry by hand) is unacceptable. The EDD will correspond exactly to the hard copy data report. Laboratories will provide the data in accordance with the EDD specifications for EQuIS at earthsoft.com. All project analytical data will be uploaded to the EQuIS project database by the CDM Smith data manager for reporting and archiving.

Per Federal Acquisition Regulation (FAR) 52.215-2 (Audit and Records), electronic and PDF laboratory data must be retained by CDM Smith for a minimum of 10 years after contract closeout. The laboratory will use a storage device that is capable of recording data for long-term, offline storage. CDM Smith will also retain backup hard copies and pdfs of field and laboratory data in project files and electronic copies on an off-site file sharing network.

2.6.3 Project Information/Records Storage and Retention

CDM Smith will retain the entire project file as hard copy and/or electronic data on ProjectWise until the completion of all project activities or sent to VHA when requested. Thereafter, all data will be transferred to VHA, and CDM Smith will archive and maintain the hard copy data in a secure and protected facility and electronic data on ProjectWise for a minimum of 10 years after contract closeout, per FAR 52.215-2 (Audit and Records). Environmental data stored in the project EQuIS database, along with geographic information system (GIS) layers with their associated metadata, will be provided to USACE/VHA and archived by CDM Smith in electronic format for transmittal to EPA and the Utah Department of Environmental Quality (UDEQ), if requested.

Backup copies of electronic data, including pdf files of documents, will be stored in ProjectWise maintained by CDM Smith during the term of the contract, and at the end of the contract, transferred to an off-site data archive site.

The official administrative record for the site is located online at www.pceplume.org. A copy of the administrative record will be maintained at the VHA Salt Lake City Health Care System at 500 Foothill Drive in Salt Lake City, Utah 84148. Project reports, including the RIWP, FSPs, and QAPP, will be available in hard and electronic copies for the duration of the CERCLA project. EPA Region 8 in Denver, Colorado, and UDEQ in Salt Lake City, Utah, will receive all copies of documents, laboratory data, and other published project information for their records.

Section 3

Data Generation and Acquisition (EPA Group B)

This section describes the sampling design; sampling methods; sample handling and custody; analytical methods; QC; instrument/equipment testing, inspection, and maintenance; instrument/equipment calibration and frequency and inspection/acceptance of supplies and consumables; nondirect measurements; and data management.

3.1 Sampling Design (Experimental Design) (B1)

The rationale and sampling design to address Phase 2 DQOs is presented in Section 4.2 of the RIWP and Section 2 of the FSP (RIWP Appendix A). A summary of the ongoing Phase 1 RI tasks and anticipated Phase 2 tasks is presented in Table 4-2 of the RIWP, and proposed Phase 2 investigation locations are shown on Figures 2-1 through 2-4 of the FSP.

The key Phase 2 RI data collection activities to support the DQOs will include the following:

- Installing additional monitoring wells to evaluate the lateral and vertical extent of COPCs. The well installation during Phase 2 will focus on completion of transects to evaluate mass discharge within the plume and to delineate the extent of the PCE plume to the north and northwest within the ESS area. If the Phase 1 RI wells do not adequately delineate the plume along the Guardsman Way and 1400 East transects, additional step-out wells will be installed in these areas. Data collection will support DQOs E1 (Hydrogeologic Features), E2 (Plume Characterization), and E3 (Plume Mass Discharge).
- Collecting geophysical data from select wells to evaluate aquifer properties to support development of the groundwater flow model and fate and transport evaluation. Data collection will support DQO E1 (Hydrogeologic Features).
- Completing aquifer tests (pumping tests and/or slug tests) to measure hydraulic properties of the aquifer to support development of the groundwater flow model and fate and transport evaluation, and to support mass discharge evaluation. Data collection will support DQOs E1 (Hydrogeologic Features) and E3 (Plume Mass Discharge).
- Measuring water levels and calculating hydraulic gradients during synoptic water level measurement events and using transducers for continuous water level measurement at select locations. These data will support mass discharge estimation and fate and transport evaluation. Data collection will support DQOs E1 (Hydrogeologic Features), E2 (Plume Characterization), and E3 (Plume Mass Discharge).
- Sampling groundwater at existing and newly installed wells to evaluate VOC concentrations and aquifer geochemistry, evaluate VOC trends over time and plume stability, and support mass discharge estimation. Data collection will support DQOs E2 (Plume Characterization), E3 (Plume Mass Discharge), E4 (Natural Attenuation), D1 (Source Mass), and D3 (Groundwater Risk).

- Collecting groundwater samples from select wells for compound-specific isotopic analysis (CSIA) to evaluate attenuation of VOCs across the plume. Data collection will support DQO E4 (Natural Attenuation).
- Collecting subsurface soil data for total ferrous minerals analysis and magnetic susceptibility to support evaluation of abiotic attenuation mechanisms. Data collection will support DQO E4 (Natural Attenuation).
- Sampling surface water to aid in delineation of the PCE plume extent and to support risk assessment. Data collection will support DQOs E2 (Plume Characterization), D3 (Groundwater Risk), and D4 (Surface Water Risk).
- Collecting soil gas samples from vapor points installed during the Phase 1 investigation near Buildings 6/7 and Sunnyside Park to evaluate the lateral and vertical extent of PCE in the vadose zone, and from soil vapor points planned for installation during Phase 2 in the ESS area to evaluate areas where future VI sampling may be warranted. Data will also be used to evaluate whether PCE in the vadose zone is likely to act as a continuing source to groundwater. Data collection will support DQOs E2 (Plume Characterization), D1 (Source Mass), D2 (Source Area Vapor Intrusion Risk), and D3 (Groundwater Risk).
- Sampling indoor air and collecting other data to support VI evaluations will be conducted at approximately 20 homes at the Site, depending on the level of access granted by homeowners. Sampling will be conducted in accordance with the VI Protocol (CDM Smith 2019c), with additional provisions (e.g., no contact sampling) added to the protocol at a later date as necessary. In addition, replacement of select piezometers installed under AOU1 in the ESS area with shallow monitoring wells may be completed to provide additional data to inform future VI investigations. Data collection will support DQO D3 (Groundwater Risk).
- Surveying sample locations and wells.

Analytical methods to support the Phase 2 RI include:

- Contaminant analyses (VOCs, 1,4-dioxane, and total metals)
- General water quality and natural attenuation parameters (stable isotopes of hydrogen and oxygen, VOC, CSIA, TOC, total dissolved solids, chloride, sulfate, nitrate/nitrite, and alkalinity)
- Geotechnical (water content, hydraulic conductivity, and fraction organic carbon), and geochemical (magnetic susceptibility and ferrous iron minerals) parameters for soil
- Water quality field parameters (pH, oxidation-reduction potential [ORP], dissolved oxygen [DO], specific conductance, temperature, and turbidity)

3.2 Sampling Methods (B2)

Sampling methods specify how samples will be collected in terms of sampling equipment, sample volumes, containers, subsamples, preservation, and decontamination requirements. The sampling may include:

- Soil and groundwater samples collected during well installation
- Soil gas samples collected during source area investigation
- Indoor air samples collected by:
  - Passive sampling
  - Summa sampling
- Groundwater samples collected by:
  - Passive diffusion sampling
  - Low-flow groundwater sampling
  - Low-yield well sampling
  - Zone isolation sampling technology wells
- Surface water samples collected by grab sampling

Methods and protocols are described in further detail in Section 3 of the FSP (RIWP Appendix A), and proposed investigation locations are identified on Figures 2-1 through 2-4 of the FSP. Sampling SOPs are provided in **Appendix A**. The project schedule that identifies dates for planned investigation activities is presented in Table 6-1 of the RIWP, and critical data requirements and data acceptability is discussed in Table 4-1 of the RIWP and is included as Appendix B.

Changes to proposed well locations or planned tasks may be required based on information gathered from previous tasks. , A new location or new task, consistent with the project DQOs, will be presented in a minor field modification (MFM) to the RIWP. Workplan modifications are discussed in further detail in Section 4.6 of the RIWP.

3.3 Sample Handling and Custody (B3)

A sample is physical evidence collected from a hazardous waste site, from the immediate environment, or from another source. Because of the potential evidentiary nature of samples, VHA must have sample possession tracked from the time of sample collection until samples are disposed.

In addition to field notebooks, VHA requires CDM Smith to use a number of documents for tracking sample custody. Field documents include sample custody seals and chain-of-custody

records. CDM Smith will use chain-of-custody procedures to maintain and document sample collection and possession, as described in Section 3.3.3.

3.3.1 Sample Labeling

All sample labels will include, at a minimum, the following information:

- Sample name/number
- Time and date of sample collection
  - For all air samples, the end date is the date of collection
- Site name and location
- Project number
- Sample type and matrix
- Container
- Preservative, if applicable
- Analysis method

The process for labeling samples, as well as the sample nomenclature, is provided in further detail in Section 3.17 of the FSP.

3.3.2 Packaging and Shipping

Samples that require cooling to 6 degrees Celsius (°C) will immediately be placed on ice for temporary storage before and during shipment to the laboratory. All samples will be packaged and labeled for shipment in compliance with current U.S. Department of Transportation regulations. Samples will be shipped or hand delivered to the laboratory as soon as practicable.

Only metal or plastic ice chests will be used for shipping samples that require cooling. For shipment to the project laboratory, samples will be properly padded to prevent breakage. Ice used in the coolers will be placed in double, 1-gallon zip-lock bags, with a minimum amount of airspace. The chain-of-custody record will be placed inside a zip-lock bag and taped to the inside of the cooler top. The cooler will be closed and taped shut with packing tape or duct tape, and a signed and dated custody seal will be properly placed across two sides of the cooler lid. The shipping air bill, if needed, will be securely attached to the exterior of the cooler.

Samples not requiring cooling, such as Summa canister and passive air samples, will be packaged in the cardboard containers they were received in from the laboratory and shipped or delivered via courier to the laboratory directly after collection. The chain-of-custody record will be placed in one of the boxes.

Commercial carriers are not required to sign the chain-of-custody record if it is sealed inside the shipping cooler and the custody seals remain intact. The laboratories are responsible for storing the samples in a secure location and following chain-of-custody procedures.

Soil and water samples will be collected in certified clean containers provided by the project laboratory. Air samples for EPA Method Modified TO-15 SIM analysis will be collected in clean, certified Summa canisters (individually certified) with clean-flow controllers. Passive absorbent air samples for EPA Modified Method TO-17 will be collected with building-dedicated diffusive bodies and badges. These items are retained in labeled bags for potential future sampling at those same structures. The requirements for sample volumes, sampling containers, preservation, and holding times are summarized in **Table 3-1**.

Table 3-1. Recommended Sample Container, Preservation, and Holding Times
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Parameter	Method	Matrix	Holding Time (From Date Sampled)	Container	Preservative
Organics					
VOCs and 1,4-Dioxane	SW8260B/C	Soil	48 hours to preservation, Encore samplers 14 days from sample collection	4 × 5 grams Encore[b] 4 oz glass jar for moisture content	≤6°C
VOCs	SW8260C	Water	14 days	3 × 40 mL glass VOA vial[a,b]	≤6°C, HCl to pH<2
1,4-Dioxane	SW8270 SIM low	Water	7 days to extraction, 40 days to analysis	1 L wide mouth glass bottle	≤6°C
VOCs	Modified TO-15 SIM	Air	30 days	6 L Summa Canister	NA
VOCs	Radiello 130 Modified TO-17	Air	30 days	Passive Sampler	NA
VOCs	HAPSITE	Air	48 hours	Tedlar bag	NA
VOCs	HAPSITE	Water	7 days	3 × 40 mL glass VOA vial	≤6°C
Inorganic					
Metals (except Mercury)	SW6020A	Water	180 days	1 L polyethylene bottle	HNO_3 to pH<2
Metals (except Mercury)	SW6020A	Soil	180 days	8 oz wide mouth glass jar	NA
Mercury	SW7470A	Water	28 days	1 L polyethylene bottle	HNO_3 to pH<2
Mercury	SW7471A	Soil	28 days	8 oz wide mouth glass jar	NA
General Chemistry and Natural Attenuation Parameters					
Chloride, Nitrate/Nitrite, Sulfate	E300.0	Water	28 days, Nitrate/Nitrite 48 hours	1 L polyethylene bottle	≤6°C
Nitrate/Nitrite-N	SM4500-NO^3E	Water	28 days	125 mL polyethylene bottle	<6°C, H_2SO_4 to pH<2
Dissolved Gases	RSK-175	Water	14 days	3 × 40 mL glass VOA vial[a,b]	≤6°C, HCl to pH<2
Sulfide	SM4500S^2-CF	Water	7 days	250 mL polyethylene bottle	≤6°C, NaOH to pH>10; ZnAc
TDS	SM2540C	Water	7 days	1 L polyethylene bottle	≤6°C

Table 3-1. Recommended Sample Container, Preservation, and Holding Times
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Parameter	Method	Matrix	Holding Time (From Date Sampled)	Container	Preservative
TOC	SW9060	Water	28 days	250 mL wide mouth glass bottle	≤6°C, H_2SO_4 to pH<2
Alkalinity	SM2320B	Water	14 days	1 L polyethylene bottle	≤6°C
Stable Oxygen and Hydrogen Isotopes	Isotope Ratio Mass Spectroscopy	Water	180 days	2 × 20 mL glass vial	≤6°C
Stable Carbon Isotopes	CSIA	Water	30 days	3 x 40 mL VOA vial	≤6°C, HCl to pH<2
DHC 16S rRNA and functional genes tceA, bvcA, and vcrA	qPCR and/or QuantArray	Water	24 hours	1 L polyethylene bottle	≤6°C
Geotechnical					
FOC	ASTM D2974	Soil	28 days	4 oz wide mouth glass jar	≤6°C
Magnetic Susceptibility	Bartington System	Soil	NA	250 mL polyethylene jar	None
Ferrous Iron Minerals	SM3500-Fe B4c	Soil	6 months	250 mL polyethylene jar	None
Sieve Analysis	ASTM D6913	Soil	NA	5 gal bucket	None
Dry Bulk Soil Density	ASTM D2937	Soil	NA	Shelby Tube	None
Hydrometer	ASTM D7928	Soil	NA	1 gal bag	None
USCS Soil Classification	ASTM D2487	Soil	NA	1 gal bag	None
Atterberg Limits	ASTM D4318	Soil	NA	From USCS bag	None
Gradation	ASTM D1140	Soil	NA	5 gal bucket	None
Vertical Permeability	ASTM D2434	Soil	NA	Shelby Tube	None
Moisture Content	ASTM D2216	Soil	NA	1 quart bag	None

[a] Additional sample volume is required for selective ion matrix analysis.
[b] Additional sample volume is required for MS/MSD analysis.

Notes:
°C = degrees Celsius
> = greater than
< = less than
≤ = less than or equal to
CSIA = compound-specific isotopic analysis
DHC = Dehalococcoides spp.
FOC = fraction of organic carbon
gal = gallon
H_2SO_4 = sulfuric acid
HCl = hydrochloric acid
HNO_3 = nitric acid
H_2SO_4 = sulfuric acid
L = liters
mL= milliliters
NA = not applicable
NaOH = sodium hydroxide
oz = ounce
qPCR = quantitative polymerase chain reaction
rRNA = ribosomal ribonucleic acid
TDS = total dissolved solid
TOC = total organic carbon
USCS = Unified Soil Classification System
VOA = volatile organic analyte
ZnAc = zinc acetate

3.3.3 Chain of Custody

A chain-of-custody record establishes the documentation necessary to trace sample possession from time of collection through sample analysis and final disposition, and serves to document the specific analyses requested from the laboratory. A sample is in the custody of a person if any of the following criteria are met:

- The sample is in a person's physical possession
- The sample is in a person's view after being in his or her physical possession
- The sample was in a person's physical possession and was then locked up or sealed to prevent tampering
- The sample is kept in a secured area

Chain-of-custody forms will be developed electronically and printed. The sampling team members will fill out the date and time of sample collection (and all other pertinent information for air sampling) in the field. When shipping the samples, the sampler will sign the bottom of the form and enter the date and time (24-hour) when samples were relinquished. The sampler will enter the carrier name and air bill number on the form. Any required special handling of analyzed samples, such as hold or return, must be written on the chain-of-custody record. A second member of the field crew will review the completed chain-of-custody record to confirm that required information is not omitted. The original signature copy of the chain-of-custody record will be enclosed in a plastic bag and secured to the inside of the cooler lid or shipping container. A copy of the chain-of-custody record will be retained for project files.

3.3.3.1 Laboratory Custody Procedures

A designated sample custodian will accept the shipped samples and verify that the packing list sample numbers match those on the chain-of-custody records. Pertinent information regarding shipment, pickup, courier, and sample receipt information including temperature upon receipt at the laboratory will be entered in the section for remarks.

The laboratory sample custodian will reconcile the information on the chain-of-custody records with the information on the sample containers, as received and document any anomalies and report these to the laboratory PM. Anomalies will be resolved with the CDM Smith project chemist and FTL. The laboratory sample custodian will email a copy of the cooler receipt form, the associated chain-of-custody records, and the sample delivery group assignment form to the CDM Smith PM and project chemist within 24 hours of sample receipt. The information on the forms will then be entered into the laboratory information management system along with the analyses being requested. USACE/VHA will be notified of any anomalies that may interfere with sample integrity.

The laboratory sample custodian is responsible for seeing that all samples are transferred to the proper analyst or stored in the appropriate secure area. Laboratory personnel are responsible for the care and custody of samples from the time they are received until the sample is exhausted or returned to the custodian using an internal chain-of-custody record to track sample movement within the laboratory. When CDM Smith has received all data and determines the analyses and

data packages to be complete, the unused portion of the sample will be properly disposed by the laboratory. All identifying stickers, data sheets, and laboratory records are retained as part of the laboratory documentation requirements listed in Section 2.6. Sample containers and remaining samples will be disposed of in compliance with all federal, state, and local regulatory requirements.

3.3.4 Custody Seals

Custody seals will be placed on coolers or shipping containers during transport of samples to the laboratory. The seals will be placed on two sides of the lid (one in front, and one on the side) and covered with tape to prevent inadvertent breaking of the seals. To prevent the opening of coolers during shipment and to confirm that the samples remain sealed under custody until arrival at the laboratory, an additional large liner bag (drum liner type) will be placed inside around the entire contents of the cooler and sealed tightly closed.

3.3.5 Field Notebooks and Field Documentation

Field notebooks/logbooks are hardbound notebooks with preprinted sequentially numbered pages in which all activities associated with the field investigation will be thoroughly described. Field notebooks are intended to provide sufficient information to reconstruct events occurring during the field project. SOP 4-1, *Field Logbook Content and Control* contains further information. General information regarding sampling activities will be recorded and includes, at a minimum, the following:

- Name and company affiliation of the person completing the entry
- Weather conditions during the field activity
- Name and affiliation of all personnel or visitors on-site
- Equipment on-site
- Stop and start times for sampling activities at each location
- Summary of daily activities, including information presented at the daily safety meeting
- Descriptions of deviations from the FSP or QAPP
- Description of any problems encountered during sampling at each location
- Other miscellaneous information that may be applicable to conditions encountered

In general, the various field documents are considered complementary in nature and of equal importance. However, in the event of direct conflicts or discrepancies among these documents, interpretations will generally be based on the following priorities:

1. Requirements for field documents, contained in contract documents and/or project documents (e.g., work plans, this QAPP). Priority will be given to the most recent version of these documents or amendments to these documents.

2. Field documents chronologically filled out first, and from which data may be used in subsequent documents. For example, if a field form was used to record a water level that was subsequently also recorded in a field logbook.

3. Forms that were developed to capture specific information have precedence over documents, such as field logbooks, which are more general in nature.

There is not a rigid order of precedence for documents, as any discrepancies between documents will need to be evaluated on a case-by-case basis using best practices and professional judgement. However, the items noted can be used as guidelines.

3.3.6 Corrections to Documentation

All original data recorded in field notebooks, sample identification tags, chain-of-custody records, and receipts-for-sample forms will be written with waterproof ink, unless prohibited by weather conditions. No accountable, serialized documents will be destroyed or disposed of, even if they are illegible or contain inaccuracies that require a correction or a replacement document.

If an error is made on an accountable document assigned to one team, the staff may make corrections simply by drawing a single line through the error, then entering the correct information. The erroneous information should not be obliterated. Any subsequent error discovered on an accountable document should be corrected by the person who made the entry. All corrections must be initialed and dated with the date of correction by the person making the correction.

3.4 Analytical Methods (B4)

An analytical method identifies the procedures used to analyze samples and their required performance standards or criteria. This subsection contains brief descriptions of the field and laboratory analytical methods that will be used to analyze various media including soil, air, surface water, and groundwater samples.

3.4.1 Field Analytical Methods

Parameters measured in the field during surface water and groundwater sample collection include pH, specific conductance, turbidity, DO, ORP, and temperature. In addition, field screening for VOCs in air and/or groundwater will be completed using a portable GC/MS (HAPSITE), photoionization detector (PID), or ColorTec. Field analytical methods will be performed in accordance with the SOPs and manufacturer's operating instructions provided in **Appendix A**.

3.4.1.1 Specific Conductance

The specific conductance of a sample will be measured by using a meter equipped with a flow-through cell or sample cup. Routine calibration (i.e., daily, before each work shift) will be performed with commercially prepared solutions according to the recommended manufacturer's calibration instructions. These solutions will not be used past the expiration date printed on the label and replaced with new solutions at least once per week.

3.4.1.2 Turbidity

The turbidity of a sample will be measured by using a meter equipped with a flow-through cell or sample vial. Routine calibration (i.e., daily, before each work shift) will be performed with commercially prepared solutions according to the recommended manufacturer's calibration instructions. These solutions will not be used past the expiration date printed on the label and replaced with new solutions at least once per week.

3.4.1.3 pH

The pH of a sample will be measured by using a meter equipped with a flow-through cell or sample vial. Routine calibration (i.e., daily, before each work shift) will be performed with commercially prepared solutions according to the recommended manufacturer's calibration instructions. These solutions will not be used past the expiration date printed on the label and replaced with new solutions at least once per week. These solutions are certified with an accuracy of plus or minus 0.01 pH unit at a specific temperature, usually 25°C.

3.4.1.4 Dissolved Oxygen

The DO of a sample will be measured by using a meter equipped with a flow-through cell or sample vial. The team will follow the manufacturer's specific instructions for the calibration, operation, and maintenance of the DO meter. The DO meter will be air calibrated daily in water-saturated air.

3.4.1.5 Oxidation-Reduction Potential

The ORP of a sample will be measured by using a meter equipped with a flow-through cell or sample vial. Routine calibration (i.e., daily, before each work shift) will be performed with commercially prepared solutions according to the recommended manufacturer's calibration instructions. These solutions will not be used past the expiration date printed on the label and replaced with new solutions at least once per week.

3.4.1.6 Temperature

The team will follow the manufacturer's specific instructions for the measurement of temperature using the multimeter.

3.4.1.7 Field Screening PID Method

The PID will be used to screen total concentrations of VOCs in air, in accordance with the SOP 1-10 *Field Measurement of Total Organic Vapors* provided in **Appendix A**. The PID will be calibrated daily with a certified standard of isobutylene that will not be used past the expiration date printed on the label.

3.4.1.8 Field Screening ColorTec Method

The ColorTec method will be used to screen total concentrations of VOCs in water, in accordance with the manufacturer's SOP provided in **Appendix A**.

3.4.1.9 Field Screening Portable GC/MS Method

A portable GC/MS will be used to measure concentrations of individual VOCs using EPA Method TO-15 as guidance for screening-level analyses of air or headspace for purged groundwater samples. The portable GC/MS will be calibrated daily in accordance with the SOP *VOC Analysis by*

HAPSITE GCMS – Water and Vapor provided in **Appendix A**. Collection of air samples with Tedlar bags and the use of the headspace sampling system for screening-level groundwater analysis using the HAPSITE is also described in SOP *VOC Analysis by HAPSITE GCMS – Water and Vapor*. HAPSITE method files and data files will be retained in the project files. Project analytes, methods, and target detection limits are provided in **Table 2-6,** and quality control measurement is provided in **Table 3-2**.

3.4.2 Laboratory Analytical Methods

The laboratories will follow EPA or other industry standard applicable methodologies. Laboratory QA manuals and SOPs are provided in **Appendices C** and **D**, respectively. Laboratory manuals and SOPs are referenced but not provided for geotechnical analyses, as they are standard ASTM methods. Additionally, laboratory QA manuals and SOPs are not provided for methods that only provide screening-level data.

The turnaround time for laboratory analysis will be 21 calendar days from sample receipt, unless otherwise stated in the associated FSP.

Project analytes, methods, and target laboratory detection limits are listed in **Tables 2-2** through **2-10**.

The following methods will be used for the project:

- **VOA – SW-846 Method 8260B, GC/MS.** The VOC GC/MS technique is used to quantitate most VOCs with boiling points below 200°C. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.
- **VOA – EPA Method Modified TO-15 SIM, GC/MS.** Volatile organics in air are collected in an evacuated Summa canister and analyzed using the GC/MS technique.
- **VOA – EPA Method Radiello 130 Modified TO-17, GC/MS.** Volatile organics in air are collected on sorbent tubes, which are desorbed and cryo-focused on the capillary column and analyzed by GC/MS.
- **Metals – SW-846 Method 6020A, Inductively Coupled Plasma – Mass Spectrometry.** This method allows the multi-elemental determination of metals by transporting ions produced in the plasma into spectrometer. Interferences must be assessed, and valid corrections must be applied.
- **Mercury – SW-846 Methods 7470A/7471A, Cold Vapor Atomic Absorption.** This technique is based on absorption by mercury vapor. Mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration.
- **1,4-Dioxane – SW-846 Method 8270 SIM low/8260B SIM, GC/MS.** The GC/MS technique is used to quantitate organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-

silica capillary column coated with a slightly polar silicone. A deuterated internal standard is also used during analyses.

- **Stable Oxygen and Hydrogen Isotopes.** Groundwater is analyzed using an autosampler and Picarro Cavity Ring-Down Spectroscopy system to determine $\delta^{18}O$ and $\delta^{2}H$ ratios.
- **Stable Carbon Isotopes (CSIA).** Groundwater is analyzed using an autosampler and thermo gas chromatograph-isotope ratio mass spectrometer system to determine $\delta^{13}C$ compound-specific ratios of VOCs.
- **Dissolved Gases (Methane, Ethane, Ethene) – Method RSK-175, GC/Flame Ionization Detector.** Dissolved gases in water are sampled and analyzed using GC coupled to a flame ionization detector that is able to detect methane, ethane, and ethene at low parts-per-billion levels.
- **TOC – SW-846 Method 9060, Carbonaceous Analyzer.** Organic carbon in samples of groundwater is converted to either carbon dioxide and measured with an infrared detector, or to methane and measured with a flame ionization detector.
- **Anions – EPA Method 300.0, Ion Chromatograph.** Common anions in water are determined by injecting a small portion of sample into the ion chromatograph equipped with a conductivity detector.
- **Nitrate/Nitrite-N – Standard Methods SM4500-NO^3E, Cadmium Reduction.** The nitrite, original and reduced, is determined by diazotizing with sulfanilamide/N-(1-naphthyl)-ethylenediamine dihydrochloride solution, which forms a highly colored azodye that is then measured colorimetrically.
- **Alkalinity – Standard Methods 2320B, Titrimetric.** Alkalinity is determined by titrating a portion of the sample to an endpoint of pH 4.5 using HCl.
- **Total Dissolved Solids – Standard Methods 2540C, Gravimetric.** A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C.
- **Total Sulfide – Standard Methods 4500S2-C/F, Iodometric Titration.** The sample is pretreated to remove interfering substances. The sample is titrated with sodium thiosulfate until disappearance of elementary iodine indicates the end point.
- **Fraction of Organic Carbon – ASTM D2974.** The fraction of organic carbon is measured by drying the sample at 440°C.
- **Ferrous Iron Mineral SM3500-Fe B4c.** Ferrous iron in soil is brought into solution by boiling with acid and hydroxylamine. The solution is treated with 1,10-phenanthroline, which chelates with ferrous iron to form an orange-red complex that is analyzed by spectroscopy. This method may be performed by CDM Smith.

- **Magnetic Susceptibility.** A Bartington magnetic susceptibility system (MS3 Meter and MS2B Sensor) is used to assess the magnetic susceptibility of soil samples. This method may be performed by Microbial Insights or by CDM Smith, using the manufacturer's SOP provided in **Appendix A**.
- **Sieve Analysis – ASTM D6913.** Particle size measurement for particles larger than fine-grained (silt/clay) soils.
- **Dry Bulk Soil Density – ASTM D2937.** Measurement of in situ dry density, used to calculate porosity and other geotechnical parameters.
- **Hydrometer – ASTM D7928.** Measurement of particle size of fine-grained (silt/clay) particles.
- **USCS Soil Classification – ASTM D2487.** Standard soil classification.
- **Atterberg Limits – ASTM D4318.** Measurement of the plasticity of soil, required for the USCS.
- **Gradation – ASTM D1140.** Test to measure percent of total soil sample that is silt/clay-sized, by wet (washing) sieve.
- **Vertical Permeability – ASTM D2434.** Measurement of permeability on an undisturbed soil sample, using a constant-head method.
- **Moisture Content – ASTM D2216.** The gravimetric (weight/weight) percent of water in a soil sample.

3.5 Quality Control (B5)

The QC samples and procedures document compliance with objectives or demonstrate the need for corrective action. Checks controlling field activities (e.g., sample collection, shipping) and checks controlling laboratory activities (e.g., digestion, analysis) are used. These checks are discussed in the following subsections. The number of QC samples associated with each sample medium, and details regarding their collection, are provided in the **Table 3-2** and **Table 3-3.** Decontamination procedures for sampling equipment is discussed in Section 3.14 of the FSP (RIWP Appendix A).

3.5.1 Field Quality Control Procedures

The QC samples are collected in the field and used to evaluate the validity of the field sampling effort. Field QC samples are collected for laboratory analysis when appropriate to check sampling and analytical precision, accuracy, and representativeness. With the exception of FDs for FOC, ferrous iron minerals, and magnetic susceptibility, field QC samples will not be collected for any soil geotechnical/geochemical analyses. Field QC samples for stable isotope samples will be limited to FDs. The following subsections discuss the types and purposes of field QC samples that will be collected.

3.5.1.1 Field Blanks

Field blanks are used to evaluate potential for ambient background cross contamination of sampled groundwater and/or air. For groundwater field blanks, ASTM Type II water (purchased and certified from a commercial vendor) will be poured directly from its original container into laboratory-supplied sample containers for VOC and 1,4-dioxane analysis. For air field blanks, the passive diffusive body is not removed from the vial but the sample is handled like an environmental sample and returned to the laboratory for analysis by EPA Method Modified TO-17. Field blanks will be collected at a frequency of 1 per sampling event.

3.5.1.2 Equipment Rinsate Blanks

Equipment rinsate blanks are used to evaluate sampling device cleanliness. Rinsate blanks are collected after a nondedicated sample collection device is subjected to standard decontamination procedures. ASTM Type II water (purchased and certified from a commercial vendor) will be poured over or through the sampling device and collected in a sample container for analysis. Equipment rinsate blanks will be collected at a frequency of 1 per day.

3.5.1.3 Trip Blanks

Trip blanks are used to assess the potential introduction of contaminants during the transportation and storage procedures. The trip blank consists of a VOA sample vial filled in the laboratory with ASTM Type II reagent grade or organic-free water, transported to the sampling site, handled like an environmental sample, and returned to the laboratory for analysis. Trip blanks are not opened in the field. Each cooler of groundwater samples sent to the laboratory for analysis of VOCs will contain one trip blank. Each shipment of passive absorbent air samples will contain one trip blank. The passive diffusive body is not removed from the vial in the field, is handled like an environmental sample, and returned to the laboratory for analysis by EPA Method Modified TO-17. If all passive air samples from the sampling event are sent in one shipment, the field blank will count as the trip blank. When an analyte is detected in the trip blank, the appropriate flag will be applied to all VOC sample results for samples packed in the cooler with the affected trip blank. No trip blanks will be collected for soil or air samples.

3.5.1.4 Field Duplicates

An FD is collected at the same time and from the same source as the original field sample, but submitted to the laboratory as a separate sample to assess the consistency of the overall sampling and analytical system. Water, soil, and air FDs will be collected and analyzed on a 10 percent basis for all analytical laboratory methods, or a minimum of 1 per sampling event if fewer than 10 samples are collected. FDs will be collected, numbered, packaged, and sealed in the same manner as other samples, and submitted blind to the laboratories.

3.5.1.5 Temperature Blank

All coolers with samples that require preservation to 6°C will contain at least one temperature blank. The temperature blank should be a polyethylene bottle filled with water and placed in a representative position inside the cooler. Each vial will be clearly marked TEMPERATURE BLANK. If the temperature blank is positioned inappropriately or is not representative of the cooler temperature measurement, the laboratory will document the deficiency and notify the CDM Smith PM.

3.5.2 Laboratory Quality Control Procedures

Laboratory QC checks are designed to determine the precision and accuracy of the analysis, to demonstrate the absence of interference and contamination from glassware and reagents, and to allow data comparability. Laboratory QC checks consist of MBs, LCSs, MSs, MSDs, and laboratory duplicates. In addition, PDS, serial dilution, and interference check samples will be evaluated for metals analysis only, and surrogates and internal standards (ISs) will be evaluated for those methods where applicable (e.g., organic methods only). The laboratory will also complete ICALs and continuing calibration checks according to specified analytical methods. Each QC check and their frequencies are discussed in the following subsections. Precision and accuracy goals for laboratory analytical methods are listed in **Tables 3-2** through **3-12**.

3.5.2.1 Method Blanks

MBs are designed to detect contamination of the field samples in the laboratory environment. MBs verify that interferences caused by contaminants in solvents, reagents, glassware, or in other sample processing hardware are known and minimized. The MB will be ASTM Type II water (or equivalent) for water samples and a clean soil matrix (such as Ottawa sand) for soil samples. If the blank contaminant concentration is not less than the reporting limit, the source of contamination will be identified, and corrective action will be taken before sample reanalysis. Analytical data will not be corrected for the presence of analytes in blanks. Analytical results for each sample will be clearly associated with a particular MB. MBs are performed per laboratory batch per analytical method.

3.5.2.2 Matrix Spikes/Matrix Spike Duplicates

MS/MSDs are aliquots of a field sample spiked with known concentrations of all target analytes. The spiking occurs during sample preparation and before analysis. Each analyte in the MS/MSD will be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. Collection of a volume of sample adequate for running analyses in triplicate is required for MS/MSDs. Therefore, the MS/MSD will be designated on the chain-of-custody documentation.

A minimum of one MS/MSD will be designated by the CDM Smith FTL for each site and included for every 20 field samples, for applicable methods. MS/MSDs are not performed for EPA Method Modified TO-15 and EPA Method Modified TO-17.

3.5.2.3 Laboratory Duplicates

A laboratory duplicate sample is a second and separate analysis of a field sample used to determine the precision of the analytical method relative to the sample matrix. Laboratory duplicate samples may be analyzed with or without an MS/MSD sample, depending on the laboratory SOP.

3.5.2.4 Postdigestion Spike

A PDS is a portion of the sample digestate that is fortified with known quantities of compounds of interest. The PDS is used to measure either positive or negative interferences that may distort the accuracy of the reported values in the native sample. Accuracy of the analytes should be within 75 to 125 percent of the known concentration added. PDSs are only evaluated for metals analyses and only performed upon failure of the MS/MSD recoveries to determine if failures were because of digestion or matrix conditions.

3.5.2.5 Serial Dilution

A 1 to 5 serial dilution is performed on a portion of the sample digestate and analyzed. The serial dilution is used to measure either positive or negative interferences that may distort the precision of the reported values in the native sample. Precision is expressed in terms of the %D between the original sample and the serial dilution results. The %D criterion should be less than 10 percent if the concentration of the analyte in the original sample is greater than 50 times the MDL. Serial dilutions are only evaluated for metals analyses.

3.5.2.6 Interference Check Sample

The interference check samples are used in inductively coupled plasma analyses (e.g., metals analyses) to verify background and interelement correction factors. They consist of two samples: A and B. Sample A contains the interfering analytes, and Sample B contains both the analytes of interest and the interfering analytes. Both samples are analyzed at the beginning and at the end of each analytical sequence. When the interference check samples results are outside the method-specified control limits, corrective action must be taken, including sample reanalysis, if appropriate.

3.5.2.7 Laboratory Control Samples

LCSs are blank spikes made from clean laboratory-simulated matrices spiked with known concentrations of all target analytes of interest. The LCS is carried through the complete sample preparation and analysis procedure. LCSs are designed to check the instrument and method accuracy. An LCS will be analyzed with every analytical batch. If corrective action specifies re-extraction and reanalysis, all samples associated with the out-of-control LCS must be re-extracted and reanalyzed after control is reestablished. All re-extraction and reanalysis must be performed within the sample holding times. If corrective action was unsuccessful or not performed, the data will be qualified for the affected analytes.

3.5.2.8 Surrogates

Surrogates are compounds similar to the target analytes in chemical composition and behavior in the analytical process but not normally found in environmental samples. Surrogates are used to evaluate accuracy, method performance, and extraction efficiency for organic methods only. Surrogates will be added to environmental samples, controls, and blanks, in accordance with the method requirements.

3.5.2.9 Internal Standards

ISs are compounds added to every GC/MS standard, MB, LCS/laboratory control sample duplicate (LCSD), MS/MSD, and sample extract at a known concentration before instrument analysis for organic methods only. They are used to quantify the target analytes. ISs provide for stable sensitivity and response during every analytical run. An IS is used to evaluate the efficiency of the sample introduction process and monitor the efficiency of the analytical procedure for each sample matrix encountered.

Table 3-2. Quality Control Samples for Field Screening of Volatile Organic Compounds in Air and Groundwater (HAPSITE)

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Daily Calibration Check Standard	Daily, before sample analysis	%R = 70–130%	Rerun, investigate source of problems. Recalibrate instrument if rerun does not meet QC acceptance limits.	Field Technician	Accuracy	%R = 70–130%
System Blank	Daily, before sample analysis	Target analytes: <RL	Rerun, maintenance.	Field Technician	Accuracy – Contamination	Target analytes: <RL
IS	Each field and QC sample	IS area -50% to +100% compared to IS from Daily Calibration Check Standard IS RT window ±0.5 minutes compared to CCV RT	Reanalyze affected samples. If similar results, report both runs. Flag data.	Field Technician	Accuracy	IS area -50% to +100% compared to IS from Daily Calibration Check Standard IS RT window ±0.5 minutes compared to CCV RT

Notes:
< = less than
± = plus or minus
% = percent
%R = percent recovery
CCV = continuing calibration verification
IS = internal standard
QC = quality control
RL = reporting limit
RT = retention time

Table 3-3. Quality Control Samples (Field and Laboratory) for Volatile Organic Compounds in Soil and Water
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	One per prep batch of 20 or fewer samples of similar matrix	Target analytes <50% RL except for common laboratory contaminants <RL	Investigate source of contamination. Rerun MB. No samples may be run until an acceptable MB has been run.	Laboratory Supervisor	Accuracy	Target analytes <50% RL except for common laboratory contaminants <RL
LCS	One per batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-4	Reprepare sample and reanalyze if recovery is high and sample is <RL narrate.	Laboratory Supervisor	Accuracy	Recovery within QC limits as defined in Table 3-4
MS	One per batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-4	If recoveries are outside limits and surrogate criteria are met, note in narrative. If both the surrogate and MS/MSD are unacceptable, reprepare the samples and QC. Check standard prep. Flag data.	Laboratory Supervisor	Accuracy	Recovery within QC limits as defined in Table 3-4
MSD	One per batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-4	Same as MS.	Laboratory Supervisor	Accuracy and Precision	Recovery within QC limits as defined in Table 3-4
IS	Each field and QC sample	IS area -50% to +100% compared to IS from CCV IS RT window ±0.5 minutes compared to CCV RT	Reanalyze affected samples. If similar results, report both runs. Flag data.	Laboratory Supervisor	Accuracy	IS area -50% to +100% compared to IS from CCV IS RT window ±0.5 minutes compared to CCV RT
Surrogate Standard	Every sample	All surrogate recoveries within laboratory specified QC limits	If sample volume available, reanalyze. Report both if second successful analysis is outside hold time or both fail QC criteria. Flag data.	Laboratory Supervisor	Accuracy	All surrogate recoveries within laboratory specified QC limits
FD	One per 10 per matrix	RPD <30% (aq), <50% (solids) for compounds with concentration >RL	Evaluate batch precision, other FD results. Note in validation report.	Data Validator	Precision	RPD <30% (aq), <50% (solids) for compounds with concentration > 5 or 2x RL

Table 3-3. Quality Control Samples (Field and Laboratory) for Volatile Organic Compounds in Soil and Water
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
EB	Collected daily with nondedicated sampling equipment	Results <RL	Evaluate sample results; flag data as necessary review decontamination procedures.	Data Validator	Accuracy	Results <RL unless target analytes in field samples are >10× those in EB
Trip Blank	One per shipment aqueous VOCs	Results <RL	Evaluate sample results; flag data as necessary.	Data Validator	Accuracy	Results <RL
Field Blank	One per sampling event	Results <RL	Evaluate sample results; flag data as necessary.	Data Validator	Accuracy	Results <RL

Notes:
< = less than
> = greater than
% = percent
± = plus or minus
aq = aqueous
CCV = continuing calibration verification
EB = equipment blank
FD = field duplicate
IS = internal standard
MS = matrix spike
MSD = matrix spike duplicate
QC =quality control
RL = reporting limit
RPD = relative percent difference
RT = retention time
SOP = standard operating procedure
VOC = volatile organic compound

Table 3-4. Precision and Accuracy for Volatile Organic Compounds in Soil and Water

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD %R[a]	Soil LCS %R[a]	RPD[b]	Water MS/MSD %R[a]	Water LCS %R[a]	RPD[b]
1,1,1-Trichloroethane	SW8260C	73–125	73–125	30	74–131	74–131	20
1,1,2,2-Tetrachloroethane	SW8260C	70–124	70–124	30	71–121	71–121	20
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	SW8260C	66–136	66–136	30	70–136	70–136	20
1,1,2-Trichloroethane	SW8260C	78–121	78–121	30	80–119	80–119	20
1,1-Dichloroethane	SW8260C	76–125	76–125	30	77–125	77–125	20
1,1-Dichloroethene	SW8260C	70–131	70–131	30	71–131	71–131	20
1,2,3-Trichlorobenzene	SW8260C	66–130	66–130	30	69–129	69–129	20
1,2,4-Trichlorobenzene	SW8260C	67–129	67–129	30	69–130	69–130	20
1,2,4-Trimethylbenzene	SW8260C	75–123	75–123	30	76–124	76–124	20
1,3,5-Trimethylbenzene	SW8260C	73–124	73–124	30	75–124	75–124	20
1,2-Dibromo-3-Chloropropane	SW8260C	61–132	61–132	30	62–138	62–138	20
1,2-Dibromoethane	SW8260C	78–122	78–122	30	77–121	77–121	20
1,2-Dichlorobenzene	SW8260C	78–121	78–121	30	80–119	80–119	20
1,2-Dichloroethane	SW8260C	73–128	73–128	30	73–128	73–128	20
1,2-Dichloropropane	SW8260C	76–123	76–123	30	78–122	78–122	20
1,3-Dichlorobenzene	SW8260C	77–121	77–121	30	80–119	80–119	20
1,4-Dichlorobenzene	SW8260C	75–120	75–120	30	79–118	79–118	20
2-Butanone (Methyl Ethyl Ketone)	SW8260C	51–148	51–148	30	56–143	56–143	20
2-Hexanone	SW8260C	53–145	53–145	30	57–139	57–139	20
4-Methyl-2-pentanone	SW8260C	65–135	65–135	30	67–130	67–130	20
Acetone	SW8260C	36–164	36–164	30	39–160	39–160	20
Benzene	SW8260C	77–121	77–121	30	79–120	79–120	20
Bromochloromethane	SW8260C	78–125	78–125	30	78–120	78–120	20
Bromodichloromethane	SW8260C	75–127	75–127	30	79–125	79–125	20
Bromoform	SW8260C	67–132	67–132	30	66–130	66–130	20
Bromomethane	SW8260C	53–143	53–143	30	53–141	53–141	20
Carbon Disulfide	SW8260C	63–132	63–132	30	64–133	64–133	20
Carbon Tetrachloride	SW8260C	70–135	70–135	30	72–136	72–136	20

Table 3-4. Precision and Accuracy for Volatile Organic Compounds in Soil and Water

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD %R[a]	Soil LCS %R[a]	RPD[b]	Water MS/MSD %R[a]	Water LCS %R[a]	RPD[b]
Chlorobenzene	SW8260C	79–120	79–120	30	82–118	82–118	20
Chloroethane	SW8260C	59–139	59–139	30	60–138	60–138	20
Chloroform	SW8260C	78–123	78–123	30	79–124	79–124	20
Chloromethane	SW8260C	50–136	50–136	30	50–139	50–139	20
cis-1,2-Dichloroethene	SW8260C	77–123	77–123	30	78–123	78–123	20
cis-1,3-Dichloropropene	SW8260C	71–130	74–126	30	73–127	73–127	20
Dibromochloromethane	SW8260C	74–126	74–126	30	74–126	74–126	20
Dichlorodifluoromethane (Freon 12)	SW8260C	29–149	29–149	30	32–152	32–152	20
Ethylbenzene	SW8260C	76–122	76–122	30	79–121	79–121	20
Isopropylbenzene	SW8260C	68–134	68–134	30	72–131	72–131	20
Methyl Acetate	SW8260C	53–144	53–144	30	56–136	50–136	20
Methyl Tert-Butyl Ether	SW8260C	73–125	73–125	30	71–124	71–124	20
Methylene Chloride	SW8260C	70–128	70–128	30	74–124	74–124	20
m,p-Xylene	SW8260C	77–124	77–124	30	80–121	80–121	20
o-Xylene	SW8260C	77–123	77–123	30	78–122	78–122	20
Styrene	SW8260C	76–124	76–124	30	78–123	78–123	20
Tetrachloroethene	SW8260C	73–128	73–128	30	74–129	74–129	20
Toluene	SW8260C	77–121	77–121	30	80–121	80–121	20
trans-1,3-Dichloropropene	SW8260C	71–130	71–130	30	73–127	73–127	20
trans-1,2-Dichloroethene	SW8260C	74–125	74–125	30	75–124	75–124	20
Trichloroethene	SW8260C	77–123	77–123	30	79–123	79–123	20
Trichlorofluoromethane (Freon 11)	SW8260C	62–140	62–140	30	65–141	70–136	20
Vinyl Acetate	SW8260C	50–151	50–151	30	54–146	54–146	20
Vinyl Chloride	SW8260C	56–135	56–135	30	58-137	58–137	20

[a] LCS and MS/MSD limits are based on DoD QSM Version 5.1, Appendix C (DoD 2017) and/or the laboratory limit, if required.
[b] RPDs are applicable to both MS/MSDs and LCSs.

Notes:
% = percent LCSD = laboratory control sample duplicate
%R = percent recovery MS/MSD = matrix spike/matrix spike duplicate
DoD = U.S. Department of Defense QSM = quality systems manual
LCS = laboratory control sample RPD = relative percent difference

Table 3-5. Quality Control Samples (Field and Laboratory) for 1,4-Dioxane in Soil and Water
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	One per prep batch of 20 or fewer samples of similar matrix	Target analytes <50% RL except for common laboratory contaminants <RL	Investigate source of contamination. Rerun MB. If MB still fails acceptance limits, reprepare/reanalyze MB and associated samples. No samples may be run until an acceptable MB has been run.	Laboratory Supervisor	Accuracy	Target analytes <50% RL Except for common laboratory contaminants <RL unless target analytes in field samples are >10× those in MB
LCS	One per batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-6	Reprepare sample and reanalyze if recovery is high and sample is <RL narrate.	Laboratory Supervisor	Accuracy	Recovery within QC limits as defined in Table 3-6
MS	One per batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-6	If recoveries are outside limits and surrogate criteria are met, note in narrative. If both the surrogate and MS/MSD are unacceptable, reprepare the samples and QC. Check standard prep. Flag data.	Laboratory Supervisor	Accuracy	Recovery within QC limits as defined in Table 3-6
MSD	A minimum of one per batch or one per 20 per matrix	Recovery within QC limits as defined in Table 3-6	Same as MS.	Laboratory Supervisor	Accuracy and Precision	Recovery within QC limits as defined in Table 3-6
IS	Each field and QC sample	IS area -50% to +100% compared to IS from CCV IS RT window ±0.5 minutes compared to CCV RT	Reanalyze affected samples. If similar results, report both runs. Flag outliers.	Laboratory Supervisor	Accuracy	IS area -50% to +100% compared to IS from CCV IS RT window ±0.5 minutes compared to CCV RT

Table 3-5. Quality Control Samples (Field and Laboratory) for 1,4-Dioxane in Soil and Water
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Surrogates	Every Sample	Recoveries within laboratory specified QC control limits;	If sample volume available, re-extract. Report both if second successful analysis is outside Holding Time or both fail QC criteria. Flag data.	Analyst, Supervisor, Laboratory QA Manager	Accuracy	All surrogate recoveries within laboratory specified QC control limits; one acid-extractable compound, one base-neutral compound can be out if >10%
FD	One per 10 per matrix; minimum of one per matrix	RPD <30% (aq), <50% (solid) for compounds with concentration >RL	Evaluate batch precision, other FD results. Note in validation report.	Data Validator	Precision	RPD <30% (aq), <50% (solid) for compounds with concentration >5 or 2x RL
EB	Collected daily with nondedicated sampling equipment	Results <RL	Evaluate sample results; flag data as necessary Review decontamination procedures.	Data Validator	Accuracy	Results <RL unless target analytes in field samples are >10× those in EB
Field Blank	One per sampling event	Results <RL	Evaluate sample results; flag data as necessary.	Data Validator	Accuracy	Results <RL

Notes:
< = less than
> = greater than
% = percent
± = plus or minus
aq = aqueous
CCV = continuing calibration verification
EB = equipment blank
FD = field duplicate
IS = internal standard
MB = method blank
MS = matrix spike
MSD = matrix spike duplicate
QA = quality assurance
QC =quality control
RL = reporting limit
RPD = relative percent difference
RT = retention time
SOP = standard operating procedure
VOC = volatile organic compound

Table 3-6. Precision and Accuracy for 1,4-Dioxane in Soil and Water

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD %R	Soil LCS %R	RPD[a]	Water MS/MSD %R	Water LCS %R	RPD[a]
1,4-Dioxane	SW8270B SIM/8270 SIM low	[b]	[b]	30	50–130	50–130	20

[a] RPDs are applicable to both MS/MSDs and LCS/LCSDs.
[b] Will use laboratory limits.

Notes:
%R = percent recovery
DoD = U.S. Department of Defense
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
MS/MSD = matrix spike/matrix spike duplicate
RPD = relative percent difference

Table 3-7. Quality Control Samples (Field and Laboratory) for Metals in Soil and Water

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Preparation Blank	One per prep batch of 20 or fewer samples of similar matrix	Target analytes <50% RL; common contaminants <RL	Redigest and reanalyze; if sample concentration is >10× blank concentration, narrate.	Laboratory Supervisor	Accuracy	Target analytes <50% RL, common contaminants <RL; unless target analytes in field samples are >10× those in MB
Interference Check Sample (A and B)	At beginning and end of instrument run and after every 20 samples	ICS-A: Unspiked analytes <RL ICS-AB: %R 80% - 120%	Reanalyze samples analyzed after last acceptable ICS-A/ICS-AB.	Laboratory Supervisor	Accuracy	ICS-A: Unspiked analytes <RL ICS-AB: %R 80% - 120%
Serial Dilution	One per prep batch of 20 or fewer samples of similar matrix	% Difference 10% if sample concentration >50× MDL	Qualify data.	Laboratory Supervisor	Accuracy	% Difference 10% if sample concentration >50× MDL
LCS	One per prep batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-8	Redigest and reanalyze; if recovery is high and sample is <RL, narrate.	Laboratory Supervisor	Accuracy	Defined in Table 3-8
LCSD	One per prep batch of 20 or fewer samples of similar matrix	RPD<20% if sample concentration >5×RL, if <5×RL ±5×RL	Check instrument performance; qualify data.	Laboratory Supervisor	Precision	Values ≥5×RL: RPD ±20% Values <5×RL: ±5×RL
MS	One per prep batch of 20 or fewer samples of similar matrix	%R, see Table 3-8	Perform PDS analysis; qualify data.	Laboratory Supervisor	Accuracy	Recovery ±25% of true value if sample <4× spike value
PDS	For elements outside of QC limits in MS	%R 80%–120%	Check instrument performance; qualify data.	Laboratory Supervisor	Accuracy	Recovery ±25% of true value
IS[a]	Each field and QC sample[a]	Percent relative intensity of each IS standard within 60–125%	Reanalyze samples after two-fold dilution.	Laboratory Supervisor	Accuracy	Percent relative intensity of each IS standard within 60–125%

Table 3-7. Quality Control Samples (Field and Laboratory) for Metals in Soil and Water
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
FD	One per 10 per matrix	RPD <30% (aq), <50% (solid) for compounds with concentration >RL	Evaluate batch precision, other FD results. Note in validation report.	Data Validator	Precision	RPD <30% (aq), <50% (solid) for compounds with concentration > 5 or 2x RL
EB	Daily	Results <RL	Evaluate sample results; flag data as necessary. Review decontamination procedures.	Data Validator	Accuracy	Results <RL unless target analytes in field samples are >10× those in EB

[a] ISs are not required but are optional for Method SW6020.

Notes:
> = greater than
< = less than
± = plus or minus
% = percent
%R = percent recovery
aq = aqueous
EB = equipment blank
FD = field duplicate
ICS = interference check sample
IS = internal standard
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
MDL = method detection limit
MS= matrix spike
PDS = postdigestion spike
QC = quality control
RL = reporting limit
RPD = relative percent difference
SOP = standard operating procedure

Table 3-8. Precision and Accuracy for Metals in Soil and Water

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD %R	Soil LCS %R	RPD[a]	Water MS/MSD %R	Water LCS %R	RPD[a]
Aluminum	SW6020A	75–125	70–130	20	75–125	70–130	20
Antimony	SW6020A	75–125	50–150	20	75–125	50–150	20
Arsenic	SW6020A	75–125	70–130	20	75–125	70–130	20
Barium	SW6020A	75–125	70–130	20	75–125	70–130	20
Beryllium	SW6020A	75–125	70–130	20	75–125	70–130	20
Cadmium	SW6020A	75–125	70–130	20	75–125	70–130	20
Calcium	SW6020A~~S~~	75–125	70–130	20	75–125	70–130	20
Chromium	SW6020A	75–125	70–130	20	75–125	70–130	20
Cobalt	SW6020A	75–125	70–130	20	75–125	70–130	20
Copper	SW6020A	75–125	70–130	20	75–125	70–130	20
Iron	SW6020A	75–125	70–130	20	75–125	70–130	20
Lead	SW6020A	75–125	70–130	20	75–125	70–130	20
Magnesium	SW6020A	75–125	70–130	20	75–125	70–130	20
Manganese	SW6020A	75–125	70–130	20	75–125	70–130	20
Mercury	SW7470A/7471A	75–125	70–130	20	75–125	70–130	20
Nickel	SW6020A	75–125	70–130	20	75–125	70–130	20
Potassium	SW6020A	75–125	70–130	20	75–125	70–130	20
Selenium	SW6020A	75–125	70–130	20	75–125	70–130	20
Silver	SW6020A	75–125	50–150	20	75–125	50–150	20
Sodium	SW6020A	75–125	70–130	20	75–125	70–130	20
Thallium	SW6020A	75–125	70–130	20	75–125	70–130	20
Vanadium	SW6020A	75–125	70–130	20	75–125	70–130	20
Zinc	SW6020A	75–125	70–130	20	75–125	70–130	20

[a] RPDs are applicable to MS/MSDs only.

Notes:
%R = percent recovery
LCS = laboratory control sample
MS/MSD = matrix spike/matrix spike duplicate
RPD = relative percent difference

Table 3-9. Quality Control Samples (Field and Laboratory) for Volatile Organic Compounds in Air (Modified TO-15 SIM)

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	Daily	Target analytes: <50% RL; common contaminants <2.5× RL	Rerun, maintenance. Flag data.	Laboratory Chemist	Accuracy – Contamination	Target Analytes <50% RL; common contaminants <2.5× RL unless results in field samples >10× that in MB
LCS/LCS Duplicate	Daily	%R 70–130% RPD<30%	Rerun, investigate source of problems. Flag data.	Laboratory Chemist	Accuracy Precision	%R 70–130% RPD<30%
LD	One per data package	RPD<20%	Flag data.	Laboratory Chemist	Precision	RPD<20%
FD	One per 10 samples	RPD<40%	Flag data.	Data Validator	Precision	RPD<40%
IS	Every sample	Area ±40% DCV	Rerun, flag data.	Laboratory Chemist	Accuracy	Area ±40% DCV
Surrogate spike	Every sample	%R 70–130% or per laboratory limits	Rerun, flag data.	Laboratory Chemist	Accuracy	%R 70–130% or per laboratory limits
Reporting Limit LCS	Daily	Results ±40% true for 90% of compounds	Flag data that fall outside control limits.	Laboratory Chemist	Sensitivity	Results ±40% true for 90% of compounds
Cleaning Procedure verification (Summa canisters)	Each canister	After cleaning, pressurized canister (using zero humid air) should not contain Target Analytes greater than 0.2 ppbV	Reclean the canister; perform verification.	Laboratory Chemist	Accuracy – Contamination	After cleaning, pressurized canister (using zero humid air) should not contain target analytes greater than 0.2 ppbV

Notes:
< = less than
> = greater than
± = plus or minus
% = percent
DCV = daily calibration verification
LCS = laboratory control sample
ppbV = parts per billion by volume
QC = quality control
RL = reporting limit

RPD = relative percent difference
SOP = standard operating procedure

Table 3-10. Quality Control Samples (Field and Laboratory) for Volatile Organic Compounds in Air (Modified TO-17)

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	Daily	Target analytes: <50% RL; common contaminants <2.5 × RL	Rerun, maintenance. Flag data.	Laboratory Chemist	Accuracy – Contamination	Target Analytes <50% RL; common contaminants <2.5× RL unless results in field samples >10× that in MB
LCS/LCS Duplicate	Daily	%R 70–130% RPD<30%	Rerun, investigate source of problems. Flag data.	Laboratory Chemist	Accuracy Precision	%R 70–130% RPD<30%
LD	One per data package	RPD<20%	Flag data.	Laboratory Chemist	Precision	RPD<20%
FD	One per 10 samples	RPD<40%	Flag data.	Data Validator	Precision	RPD<40%
IS	Every sample	Area ±40% DCV	Rerun, flag data.	Laboratory Chemist	Accuracy	Area ±40% DCV
Surrogate spike	Every sample	%R 70–130% or per laboratory limits	Rerun, flag data.	Laboratory Chemist	Accuracy	%R 70–130% or per laboratory limits
Reporting Limit LCS	Daily	Results ±40% true for 90% of compounds	Flag data that fall outside control limits.	Laboratory Chemist	Sensitivity	Results ±40% true for 90% of compounds
Field Blank	One per sampling event	Results <RL	Evaluate sample results; flag data as necessary.	Data Validator	Accuracy	Results <RL unless target analytes in field samples are >10× those in EB

Notes:
< = less than
> = greater than
± = plus or minus
% = percent
DCV = daily calibration verification
LCS = laboratory control sample
ppbV = parts per billion by volume
QC = quality control
RL = reporting limit
RPD = relative percent difference
SOP = standard operating procedure

Table 3-11. Quality Control Samples (Field and Laboratory) for General Chemistry Parameters in Water
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Preparation Blank[a]	One per prep batch of 20 or fewer samples of similar matrix	Target analytes <50% RL; common contaminants <RL	Reprepare and reanalyze if sample concentration is >10× blank concentration narrate.	Laboratory Supervisor	Accuracy	Target analytes <50% RL, common contaminants <RL; unless target analytes in field samples are >10× those in MB.
LCS[b]	One per prep batch of 20 or fewer samples of similar matrix	Recovery within laboratory statistical QC limits as defined in Table 3-11	Reprepare and reanalyze if recovery is high and sample is <RL narrate.	Laboratory Supervisor	Accuracy	Recovery within laboratory statistical QC limits as defined in Table 3-11
LCSD[b]	One per prep batch of 20 or fewer samples of similar matrix	RPD ≤20% if sample concentration >5×RL; ± RL if <5×RL	Check instrument performance, qualify data.	Laboratory Supervisor	Precision	Values ≥5×RL: RPD ±20% Values <5×RL: ±5× RL
MS[c]	One per prep batch of 20 or fewer samples of similar matrix	Recovery within laboratory statistical QC limits as defined in Table 3-11	Reprepare and reanalyze if recovery is high and sample is <RL narrate.	Laboratory Supervisor	Accuracy	Recovery within laboratory statistical QC limits (<4× spike value) as defined in Table 3-11
FD	One per 10 per matrix	RPD <30% for compounds with concentration >RL	Evaluate batch precision, other FD results. Note in validation report.	Data Validator	Precision	RPD <30% for compounds with concentration > 5x RL
EB[a]	Daily	Results <RL	Evaluate sample results; flag data as necessary. Review decontamination procedures.	Data Validator	Accuracy	Results <RL unless target analytes in field samples are >10× those in EB

[a] Not applicable to Method SM2540C (total dissolved solids) or Method SM2320B (alkalinity).
[b] Not applicable to Method SM2320B (alkalinity).
[c] Not applicable to Method SM2540C (total dissolved solids), Method RSK-175, or Method SM2320B (alkalinity).

Notes:
< = less than
> = greater than
± = plus or minus
% = percent
EB = equipment blank
FD = field duplicate
QC = quality control
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
MS = matrix spike
RL = reporting limit
RPD = relative percent difference
SOP = standard operating procedure

Table 3-12. Precision and Accuracy for General Chemistry Parameters in Water

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Water MS/MSD %R	Water LCS %R	RPD[c]
Chloride	EPA 300.0[a]	87 – 111	87 – 111	20
Nitrate/Nitrite	EPA 300.0[a]	88 – 111	88 – 111	20
Nitrate/Nitrite	SM4500-NO3E	75 – 125	80 – 120	20
Sulfate	EPA 300.0[a]	87 – 112	87 – 112	20
Sulfide	SM4500S2-CF	80 – 120[b]	80 – 120[b]	20
TOC	SW9060	80 – 120[b]	80 – 120[b]	20
Alkalinity	SM2320B	80 – 120[b]	80 – 120[b]	20
TDS	SM2540C	NA	80 – 120[b]	20
Ethane	RSK-175	70 - 140	70 - 140	30
Ethene	RSK-175	70 - 140	70 - 140	30
Methane	RSK-175	70 - 140	70 - 140	30

[a] LCS and MS/MSD limits based on DoD QSM Version 5.1, Appendix C (DoD 2017). For Method 300.0, used same limits as Method 9056.
[b] Laboratory limits.
[c] RPDs are applicable to MS/MSDs only.

Notes:
%R = percent recovery
DoD = U.S. Department of Defense
LCS = laboratory control sample
MS/MSD = matrix spike/matrix spike duplicate
NA = not applicable
QSM = quality systems manual
RPD = relative percent difference
TDS = total dissolved solids
TOC = total organic carbon

Table 3-13. Quality Control Samples (Field and Laboratory) for Oxygen and Hydrogen Stable Isotope Parameters in Water

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
PT Reference Water[a]	Run at the beginning of run and after every eight samples	Not vary by more than 0.9‰ for δ^2H and 0.12‰ for $\delta^{18}O$ (one standard deviation)	If monthly values exceed the thresholds, the data from individual runs should be reviewed to identify and remedy potential errors in data processing or any systematic changes in instrument performance and their cause.	Laboratory Supervisor	Precision	Not vary by more than 0.9‰ for δ^2H and 0.12‰ for $\delta^{18}O$ (one standard deviation)
PZ Reference Water[a]	One per sample tray run	Standard deviation should be no more than 20% of the average parameter value across the month	Review data from individual runs to identify and remedy potential error or systematic changes. Reanalyze sample and reassess if necessary.	Laboratory Supervisor	Precision	Less than or equal to 20% average over the month
UT Reference Water[a]	One per sample tray run	Standard deviation should be no more than 20% of the average parameter value across the month	Review data from individual runs to identify and remedy potential error or systematic changes. Reanalyze sample and reassess if necessary.	Laboratory Supervisor	Precision	Less than or equal to 20% average over the month
FD	One per 10 per matrix	Standard deviation of calibrated isotopic values for 3 or more replicate injections of the unknown sample must be less than 0.2‰ ($\delta^{18}O$) and 0.75‰ (δ^2H)	Evaluate batch precision, other FD results. Note in validation report.	Data Validator	Precision	RPD<30% for compounds with concentration >RL

[a] PT, PZ, and UT are internal reference materials developed by the Stable Isotopes Ratio Facility for Environmental Research, University of Utah, for use as QC standards to monitor method performance.

Notes:

< = less than
> = greater than
% = percent
‰ = per mil
FD = field duplicate
QC = quality control
RL = reporting limit
RPD = relative percent difference
SOP = standard operating procedure

Table 3-14. Quality Control Samples (Field and Laboratory) for Carbon Stable Isotope Parameters in Water
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
LCS[a]	One per prep batch of 20 or fewer samples of similar matrix	%R = 70–130%	Reprepare and reanalyze if recovery is high and sample is <RL narrate.	Laboratory Supervisor	Accuracy	%R = 70–130%
FD	One per 10 per matrix	RPD<30% for compounds with concentration >RL	Evaluate batch precision, other FD results. Note in validation report.	Data Validator	Precision	RPD<30% for compounds with concentration >RL

[a] Reference materials are maintained by the Stable Isotope Laboratory for use as QC standards to monitor method performance.

Notes:
< = less than
> = greater than
% = percent
%R = percent recovery
FD = field duplicate
QC = quality control
RL = reporting limit
RPD = relative percent difference
SOP = standard operating procedure

Table 3-15. Quality Control Samples (Field and Laboratory) for DHC and Functional Genes in Water
Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Assay Negative Control (Blank)	One per analytical assay plate	Values for positive samples are set above any fluorescence for the negative control	Rerun assay; may have to reoptimize assay.	Laboratory area supervisor	Accuracy	Values for positive samples are set above any fluorescence for the negative control
DNA Extraction – Negative Control	One per analytical batch	CT ≤ assay negative control	Rerun assay or re-extract samples if problem persists.	Laboratory area supervisor	Accuracy	CT ≤ assay negative control
Positive Control	One per analytical assay plate	Calculated concentration within ±20% of same concentration on standard curve	Rerun assay/check reagents.	Laboratory area supervisor	Accuracy	Calculated concentration within ±20% of same concentration on standard curve
FD	One per 20 per matrix	RPD<50%	Evaluate batch precision, other FD results. Note in validation report.	Data Validator	Precision	RPD<30%

Notes:
< = less than
≤ = less than or equal to
% = percent
± = plus or minus
CT = counts
FD = field duplicate
QC = quality control
RL = reporting limit
RPD = relative percent difference
SOP = standard operating procedure

3.6 Instrument/Equipment Testing, Inspection, Maintenance (B6) and Calibration (B7)

The following subsections discuss inspection, testing, and regularly scheduled preventive maintenance to keep all field and laboratory equipment in good working condition. Required field equipment is identified in the SOP for each work task (**Appendix A**).

3.6.1 Field Equipment

Field personnel will record service and maintenance information of all field equipment in the project field logbook. Instrument problems encountered during field work will be remedied in the field, if possible, and documented in the field notes. Specific preventive maintenance procedures will follow the manufacturer's recommended schedule. Preventive maintenance will be the responsibility of the FTL. Field equipment that becomes inoperable will be removed from service and tagged to indicate that repair, recalibration, or replacement is needed. Field team leaders will be notified so that prompt service can be completed or substitute equipment can be obtained. Back-up systems will be prearranged for each instrument in use and will be calibrated before use in the field. The maintenance schedule is tracked internally by the FTL using a spreadsheet in conjunction with an Outlook calendar reminder via email.

Field equipment calibration is necessary so that measurements are accurate in reference to a known standard. Equipment will be calibrated according to the manufacturer's instructions, but at least daily, with more frequent calibration to correct drift or if the daily event indicates the instrument is not holding calibration. More complete information on field equipment calibration is discussed further in SOP 5-1, *Control of Measurement and Test Equipment* (**Appendix A**). A calibration will be verified at any time during the day when measurements are suspected to be erroneous. Calibration solutions will be available on-site with each instrument and will be renewed before the expiration dates stamped on the manufacturer's container. Documentation of each calibration will include the lot number and expiration of the calibration standards. Additional batteries will be maintained on-site. All calibration information will be recorded on daily field forms and in the project logbook. Calibration records for the equipment will be readily available for reference. In addition, the portable GC/MS (HAPSITE) will be calibrated daily in accordance with the manufacturer's operating instructions provided in the SOP (**Appendix A**).

Field equipment that fails calibration or becomes inoperable during use will be removed from service and either segregated to prevent inadvertent use or tagged to indicate it is out of calibration. Such equipment will be repaired and satisfactorily recalibrated. Equipment that cannot be repaired will be replaced.

Results of activities performed using equipment that has failed recalibration will be evaluated by the CDM Smith FTL and the project data manager. If the activity results are adversely affected, the results of the evaluation will be documented and the CDM Smith PM and (other data users) will be notified. The CDM Smith PM will take appropriate action, including the rejection of the data, if needed, with recollection of the data where practical, and will obtain concurrence from USACE/VHA for issues with field data that would impact results of the investigation.

Table 3-16. Field Equipment Calibration, Maintenance, Testing, and Inspection

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Field Equipment	Activity[1]	Description	SOP Reference	Responsible Person	Frequency	Acceptance Criteria	Corrective Action[2]
YSI DSS Pro flow-through water quality meter or equivalent	Calibration	Calibrate at the beginning of the day	Manufacturer's specification 5-1 Control of Measurement and Test Equipment	FTL	Calibrate daily Check daily, before each use	±10% of calibrated value	Manually zero meter or service as necessary and recalibrate
	Maintenance	Performed before shipment and as needed	Manufacturer's specification	FTL			
	Testing	Check periodically during field activities and at end of day					
LaMotte Turbidity Meter or equivalent	Calibration	Calibrated by LaMotte	Manufacturer's specification	FTL	Check daily, before each use	±10% of calibrated value	Service as necessary Possible replacement required
	Testing	Check daily, before use					
HACH Colorimeter	Calibration	Calibrated by HACH	Manufacturer's specification	FTL	Check daily, before each use	±10% of calibrated value	Service as necessary Possible replacement required
	Testing	Check daily, before use					
RAE Systems MiniRAE 3000 organic vapor meter/ photoionization detector or equivalent	Calibration	Calibrate with known concentration of Isobutylene 100 ppm (calibration gas)	5-1 Control of Measurement and Test Equipment Manufacturer's specification	FTL	Calibrate daily	±10% of calibrated value	Manually zero, recalibrate or service as necessary Possible replacement required
	Maintenance	Performed before shipment and as needed					
	Testing	Check against calibration gas as needed					

Notes:
[1] All equipment will be inspected prior to use by the FTL.
[2] No spare parts for field equipment are available onsite and will be supplied as needed by the manufacturer.

% = percent
± = plus or minus
FTL = field team leader
RPD = relative percent difference
SOP = standard operating procedure

3.6.2 Laboratory Equipment

The laboratories will follow a maintenance schedule for each instrument used to analyze samples as provided in the laboratory QA manual. Instrument maintenance logbooks are maintained in laboratories at all times. The logbooks, in general, contain a schedule of maintenance as well as a complete history of past maintenance, both routine and nonroutine. Preventive maintenance is performed according to the procedures described in the manufacturer's instrument manuals including lubrication, source cleaning, detector cleaning, and the frequency of such maintenance. Chromatographic carrier gas-purification traps, injector liners, and injector septa are cleaned or replaced on a regular basis. Precision and accuracy data are examined for trends and excursions beyond control limits to determine evidence of instrument malfunction. Maintenance will be performed when an instrument begins to degrade as evidenced by the degradation of peak resolution, shift in calibration curves, decrease in sensitivity, or failure to meet one QC criteria.

Instrument downtime is minimized by keeping adequate supplies of all expendable items, where expendable means an expected lifetime of less than 1 year. These items include gas tanks, gasoline filters, syringes, septa, gas chromatography columns and packing, ferrules, printer paper and ribbons, pump oil, jet separators, open-split interfaces, and mass spectroscopy filaments.

Analytical instruments will be calibrated in accordance with the method specified by the laboratory. All analytes reported must be present in the initial and continuing calibrations, and these calibrations must meet the acceptance criteria specified in the method. The laboratory will maintain records of standard preparation and instrument calibration. Records will unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards will be traceable to standard materials.

ICAL curves must be verified using a standard from a source independent of the one used to establish the ICAL. All target compounds must be included with the ICAL verification, typically at a concentration around the midpoint of the calibration curve. Failure of the ICAL verification requires corrective action.

The ICAL curves must be verified daily before sample analysis using a CCV. The CCVs are required check samples at the beginning of each analytical sequence and as specified in each analytical method. All target compounds must be included within the CCV, typically at a concentration around the midpoint of the calibration curve. Failure of the ICAL or CCV requires corrective action.

Standard materials used in calibration and to prepare samples will be traceable to the National Institute of Standards and Technology, EPA, the American Association of Laboratory Accreditation, or other approved sources if available. The standard materials will be current; the expiration dates for standards will not exceed the manufacturer’s expiration date or 1 year from the date of receipt, whichever occurs first. Expiration dates for laboratory-prepared stock and diluted standards will be no later than the expiration date of the stock solution or material, or the date calculated from the holding time allowed by the applicable analytical method, whichever occurs first. The laboratory will label standard and QC materials with expiration dates.

3.7 Inspection/Acceptance of Supplies and Consumables (B8)

The FTL will obtain and verify all supplies and consumables are on hand when needed and have been inspected and tested for suitability of use. Most supplies and consumables to be used in the field will be procured from an environmental sampling supply vendor. Before any item is used in the field, it will be inspected and tested. Any defective material will be replaced before the sampling event begins. All sample containers, with appropriate preservation, will be procured from a commercial provider. Containers may be supplied by the laboratories; however, the laboratory will also purchase containers from a commercial provider. All containers must be certified clean including Summa canisters, which will be individually certified. All containers and coolers will be inspected before they are used for collecting and shipping samples. Appropriate materials, including bubble wrap, plastic bags, tape, and supplies, will be available for packing samples to avoid breakage during transportation.

Supplies and consumables for field instrumentation to be used during the investigation will be consistent with the corresponding operating manual for the device. SOPs for the operation of field devices and equipment to be used during the investigation can be found in **Appendix A**.

3.8 Data Acquisition Requirements – Nondirect Measurements (B9)

Nondirect measurements including historical data (e.g., water quality data, groundwater elevation measurements) were used in the development of the conceptual site model and the project objectives. Relevant historical data will be stored in a project-specific database built in the EarthSoft EQuIS system. All manually generated information (e.g., field test results and GIS sample coordinates) will undergo QC verification by the FTL. The acceptance criteria for existing data are provided in **Tables 2-3** through **2-11**, as applicable. If specific criteria are not available, professional judgment will be applied. Data that meet current acceptance criteria will be used without any limitation. Data that may not or do not meet current acceptance criteria will be excluded from use or qualified for use in the document where those data are used or reported. In the case of the Groundwater Flow Model and Solute Transport Model, the acceptance criteria and any limitations on use of data will be documented in the groundwater model technical memorandum. Computer programs generating or maintaining electronic information will be verified by the developer or manufacturer before implementation. All computer systems will have a minimum security of password protection. All project information will be considered confidential and only project personnel will be allowed access.

3.9 Data Management (B10)

Data management includes the collection, processing, tracking, storing, and reporting of environmental and spatial data used in the project. The project data manager will oversee the data handling and management processes of these data using the project data management plan (DMP). The DMP provides information on the data flow and QC interface for the project's field measurement, analytical laboratory, and spatial data. The DMP assigns data management roles and responsibilities for specific QC steps that cover activities, including:

- planning

- field data collection
- laboratory coordination
- validation
- data compilation
- reporting
- preparation of final deliverables

The DMP (RIWP Appendix E) is discussed in Section 5 of the RIWP and will be supported with information from the associated FSP (RIWP Appendix A).

Sample information, field measurement data, and analytical data will be stored in the project's EQuIS Microsoft SQL Server database. The database is located on a CDM Smith server and is backed up nightly. These data are made available through the project's EQuIS Enterprise site, which allows users to query and download data. Backup copies of electronic data files, including field measurement and analytical data, will be stored off-site on ProjectWise maintained by CDM Smith during the term of the contract, and at the end of the contract, transferred to an off-site data archive site.

Spatial data used for project analysis and figures are housed in a project geodatabase. The geodatabase is stored on a CDM Smith network server, which is backed up nightly. GIS metadata will also be stored in the geodatabase.

The CDM Smith team will retain the entire project file as hard copy and/or electronic data on CDM Smith ProjectWise until the completion of all project activities. Thereafter, all data will be provided to USACE/VHA and archived by CDM Smith in a secure and protected facility and maintained for a minimum of 3 years after contract closeout, per FAR 52.215-2 (Audit and Records). Data stored in the EQuIS database, along with GIS layers with their associated metadata, will be provided to USACE/VHA and archived by CDM Smith in electronic format for transmittal to EPA and UDEQ, if requested.

The official administrative record file for the site is located online at www.pceplume.org. Electronic copies of project reports, including the RIWP, sampling analysis plan, and QAPP, will also be provided to EPA and UDEQ. A copy of the administrative record file is also located at the VHA Salt Lake City Health Care System, 500 Foothill Drive in Salt Lake City, Utah 84148. EPA Region 8 in Denver, Colorado, and UDEQ in Salt Lake City, Utah, will receive all copies of documents, laboratory data, and other published project information for their records.

Section 4

Assessment and Oversight (EPA Group C)

This section addresses the activities required to properly assess the effectiveness of the project implementation and the associated QA/QC activities. It includes the following:

- Assessment and Response Action (C1)
- Reports to Management (C2)

4.1 Assessments and Response Actions (C1)

This subsection presents activities for assessing the effectiveness of QAPP implementation, which includes audits and corrective action.

4.1.1 Laboratory Audits

Prior to receiving samples, an on-site technical system audit (TSA) of each laboratory will be performed, as needed, with the exception of the stable isotope and geotechnical laboratories. TSAs are the most frequently performed type of audit and consist of a project-specific on-site evaluation of a measurement or data collection system. TSAs for project activities are performed to verify that requirements (procedural, regulatory, or contractual) are being met. EPA and UDEQ will be notified when a TSA is to be performed. The objective of a TSA is to assess all system facilities and to document system operations and maintenance, experimental procedures, recordkeeping, calibration procedures, reporting requirements, data validation, and QC procedures, as defined in the approved planning documents. Any undocumented or unauthorized deviations from the planning documents and corrective action are noted in the written laboratory audit report. A TSA report will be developed, which will include any necessary corrective actions for the laboratory.

4.1.2 Corrective Action Procedures

Conditions adverse to data quality must be promptly investigated, evaluated, and corrected. Adverse conditions may include instrument malfunctions, deficiencies in QA/QC criteria, deviations from SOPs, and errors in data reduction, validation, or documentation.

4.1.2.1 Field Corrective Action Procedures

All CDM Smith project personnel, subcontractors, consultants, VHA, and USACE project personnel will be responsible for identifying project deficiencies and notifying the CDM Smith QAS and QAM. Any defects and deficiencies identified will be documented in the daily report by the CDM Smith FTL and added to a tracking system (e.g., electronic spreadsheet) by the CDM Smith QAS. The CDM Smith QAS will describe any deficient items and the change in status of any deficiencies in the daily report and in a nonconformance report. USACE and VHA will be notified of all identified deficiencies, the prescribed corrective actions, and the status of the corrective action through completion. The information will be provided to USACE and VHA in the daily report and during coordination meetings.

The effective implementation of the QAPP in the field will be evaluated through a system audit during the field phase of the investigation. The field system audit will assess compliance with the FSP/QAPP procedures and SOPs for field investigation, including recordkeeping, equipment calibration, sampling procedures, decontamination, and field data verification. EPA and UDEQ will be notified when a field audit is to be performed. System audits will be conducted by an individual not working directly on the project. The FTL will verify that the following corrective actions for nonconformances are implemented:

- Evaluate all reported nonconformances
- Control additional work on nonconforming items
- Determine disposition or action to be taken
- Verify that corrective actions were taken
- Maintain a log of nonconformances
- Review nonconformance reports
- Evaluate disposition or action taken
- Monitor inclusion of nonconformance reports in final site documentation and document control
- Notify the CDM Smith PM, who will notify USACE of nonconformances and corrective actions; USACE will then notify VHA

The CDM Smith FTL will guarantee that no additional work dependent on the nonconforming activity is performed until the nonconformance is corrected. Also, the FTL will implement corrective action as initiated by the CDM Smith QAM. Each nonconformance report will be evaluated, and the disposition and action taken will be recorded.

For any issued deliverable or document, all staff will be responsible for reporting all suspected QA nonconformance by initiating a preventive/corrective action notification.

4.1.2.2 Laboratory Corrective Action Procedures

The laboratory QA plan defines the laboratory internal corrective action process and requirements. When a condition adverse to data quality is noted at the laboratory, the cause of the condition will be determined and corrective action will be taken to re-establish control. Identification, cause, reference documents, corrective action taken, and effectiveness will be documented and reported to the CDM Smith PM by the laboratory QC manager. Implementation of corrective action is verified by documented follow-up action. A record of the action taken and results will be attached to the data report package.

Project personnel have the responsibility, as part of normal work duties, to identify, report, and solicit approval of corrective actions for conditions adverse to data quality. Examples of corrective actions include the following:

- When project-specific acceptance criteria are not attained (objectives for precision, accuracy, and completeness)
- When the prescribed procedure or any data compiled contains an error
- When equipment or instrumentation is determined to be faulty
- When the traceability of samples, standards, or analytical results is questionable
- When QA requirements have been violated
- When designated approvals have been circumvented
- As a result of systems or performance audits
- As a result of regular management assessments
- As a result of intralaboratory or interlaboratory comparison studies
- Any other instance of conditions that adversely affect quality

Analysts conducting sample analysis activities may take immediate corrective action for out-of-control conditions. Faulty calibrations and blank contamination are examples of out-of-control conditions. Immediate corrective and successful action will reduce questionable data quality. Corrective actions for these types of failures should be documented in an instrument logbook, which will be available for the final data report.

Laboratory staff will monitor work performance in the normal course of daily responsibilities. The laboratory QA manager or designated alternate will audit work at the laboratory. Items, activities, or documents ascertained to be noncompliant with QA requirements will be documented, and corrective actions will be mandated in the audit report. The laboratory QA manager will log, maintain, and control the audit findings.

The laboratory QA manager will document all out-of-control events or nonconformance with QA protocols. The report will summarize each nonconformance condition. A copy of the reports will be submitted with the final data package. The laboratory QA manager, in consultation with the CDM Smith PM, may initiate a stop-work order if corrective actions are insufficient.

4.2 Reports to Management (C2)

This subsection describes how management (USACE, VHA, EPA, and UDEQ) will be kept informed of project oversight and assessment activities and findings. It describes what kinds of reports will be written and how often they will be submitted for review to the approving authorities.

4.2.1 Report Type and Frequency

Project reports submitted during the course of the project will include, but may not be limited to, the following submittals in accordance with the project schedule and key investigation events:

- **Progress Reports** – Communicate the progress made on a monthly basis regarding site investigation, community involvement, achievement of project milestones, and the myriad

of actions that encompass the investigation. These reports will be written by the CDM Smith PM or designee and submitted to USACE and VHA.

- **Data Summary Reports** – Communicate the conclusions of individual investigation events for the site and any laboratory analysis associated with the event. These reports will be written by the CDM Smith PM or designee and submitted to USACE and VHA.
- **Quality Control Summary Reports and Data Usability Assessment Reports** – Summarize the number and types of samples collected, including field QC samples, analytical methods performed, and data evaluation purpose. Provide a summary of the overall completeness of the data set and discuss any limitations in the use of the analytical data. Refer to Section 5.3.1 for additional details.
- **Nonconformance Reports** – Identify any deviations of planned or expected scope-of-work activities relating to the investigation. Reports may pertain to field data collection and health and safety events. These reports will be written by the CDM Smith PM or QAM and submitted to USACE and VHA.
- **Laboratory Audit Reports/Corrective Action Reports (CARs)** – Communicate the result of any third-party audit or review of the analytical laboratory supporting the investigation activities. This report will be written by the project chemist. The reports will be submitted to the CDM Smith PM, USACE, and VHA.
- **RPM Meetings** – Conduct quarterly conference calls among USACE, VHA, EPA, UDEQ and other key stakeholders to discuss current planning and investigation.
- **Interim RI Report** – Summarize field data collection efforts for the site and include field forms, validated analytical data, groundwater modeling calibration results, and recommendations for additional investigation activities.
- **RI Report** – Communicate the conclusions of all investigation events and any laboratory analysis associated with the event. This report will also contain data validation reports and deviations from the FSP and QAPP. The RI report will incorporate data and information from the previous and current investigations into a comprehensive document. The report format will closely follow the *Guidance for Conducting Remedial Investigations and Feasibility Studies under the Comprehensive Environmental Response Compensation and Liability Act (CERCLA)* (EPA 1988) and will include historical and background information, field measurements, analytical results, a summary of field protocols, the identified vertical and horizontal extent of contamination, geologic data interpretations, and ecological and baseline risk assessment results. CDM Smith will prepare a preliminary draft report, responses to VHA comments on the preliminary draft report, a draft report, responses to stakeholder comments, and a final report incorporating all review comments and documenting the results and findings of the RI. This report will be written and submitted by CDM Smith to USACE and VHA, with distribution by VHA to EPA and UDEQ.

Table 4-1 provides a summary of the above reports and communications and identifies the responsible author, reporting frequency, and the recipient(s) of the deliverable.

Table 4-1. Summary of Reports to Management

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Reports	Frequency	Provided to	Format	Prepared by
Progress reports	Monthly	USACE and VHA	Written	CDM Smith PM
Site investigation reports, field summary reports (i.e., data summary reports), data validation and usability reports, preliminary draft RI report, and final RI report	As needed	USACE and VHA, with distribution by VHA to EPA and UDEQ	Written	CDM Smith
Field change requests to document deviations from the QAPP, RIWP, and FSP	As needed	USACE and VHA, with distribution by VHA to EPA and UDEQ	Written	CDM Smith
Nonconformance reports	As needed	USACE and VHA, with distribution as needed by VHA to EPA and UDEQ	Written	CDM Smith PM, QAM
Laboratory audit (TSA)/CARs	As scheduled	USACE and VHA, with distribution by VHA to EPA and UDEQ	Written	CDM Smith project chemist or designee
Field (QAPP) implementation audit/CARs	As scheduled, at least once during field work	USACE and VHA, with distribution by VHA to EPA and UDEQ	Written	CDM Smith QAS
RPM Meetings	Quarterly	USACE and VHA, with distribution by VHA to EPA and UDEQ	Verbal, with minutes Written	CDM Smith

Notes:
CAR = corrective action report
EPA = U.S. Environmental Protection Agency
PM = project manager
QAPP = quality assurance project plan
QAM = quality assurance manager
RI = remedial investigation
RPM = Remedial Project Manager
TSA = technical system audit
UDEQ = Utah Department of Environmental Quality
USACE = U.S. Army Corps of Engineers
VHA = Veterans Health Administration

Section 5

Data Validation and Usability (EPA Group D)

This section addresses QA activities that will occur after the data collection phase is completed, which include the following:

- Data Review, Verification and Validation (D1)
- Verification and Validation Methods (D2)
- Reconciliation with User Requirements (D3)

5.1 Data Review, Verification, and Validation (D1)

Data for all parameters will undergo two levels of review and validation, one at the laboratory and another by the CDM Smith project chemist or designee, to include third-party reviewers. Verification of data generated from field activities will also be performed.

5.1.1 Field Data Verification

Field personnel will be responsible for following the sampling and documentation procedures described in the FSP so that defensible and usable data are obtained. Integrity of information and field activity data will be the responsibility of the CDM Smith FTL. Project team personnel will validate field data through reviews to identify inconsistencies or anomalous values.

All information generated in the field (all data, calibration, logbook, field data sheets, chain-of-custody records, inspection reports, drilling logs, and nonconformance reports) will be assembled for review and will be verified by a peer review process. Example field forms are provided in the SOPs (**Appendix A**). If possible, any inconsistencies discovered will be resolved immediately by seeking clarification from the field personnel responsible for the data collection. Integrity of field sample custody is accomplished according to the sample custody procedures defined in Section 3.3. The field documentation verification process is summarized in **Table 5-1**.

Table 5-1. Data Verification Processes for Field and Laboratory Results

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Verification Input	Description	Internal/External (I/E)	Responsible for Verification (Name, Organization)
Field logbook	All entries complete, signed, corrections properly initialed, sample list corresponds to chain of custody	I	CDM Smith FTL
Chain-of-custody forms	Field chain of custody is completed with legible sample ID, dates, times, all analytical parameters correctly entered, preservatives noted, signatures Laboratory chain of custody indicates any errors, signatures signifying acceptance of custody	I	CDM Smith FTL
		E	Laboratory sample custodian
Sample location data (GPS coordinates)	All sample locations have associated northings, eastings, elevation	I	CDM Smith FTL
Field originated NCRs/CARs	When required, properly completed with appropriate corrective action specifies and signatures where required; properly filed	I	CDM Smith QAM
Field summary reports	Submitted on daily basis, properly archived, each day of work week accounted for, all information present	I	CDM Smith PM
Field surveillance (TSA) report	Report details date of surveillance, person(s) conducting, findings and observations recorded, NCR/CAR as needed, follow-up response documented	I	CDM Smith QAM
Sample receiving document	Laboratory verified against chain of custody	E	Laboratory sample custodian
Draft laboratory results	All samples have results as requested, IDs match chain of custody, all QC present and reported per QAPP	E	Laboratory QA manager or designee
Laboratory surveillance report	Report details date of surveillance, person(s) conducting, findings and observations recorded, NCR/CAR as needed, follow-up response documented	I	CDM Smith QAM designee
Analytical data package	Verify data package for completeness including the presence of laboratory case narrative, sample receipt form, holding times record. Sample results, blank results, MS/MSD summary forms, LCS summary forms. Surrogate and internal standard summary forms (where appropriate), initial and continuing calibration summary and raw data	I	CDM Smith Project chemist during data validation stage
		E	Laboratory QA manager or designee
Laboratory originated NCRs/CARs	When required, properly completed with appropriate corrective action specifies and signatures; properly filed	E	Laboratory QA manager or laboratory PM
Field change request to document QAPP modifications or deviations	Verify that all QAPP modifications are documented	I	CDM Smith QAM

Table 5-1. Data Verification Processes for Field and Laboratory Results

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Verification Input	Description	Internal/External (I/E)	Responsible for Verification (Name, Organization)
Analytical EDDs	Verify that all SDGs are reported in specified format and have required deliverables	E	Laboratory QA manager or laboratory PM

Notes:
CAR = corrective action report
EDD = electronic data deliverable
FTL = field team leader
GPS = Global Positioning System
ID = identification
LCS = laboratory control sample
MS/MSD = matrix spike/matrix spike duplicate
NCR = nonconformance report
PM = project manager
QA = quality assurance
QAM = quality assurance manager
QAPP= quality assurance project
QC = quality control
SDG = sample delivery group
TSA = technical system audit

5.1.2 Laboratory Data Reduction, Review, and Approval

The laboratory review of CLP-equivalent data is a four-step process involving an evaluation by the analyst, a peer review, an administrative review, and a QA review. The review will include the following:

- Instrument calibrations
- Raw data
- Transcribed data
- Calculations, including calculations of PARCCS

The analyst will review all laboratory data before reporting. The establishment of detection and control limits will be verified. Any data outside of the established detection or control limits specified in the analytical methods will be identified. Any trends or problems with the data will be evaluated. The absence of records supporting the establishment of control criteria or detection limits will be noted. Analytical batch QC, calibration check samples, ICALs, CCVs, CARs, results of reanalysis, sample holding times, and sample preservations will be evaluated.

Samples associated with out-of-control QC data will be identified in the data package case narrative. An assessment of the utility of such analytical results will be made. **Table 5-2** provides the common data qualifiers that should be used to identify specific data limitations.

The review of laboratory data completeness will be documented to confirm the following activities:

- All samples and analyses specified on the chain-of-custody record were analyzed and reported
- Complete records exist for each analysis and the associated QC samples
- Procedures specified in this QAPP were implemented

An analyst other than the original data processor will perform a peer review of all steps of the data processing. One hundred percent of all data will be reviewed. All input parameters, calibrations, and transcriptions will be checked. Each page of checked data will be signed and dated by the verifier.

QC sample results are compared against stated criteria for accuracy and precision. QC data must meet acceptance levels before processing the analytical data. If QC standards are not satisfactory, the cause will be determined. If the cause can be corrected without affecting the integrity of the analytical data, data processing will proceed. If a resolution jeopardizes the integrity of the data, reanalysis will ensue.

The laboratory PM will perform an administrative review on each data deliverable package. The review will determine if requirements of the laboratory and the data deliverable have been met and are complete.

A review of approximately 10 percent of all data deliverable packages from a laboratory QA manager must occur before the administrative review and final release of the data deliverable to verify that all QC requirements of the project and analytical methods have been met. The data packages will be randomly selected for review.

Each step of this review process involves evaluating data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience is essential to provide data of consistently high quality.

Table 5-2. Laboratory Data Validation Qualifiers

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Qualifier	Explanation of Qualifier
U	The compound was analyzed for but was not detected above the reported MDL.
J	The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample, or the reported concentration is greater than the instrument detection limit but less than the QAPP-specified reporting limit.
R	The sample results are rejected because of serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.
UJ	The analyte was not detected above the reported method detection limit. However, the reported limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

Notes:

MDL = method detection limit

QAPP = quality assurance project plan

QC = quality control

5.2 Verification and Validation Methods (D2)

Data validation and verification generated during field and laboratory activities are essential to obtaining data of defensible and acceptable quality. Data to be used in evaluating project technical objectives must be assessed to determine whether the data are of sufficient quality to allow their unrestricted use. It is the joint responsibility of VHA and CDM Smith to verify that the data collected meet the requirements specified in Section 3.

The *Guidance for Evaluating Performance Based Chemical Data* (performance-based [PB] review) (USACE 2005), the *EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review* (EPA 2017a), and the *EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review* (2017b) have been used to develop the data validation specifications for this QAPP. The USACE PB review guidance specifically states, "The data review protocols presented in this document should not be viewed as prescriptive algorithms but as strategies intended for the purposes of guidance." The NFGs have not and cannot be applied prescriptively, as the purpose of the NFGs were to "aid the data viewer in determining the usability of analytical data generated using the United States Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) Statement of Work (SOW)". The methods employed for this RI are beyond the scope of the CLP methods, and no CLP methods are being used. Instead, both the PB review and NFG documents have been used as guide to develop the data validation guidance for the methods utilized, as specified in the paragraphs below and in Section 5.3. Every method and their associated QC indicators have been addressed by the QAPP validation specifications. The data validation specifications provided in the QAPP are designed to produce defensible data of acceptable quality for all QAPP methods.

After sampling is complete and the laboratory has submitted the final data package, CDM Smith will conduct data validation using the calibration and QC requirements specified in Section 3. Data validation will not be performed on the geotechnical, HAPSITE, or other screening-level analyses. A summary of the validation procedure is provided in **Table 5-3** and the data validation flagging criteria is specified in **Tables 5-4** and **5-5**. Ninety percent of all samples analyzed will receive an EPA Stage 3 validation/verification review (EPA 2009), and 10 percent of the samples for each analysis performed will receive an EPA Stage 4 validation/verification review.

Laboratory data validation will be conducted by a CDM Smith project chemist or designee not directly involved with data collection or the site investigation. The EDD provided by the laboratory will be entered into a proprietary Microsoft-Access based data validation software program developed by CDM Smith, and the software program and/or manual validation will be used to identify data that should either be qualified or rejected for project use. Data qualifiers will be reconciled with outputs from the validation, and will be added to the final, validated data. A validation report will be prepared presenting the results of the review and results that required qualification.

Data will be evaluated relative to the criteria established in Section 5.1. The validation qualifiers that will be applied are summarized in **Tables 5-4** and **5-5**. The QC summary report identifies any nonconformances and explains any limitations on data use.

Table 5-3. Data Validation Process Summary for Field and Laboratory Results

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Validation Step Tier 3/4	Validation Input	Description	Responsible for Validation (Name, Organization)
3/4	Field logbook	Sampling protocols followed per the QAPP, RIWP, FSP, appropriate QC samples collected, proper preservation	CDM Smith FTL
3/4	Field analytical results and logbook	Calibrations, blanks, duplicates all recorded and meet criteria	CDM Smith FTL
3/4	Sample location data (GPS coordinates)	Northings, eastings, and elevations traceable to all sample locations	CDM Smith FTL
3/4	Field originated NCRs/CARs	All issues properly documented, corrective actions implemented and effective	CDM Smith FTL
3/4	Field surveillance report	All issues properly documented, corrective actions implemented and effective	CDM smith FTL
3/4	Chain-of-custody forms; sample receiving document	Sample IDs for laboratory data match the reported data, all samples have data reported for requested analysis unless noted in the narrative	CDM Smith Project Chemist/QAM
3/4	Analytical data package Laboratory SOPs/reference methods QAPP MPC	Holding times met all method criteria	CDM Smith Project Chemist/QAM
3/4		Review of dilutions and reanalyses results against reported data; when multiple analyses-appropriate run was reported, proper units are reported	CDM Smith Project Chemist/QAM
3/4		Calibrations analyzed at required frequency and met criteria	CDM Smith Project Chemist/QAM
3/4		Comparison of QC sample results (such as surrogate, ISs, spikes, and blanks) all match criteria in method and QAPP	CDM Smith Project Chemist/QAM
3/4		Blanks free of contamination; if analytes present >RDL samples properly qualified if sample concentration <10× blank concentration	CDM Smith Project Chemist/QAM
3/4		Detection limits, project action limits met	CDM Smith Project Chemist/QAM
4		Recalculation of sample results from the laboratory instrument responses, and comparison of recalculated results to laboratory reported results. Evaluate sample results by checking selected instrument output (e.g., chromatograms, mass spectra, atomic emission spectra, instrument background, interference corrections) for correct identification and quantitation of analytes (e.g., peak integrations, use of appropriate ISs, elution order of analytes, and interferences)	CDM Smith Project Chemist/QAM
3/4	Laboratory surveillance report	All issues properly documented, corrective actions implemented and effective	CDM Smith Project Chemist/QAM
3/4	Laboratory NCRs/CARs	All issues properly documented, corrective actions implemented and effective	Laboratory Project Chemist/QAM
3/4	Memorandum about QAPP modifications	All issues properly documented, corrective actions implemented and effective	CDM Smith Project Chemist/QAM
3/4	Analytical EDDs	All data reported in excel format; EDD verified against hard copy laboratory report	CDM Smith Project Chemist/QAM

Notes:

CAR = corrective action report
EDD = electronic data deliverable
GPS = Global Positioning System
FTL = field team leader
ID = identification
IS = instrument standard
MPC = measurement performance criteria
NCR = nonconformance report
QAPP = quality assurance project plan
QAM = quality assurance manager
RDL = representative detection limit
SOP = standard operating procedure

Table 5-4. Data Validation Flagging Criteria for Organic Methods

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Check	Evaluation	Data Flag	Samples Affected
Holding Time	Holding time exceeded for extraction or analysis	J positive results; UJ nondetects	Sample
	Holding time exceeded by a factor of two (or other applicable criteria based on method)	J positive results; R nondetects	
Sample Preservation	Sample not preserved; or not analyzed within 7 days from sample collection (SW8260C VOA)	J positive results; UJ nondetects; R nondetects based on professional judgement	Sample
Sample Integrity	Bubbles (>pea size) in VOA vial used for analysis	J positive results; UJ nondetects	Sample
Temperature	>6°C	J positive results; UJ nondetects	All samples in same cooler
ICAL	RRF <0.050 but >0.01, RRF <0.010 for poor responders (SW8260C and SW8270D)	J positive results, UJ nondetects J positive results, R nondetects	All associated samples in analysis batch
	Method specified calibration criteria exceeded (e.g., %RSD exceeds criteria AND calibration curve not used; OR calibration curve used, but with coefficient of correlation or determination <0.99)	J positive results, UJ nondetects (use professional judgement for nondetects)	
Calibration Verification (second-source and CCV)	RRF <0.050 but >0.01, RRF <0.010 for poor responders (SW8260C and SW8270D)	J positive results, UJ nondetects J positive results, R nondetects	All associated samples in analysis batch
	%D >20.0% or applicable analyte specific criteria	J positive results, UJ nondetects	
LCS	%R >UCL	J positive results	All samples in preparation batch
	%R <LCL	J positive results, UJ nondetects	
	%R <10%	J positive results; R nondetects	
	Analyte not included in LCS	J positive results, UJ nondetects	
Calibration Blank MB	Evaluate the existence and magnitude of contamination from applicable blank sample	U positive sample results <reporting limit (see specific guidance criteria for organic analyses)	All samples in preparation batch or analytical batch, whichever one applies, associated with MB or calibration blank
EB Field Blank	Evaluate the existence and magnitude of contamination from applicable blank sample	U positive sample results <reporting limit (see specific guidance criteria for organic analyses)	All samples, same site, matrix and date (water) or all samples, same site, matrix (soil) associated with the EB

Table 5-4. Data Validation Flagging Criteria for Organic Methods

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Check	Evaluation	Data Flag	Samples Affected
Trip Blank	Evaluate the existence and magnitude of contamination from applicable blank sample	U positive sample results <reporting limit (see specific guidance criteria for organic analyses)	All samples shipped in the same cooler as the trip blank
MSs			
%R	%R >UCL	J positive results	MS analytes in parent sample and FD if any
	%R <LCL	J positive results, UJ nondetects	
	%R <10%	J positive results; R nondetects	
	Analyte not included in MS or MSD	J positive results; UJ nondetects (if warranted based on technical review)	
RPDs	RPD >UCL	J positive results	
Surrogates			
SW8260C/TO-15	%R >UCL	J+ positive results	All analytes in sample
	%R <LCL and none <10%	J- positive results; UJ nondetects	
	%R <10%	J- positive results; R nondetects	
SW8270D	%R >UCL	J+ positive results	All associated analytes in sample
	%R <LCL but not <10%	J- positive results; UJ nondetects	
	%R <LCL and <10%	J- positive results; R nondetects	
IS	Area response <20% of opening CCV	J+ positive results; R nondetects	Associated analytes in sample
	20% area <area response <50% of opening CCV	J+ positive results; UJ nondetects	
	Area response >200% of opening CCV	J- positive results	
	RT shift between sample/blank and opening CCV from ICAL >10.0 seconds	R positive results; R nondetect results	
FD	Concentration of reported analytes are >5× the reporting limit in either sample and RPD >UCL (30% for water samples; 50% for soil samples; 40% for air samples)	J positive results	FD pair
	One or both sample results <5× the reporting limit and a difference of ± the reporting limit for water and air (±2× for soil)	J positive; UJ nondetect	
Confirmation	RPD between primary and confirmation results >25%	J positive results	Sample

Table 5-4. Data Validation Flagging Criteria for Organic Methods

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Check	Evaluation	Data Flag	Samples Affected

Notes:

All QA/QC criteria are included in Tables 3-2 through 3-15 and will be used for verification criteria.

Organic methods include SW8260C, SW8270SIM, Modified TO-15 SIM, and Modified TO-17.

Spike recovery limits do not apply when the sample concentration exceeds the spike concentration by a factor of 4 or more.

For methods requiring confirmation, the qualification applies to primary analysis results (either of the two columns/detectors may be designated as the primary column/detector).

Where one MS recovery meets acceptance criteria and the other MS of the pair does not, professional judgment may be used to determine if the parent sample should be qualified for matrix effects by comparing the MS recoveries to other QC results within the batch or sample site.

Qualifiers may not apply in cases where a surrogate coelutes with a nontarget analyte.

Qualifiers may not apply in cases where low surrogate recoveries are because of sample dilution.

J = The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample, or the reported concentration is greater than the instrument detection limit but less than the QAPP-specified reporting limit.

J+ = The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample, or the reported concentration is greater than the instrument detection limit but less than the QAPP-specified reporting limit. The result is considered biased high.

J- = The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample, or the reported concentration is greater than the instrument detection limit but less than the QAPP-specified reporting limit. The result is considered biased low.

R = The sample results are rejected because of serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.

U = The compound was analyzed for but was not detected above the reported MDL.

UJ = The analyte was not detected above the reported MDL. However, the reported limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

%D = percent difference
%R = percent recovery
°C = degrees Celsius
CCV = continuing calibration verification
EB = equipment blank
FD = field duplicate
ICAL = initial calibration
LCL = lower control limit
LCS = laboratory control sample
MB = method blank
MDL = method detection limit
MS = matrix spike
QA = quality assurance
QAPP = quality assurance project plan
QC = quality control
RPD = relative percent difference
RRF = relative response factor
RSD = relative standard deviation
UCL = upper control limit
VOA = volatile organic analyte

Table 5-5. Data Validation Flagging Criteria for Inorganic Methods

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Check	Evaluation	Flag	Samples Affected
Holding Time	Holding time exceeded for extraction, digestion, or analysis	J- positive results; R nondetects for mercury; UJ nondetects for all other analytes	Sample
	Holding time for digestion or analysis exceeded by a factor of 2 (or other applicable criteria based on method)	J- positive results; R nondetects	
Sample Preservation	Sample preservation requirements not met (if sample preservation was not done in the field but performed at the laboratory upon sample receipt, no flagging is required)	J- positive results; R nondetects	Sample
Temperature	>6°C (not applicable to metals except mercury)	J positive results; UJ nondetects	Samples in same cooler
ICAL (multipoint only)	Correlation coefficient ≤ 0.995	J positive results; UJ nondetects	All associated samples in analytical batch
Calibration Verification (ICAL verification, CCV)	%R >110, except ICV SM4500-NO^3E (115)	J+ positive results	All associated samples in analytical batch
	%R <90, except ICV SM4500-NO^3E (85)	J- positive results, UJ nondetects	
Interference Check Sample (SW6020 only)	%R >UCL	J+ positive results	All associated samples in analytical batch
	%R <LCL	J- positive results; UJ nondetects	
Low-Level Calibration Verification (SW6020 only)	%R >130	J positive results	All associated samples in analytical batch
	%R <30	J positive results, UJ nondetects	
LCS	%R >UCL	J+ positive results; no action for nondetects	All samples in preparation batch
	%R <LCL	J- positive results; UJ nondetects	
	%R <40% (<20% for antimony or silver)	J- positive results; R nondetects	
	Analyte not included in LCS	J positive results, UJ nondetects	
IS (SW6020A)	Area >125%	J positive results; UJ nondetects	Associated analytes in sample
	Area <60% but not <30%	J positive results; no action for nondetects	
	Area <30%	R positive results; Nondetects use professional judgment – UJ or R	

Table 5-5. Data Validation Flagging Criteria for Inorganic Methods

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Check	Evaluation	Flag	Samples Affected
MB	Evaluate the existence and magnitude of contamination from applicable blank sample	U positive sample results <RL (see specific guidance)	All samples in preparation batch or analytical batch, whichever one applies
Calibration Blank (ICB, CCB)	Evaluate the existence and magnitude of contamination from applicable blank sample	Positive blank detections: U detected sample results <RL (see specific guidance) Negative blank detections (see specific guidance)	All samples that are either before or after the CCB/ICB
EB Field Blank Ambient Blank	Evaluate the existence and magnitude of contamination from applicable blank sample	U positive sample results <RL (see specific guidance)	All samples, same site, matrix and date (water) or all samples, same site, matrix (soil) associated with the EB
MSs			
%R (see specific guidance)	%R <30 PDS %R <75% PDS %R ≥75%	J- positive results R nondetects J positive results UJ nondetects	MS analytes in all associated samples
	%R 30-74% PDS %R <75% PDS %R ≥75%	J- positive results, UJ nondetects J positive results UJ nondetects	
	%R >125% PDS %R >125% PDS %R ≤125%	J+ positive results; no qualifiers for nondetects J positive results; no qualifiers for nondetects	
	%R <30% and no PDS performed (not required for silver)	J- positive results, R nondetects	
	%R 30-74% and no PDS performed (not required for silver)	J- positive results, UJ nondetects	
	%R>125% and no PDS performed (not required for silver)	J+ positive results, no qualifiers for nondetects	
	Analyte not included in MS or MSD	J positive results, UJ nondetects	
RPD	RPD>UCL	J positive results	
Inductively Coupled Plasma Serial Dilution Test	If concentration is >50× the MDL and percent difference >UCL	J positive results, UJ nondetects	All samples in data package associated with serial dilution sample

Table 5-5. Data Validation Flagging Criteria for Inorganic Methods

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Check	Evaluation	Flag	Samples Affected
FD	Concentration of reported analytes are >5× RL in either sample and RPD >UCL (30% for water samples; 50% for soil samples)	J positive results	FD pair
	One or both sample results <5×RL and a difference of ± the reporting limit for water (±2× for soil)	J positive; UJ nondetect	
LCSD	Concentration of reported analytes are >5×RL in either sample and RPD >20%	J positive results	All sample result(s)
	One or both sample results <5×RL and a difference of ± the reporting limit (±2× for soil)	J positive results, UJ nondetects	

Notes:

All QA/QC criteria are included in Tables 3-2 through 3-13 and will be used for verification criteria.

Inorganic methods include SW6020A, SW7470A, SW7471A, and General Chemistry Parameters.

Spike recovery limits do not apply when the sample concentration exceeds the spike concentration by a factor of 4 or more.

Specific Guidance: *National Functional Guidelines for Inorganic Superfund Methods Data Review* (EPA 2017a)

Table 5-5. Data Validation Flagging Criteria for Inorganic Methods

Quality Assurance Project Plan, Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

QC Check	Evaluation	Flag	Samples Affected

J = The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample, or the reported concentration is greater than the instrument detection limit but less than the QAPP-specified reporting limit.

J+ = The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample, or the reported concentration is greater than the instrument detection limit but less than the QAPP-specified reporting limit. The result is considered biased high.

J- = The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample, or the reported concentration is greater than the instrument detection limit but less than the QAPP-specified reporting limit. The result is considered biased low.

R = The sample results are rejected because of serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.

U = The compound was analyzed for but was not detected above the reported MDL.

UJ = The analyte was not detected above the reported MDL. However, the reported limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

%R = percent recovery
°C = degrees Celsius
CCV = continuing calibration verification
EB = equipment blank
ICAL = initial calibration
FD = field duplicate
IS = internal standard
LCL = lower control limit
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
MB = method blank
MDL = method detection limit
MS = matrix spike
PDS = post digestion spike
QA = quality assurance
QAPP = quality assurance project plan
QC = quality control
RPD = relative percent difference
UCL = upper control limit

5.3 Reconciliation with User Requirements (D3)

The CDM Smith QAM is responsible for implementing the system inputs to data quality. All data quality issues concerning field sampling efforts, laboratory analysis, data validation, database management, and data reporting will be referred to the QAM.

5.3.1 Quality Control Summary Report

The CDM Smith project chemist will prepare the QC summary report. This report will include limitations of the data and recommendations on the usability of the laboratory data for decision making. The report findings will be presented so that the completion of project objectives and overall data quality can be verified for the sample results. The data validation and usability report will include the following:

- **Introduction.** Summarizes the purpose of the QA review and validation process and the samples reviewed.
- **Analytical data.** Summarizes the number and types of samples collected, including field QC samples, analytical methods performed, and data evaluation purpose.
- **Findings.** Provides overall summaries of the data validation findings (e.g., only the criteria exceedances that resulted in data qualification are discussed) specific for each analytical method.
- **Overall Assessment.** Provides a summary of the overall completeness of the data set and discusses any limitations in the use of the analytical data.
- Copies of all data validation reports and data quality assessment evaluation.

The QC summary report will assess only the laboratory data packages and EDDs. No assessment of sample collection and associated documentation will be performed as part of this report, since that is addressed in the field systems audit (Section 4.1.2.1). The QC summary report will be provided as an appendix to each data summary report, as well as the RI report.

5.3.2 Project-specific Measurement Performance Criteria

The project-specific measurement performance criteria are summarized in **Tables 3-2** through **3-15**.

Section 6
References

U.S. Army Corps of Engineers (USACE). 2005. *Guidance for Evaluating Performance Based Chemical Data*. Engineer Manual 200-1-10. June.

U.S. Department of Defense (DoD). 2017. *Quality Systems Manual*. Version 5.1. January.

U.S. Environmental Protection Agency (EPA). 1988. *Guidance for Conducting Remedial Investigations and Feasibility Studies under the Comprehensive Environmental Response Compensation and Liability Act (CERCLA)*. Interim Final. October.

U.S. Environmental Protection Agency (EPA). 1996. *Soil Screening Guidance: User's Guide*. EPA/9355.4-23. July.

U.S. Environmental Protection Agency (EPA). 2001. *EPA Requirements for Quality Assurance Project Plans*. EPA QA/R-5. EPA/240/B-01/003. March. Reissued 2006.

U.S. Environmental Protection Agency (EPA). 2002a. *EPA Guidance for Quality Assurance Project Plans*. EPA QA/G-5. EPA/600/R-02/009. December.

U.S. Environmental Protection Agency (EPA). 2002b. *Supplemental Guidance for Developing Soil Screening Levels for Superfund Sites*. EPA/9355.4-24. December.

U.S. Environmental Protection Agency (EPA). 2006. *Guidance on Planning Using the Data Quality Objectives Process*. EPA QA/G-4. Printing Office. Washington, DC. February.

U.S. Environmental Protection Agency (EPA). 2009. *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*. EPA/540/R-08/005. January.

U.S. Environmental Protection Agency (EPA). 2017a. *EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review*. EPA/540/R-13/001. January.

U.S. Environmental Protection Agency (EPA). 2017b. *EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review*. EPA/540/R-014/002. January.

U.S. Environmental Protection Agency (EPA). 2019. *Regional Screening Levels (RSLs) – User's Guide*. Accessed April 23, 2020. https://www.epa.gov/risk/regional-screening-levels-rsls-generic-tables. November.

Appendix A
Field Standard Operating Procedures

Surface Water Sampling	SOP 1-1 Revision: 10 Date: February 2015

Approved : *Signature* **Technical Review:** Curt Coover

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define requirements for collection and containment of surface water samples.

2.0 Background

Surface water samples are collected to determine the type(s) and level(s) of contamination in a particular surface water body and/or its biological disposition.

2.1 Definitions

Surface Water - Water that flows over or rests on the land and is open to the atmosphere. This includes ditches, streams, rivers, lakes, pools, ponds, and basins.

Shallow Surface Water - Water within 1 to 3.3 feet (0.3 to 1 meter) of the surface of a body of water.

Deep Surface Water - Water deeper than 3.3 feet (1 meter) of the surface of a body of water.

Grab Sample - A discrete portion or aliquot taken from a specific location at a given point in time.

Simple Composite - Two or more subsamples taken from a specific media and site at a specific point in time. The subsamples are collected and mixed, and then a single average sample is taken from the mixture.

Temporal Composite - Two or more subsamples taken from a specific media and site over a period of time. The subsamples are collected and mixed, and then a single average sample is taken from the mixture.

Churn Splitter - Large vessel for compositing subsamples. Includes a mechanism to agitate the water to keep solids suspended.

2.2 Associated Procedures

- SOP 1-2, *Sample Custody*
- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-2, *Photographic Documentation of Field Activities*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that field personnel are trained in the use of this SOP, related SOPs, and the required equipment.

Field Team Leader - The field team leader (FTL) is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any other SOPs pertaining to specific media sampling. The FTL also must ensure that the quantity and location of surface water samples collected meet the requirements of the site-specific plans.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

All or part of the equipment listed under the "as needed" category may be required at any specific site, depending on the plan(s) for that site.

- Site-specific plans
- Field logbook
- Indelible black-ink pens and markers
- Labels and appropriate forms/documentation for sample shipment
- Appropriate sample containers
- Insulated cooler and waterproof sealing tape
- Ice bags or "blue ice"
- Plastic zip-top bags
- Clear waterproof tape
- Personal protective clothing and equipment
- Latex or appropriate gloves
- Rubber boots and/or rubberized waders
- Life jacket
- Kimwipe or paper towels
- Clean plastic sheeting
- Tap and deionized water
- Appropriate photographic equipment and supplies
- Appropriate decontamination equipment and supplies

As needed:

- Pond sampler with 1-liter (L) beaker (preferably Teflon®), clamp, and heavy-duty telescoping pole
- Weighted bottle sampler, 1-L capacity (preferably Teflon) and handle; see USGS Open File Report 2005-1087 for selection of sampler; a Kemmerer or Van Dorn sampler may be used if Teflon is not required
- Churn splitter
- Peristaltic pump or suitable replacement
- Temperature, pH, and conductivity meter(s), dissolved oxygen meter, redox potential meter (as required by project plan)
- Boat with depth finder for deep water or inaccessible shorelines
- Global positioning system (GPS) unit
- Tape measure
- Any personal protective equipment specified in the site-specific health and safety plan
- Spare parts for all equipment

5.0 Procedures

5.1 Preparation

The following steps should be taken when preparing for sampling surface water:

1. Review site-specific health and safety plan and project plans before initiating sampling activity.

2. Don the appropriate personal protective clothing as dictated by the site-specific health and safety plan.

3. Select wadeable stream/river sampling locations that exhibit cross-sectional homogeneity and are well-mixed. Avoid areas where the channel is constricted or bends where scouring may have occurred. For lake samples, the investigator should consider the lake stratification caused by seasonal temperature differences. If possible, select a location that can be described precisely, such as xx feet upstream of xx bridge. Use caution when wading streams more than 1 to 2 feet deep. Flowing water can be a safety hazard.

4. Prepare sampling site by laying out clean plastic sheeting on the ground or any flat, level surfaces near the sampling area and place equipment to be used on the plastic.

5. Make field measurements as required by the project plans in physical, chemical, and biological characteristics of the water (e.g., discharge, gage height, temperature, dissolved oxygen, conductivity, pH).

6. The samples shall be collected from areas of least to greatest contamination (when known) and, when collecting several samples in 1 day, always collect from downstream to upstream.

7. The sampler should be facing upstream when sampling, both for proper sample collection and for safety (ability to observe floating objects).

8. Document the sampling events, recording all information in the designated field logbook and take photographs if required or if possible. Document any and all deviations from this SOP and include rationale for changes.

9. The collection points shall be located on a site map and described in the field logbook. Use GPS if required or if possible.

10. Label each sample container with the appropriate information. Secure the label by covering it with a piece of waterproof clear tape.

11. Decontaminate reusable sampling equipment after sample collection according to SOP 4-5.

12. Processes for verifying depth of samples must be included in site-specific project plans.

13. Check that a trip blank/temperature blank, when necessary, is included in the chilled cooler. Quality assurance/quality control sample requirements vary from project to project. Consult the project-specific work plan for quality requirements.

5.2 Shallow Surface Water Sample Collection for Wadeable Streams

5.2.1 Method for Collecting Samples for Volatile Organic Compound Analysis

All volatile organic compound (VOC) samples should be discrete samples. The following steps must be taken when collecting shallow surface water VOC samples:

If the volatile organic analysis (VOA) vials do not require a preservative:

1. Approach the sample location from downstream; do not enter the sample area. Slowly submerge VOA vials completely into an area of gently flowing water and fill. Do not disturb bottom sediments. The open end of the vials should be pointed upstream

Note: When collecting samples for VOC analysis, avoid collecting from a surface water point where water is cascading and aerating.

2. Cap the VOA vial while it is underwater. Be sure to dislodge all air bubbles from the cap before sealing the vial.

3. Turn the capped vial upside down and check for air bubbles. Tap the bottom of the vials to dislodge any bubbles that may have formed around the cap or sides. Discard and resample if bubbles are present.

4. Proceed to Step 5 below.

If the VOA vials require a preservative:

1. Collect a sufficient sample in a clean glass jar as in Steps 1 and 2 above for unpreserved vials. Specific sampling devices to be used must be specified in site-specific plans.

2. Decant the sample immediately into prepreserved VOA vials. It is recommended that the amount of preservative be predetermined on a separate aliquot of sample that is subsequently discarded. Tip vials slightly while filling to reduce turbulence until nearly filled. Then straighten vial to vertical for final filling. Ensure that a meniscus is raised above the lip of the vial before capping.
3. Cap each vial once the meniscus has formed.

4. Turn the capped vial upside down and check for air bubbles. Tap the bottom of the vials to dislodge any bubbles that may have formed around the cap or sides. Discard and resample if bubbles are present.

5. Wipe the outside of sample vials with a Kimwipe or clean paper towel. Affix a completed sample label.

6. Place sample vial(s) in a zip-top plastic bag and seal the bag.

7. Immediately pack all samples into a chilled cooler.

5.2.2 Method for Collecting Discrete Shallow Surface Water Samples for Nonvolatile Organic or Inorganic Compound Analysis

The following steps must be followed when collecting discrete shallow surface water samples for nonvolatile organic or inorganic compound analysis:

1. Directly dip the sample container, with the opening facing upstream, into the surface water and fill. If wading is necessary, approach the sample location from downstream; do not enter the actual sample area. Do not disturb underlying sediments.

2. Filter samples if required by the site-specific plan.

3. Add appropriate preservatives to the sample containers if required and check pH.

Note: Use a separate container when field testing pH, conductivity, temperature, etc. Do not insert pH paper or probe directly into sample container.

4. Cap the sample containers and wipe the outer surfaces of the sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.

5. Place sample container(s) in individual zip-top plastic bags, if possible, and seal the bags.

6. Immediately pack all samples into a chilled cooler.

5.2.3 Method for Collecting Simple Composite Shallow Surface Water Samples for Nonvolatile Organic or Inorganic Compound Analysis

If the QAPP requires the use of simple composite samples, then a sampler capable of collecting composite samples is required. For width and depth integrated (WDI) composite samples, a DH-48 or DH-81 are recommended, but the QAPP may specify an alternative. The following steps must be followed when collecting simple composite shallow surface water samples for nonvolatile organic or inorganic compound analysis:

1. Record the gage height, if any, before and after sampling.

2. Select the number of width increments based on the requirements of the QAPP. Generally, small well mixed streams require few increments while large or poorly mixed streams require more increments.

3. For fewer than six width increments, subsample locations can be visually estimated. For more than five width increments, string a tape measure across the stream above the water surface to be able to accurately identify the subsample locations. Increments should be evenly spaced across the stream for equal width-integrated (EWI) sampling.

4. If depth-integrated sampling is required, collect a subsample at each width increment by submerging the sampler, orifice facing upstream, from the surface to near the bottom and back up to the surface again in an even steady motion. Do not disturb the sediment at the bottom. The sampler should be retrieved less than full. If the sampler is full, empty it and repeat the subsample collection.

5. If depth-integrated sampling is not required, submerge the sampler with the orifice facing upstream into the surface water and fill.

6. Empty the sampler into a churn splitter or temporary container for later splitting.

7. Repeat Steps 4 to 6 for each width increment.

8. If temporary containers were used, empty into churn splitter. Operate the churn splitter by moving the churn up and down in a steady motion fast enough to homogenize the sample without causing aeration. While the churn is in motion, fill the sample bottles from the tap on the churn.

9. Follow Steps 2 though 6 in Section 5.2.2.

5.2.4 Method for Collecting Temporal Composite Shallow Surface Water Samples for Nonvolatile Organic or Inorganic Compound Analysis

If the QAPP requires the use of temporal composite samples, this can be accomplished using a series of discrete samples collected by hand or an automated sampler, or using a series of simple composite samples. Refer to the preceding sections for collecting the subsamples. The compositing scheme can be time-based (e.g., once per hour for 4 hours) or time-discharge (or time gage height) based (e.g., once per hour until the gage height exceeds xx feet, then change to once per 15 minutes).

Because of the project-specific nature of temporal composite sampling, the specific requirements should be identified in the QAPP. The following are general steps to be followed to collect temporal composite samples:

1. Provide for a method of measuring discharge or gage height before, during, and after sample collection as required in the QAPP.

2. Select the number of time increments based on the requirements of the QAPP. If the time increments change based on a change in flow or water quality, specify the trigger, the new time increment, and any additional trigger to return to the previous increment.

3. Calculate the storage volume for the subsamples and provide a churn splitter of adequate size to contain the entire sample to be composited.

4. Collect the samples according to a method described in this SOP or alternate specified in the QAPP.

5. Provide for cold storage of subsamples, if possible. Do not process any subsamples by filtering or preserving unless specified in the QAPP.

6. Following collection of all subsamples, empty the containers into a churn splitter. If discrete data are required including laboratory or field analysis, retain a portion of the subsample.

7. Operate the churn splitter by moving the churn up and down in a steady motion fast enough to homogenize the sample without causing aeration. While the churn is in motion, fill the sample bottles from the tap on the churn.

8. Follow Steps 2 though 6 in Section 5.2.2.

9. Field parameters should be measured in the surface water at the time of collection. Some field parameters can be measured on the subsamples at the time of compositing, but the temperature and temperature-dependant parameters will not be representative.

5.3 Deep Surface Water Sample Collection

5.3.1 Method for Collecting Samples at Specified Depth Using a Weighted Bottle Sampler

The following steps must be followed when collecting surface water samples at specific depths using a weighted bottle sampler:

1. Lower the weighted bottle sampler to the depth specified in the site-specific plan.
2. Remove the stopper by pulling on the sampler line; allow the sampler to fill with water.
3. Release the sampler line to reseat the stopper and retrieve the sampler to the surface.
4. Wipe the weighted bottle sampler dry with a Kimwipe or clean paper towel.
5. Remove the stopper slowly. Fill the specified number of sample containers by slightly tipping the sampler against each sample bottle. Samples to be used for VOC analysis should be decanted directly from the sampler first into prepreserved VOA vials. It is recommended that the amount of preservative be predetermined on a separate aliquot of sample that is subsequently discarded. Add appropriate preservatives to the other sample containers and check pH. Samples may be pooled in stainless steel, glass, or Teflon containers to obtain the necessary volumes. Filter samples if required. Collect sample in separate container for pH, conductivity, temperature, and other measurements if necessary.
6. Close each sample container with the Teflon-lined cap once it is filled. Check for air bubbles in the VOC sample containers. If bubbles are present, discard and resample.
7. Wipe the outside of the sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.
8. Place sample container(s), if possible, in individual zip-top plastic bags, and seal the bags.
9. Immediately pack all samples into a chilled cooler.

5.3.2 Method for Deep Surface Water Sample Collection Using a Peristaltic Pump

The following steps must be followed when collecting deep surface water samples using a peristaltic pump:

1. Install clean medical-grade silicon or Teflon tubing on the pump head. Leave sufficient tubing on the discharge side for convenient dispensing of liquid directly into sample containers.
2. Select the appropriate length of Teflon intake tubing necessary to reach the specified sampling depth. Attach the intake sampling tube to the intake pump tube.
3. Lower the intake tube into the surface water at the specified sampling location to the specified depth; make sure the end of the intake tube does not touch underlying sediments.
4. Start the pump and allow at least three tubing volumes of liquid to flow through and rinse the system before collecting any samples. Do not immediately dispense the purged liquid back to the surface water body. Instead, collect the purged liquid and return it to the source after sample collection is complete.
5. Fill the specified number of sample containers directly from the discharge line. Filter samples if required by the site-specific plan. While filling, allow the liquid to flow gently down the inside of the sample bottle to minimize turbulence.

For VOC samples, fill prepreserved VOA vials and allow a meniscus to form above the top of the container before capping. It is recommended that the amount of preservative be predetermined on a separate aliquot of sample that is subsequently discarded. Check VOA vials to ensure that there are no air bubbles. Add appropriate preservatives to the other samples and check pH.

Note: Use a separate container when field-testing pH, conductivity, temperature, etc. Do not insert pH paper or probe directly into sample container.

6. Cap the sample container(s). Wipe the outside of sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.
7. Place sample container(s) in individual zip-top plastic bags and seal the bags.
8. Immediately pack all samples into a chilled cooler.
9. Drain the pump system, rinse it with deionized water, and wipe it dry. Replace all tubing with new tubing before sampling at another sampling location. Place all used tubing in plastic bags to be discarded or decontaminated according to the site-specific plans.

6.0 Restrictions/Limitations

Peristaltic pumps are generally not capable of lifting water distances greater than 20 to 25 feet (6 to 7.5 meters) above the normal hydrostatic level.

Grab sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration is necessary to minimize sample disturbance and, hence, analyte loss. The representativeness of this sample, however, is difficult to determine because the collected sample represents a single point and has been disturbed.

7.0 References

_____. Region 4. *Operating Procedure, Surface Water Sampling*, Science and Ecosystem Support Division. February 28, 2013 or current revision.

U. S. Geological Survey. *National Field Manual for the Collection of Water-Quality Data*, *Chapter A4*. Version 2.0 September 2006.

_____. A Guide to the Proper Selection and Use of Federally Approved Sediment and Water Quality Samplers. Open-File Report 2005-1087. 2005.

Sample Custody	SOP 1-2 Revision: 8 Date: February 2015

Approved: Signature **Technical Review:** Scott Kirchner

1.0 Objective

Because of the evidentiary nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until their derived data are introduced as evidence in legal proceedings. To maintain and document sample possession, sample custody procedures are followed. All paperwork associated with the sample custody procedures will be retained in CDM Smith files unless the client requests that it be transferred to them for use in legal proceedings or at the completion of the contract.

Note: Sample custody documentation requirements vary with the specific EPA region or client. This technical standard operating procedure (SOP) is intended to present basic sample custody requirements, along with common options. Specific sample custody requirements shall be presented in the project-specific quality assurance (QA) project plan or project-specific modification or clarification form (see Section U-1).

2.0 Background

2.1 Definitions

Sample - A sample is material to be analyzed that is contained in single or multiple containers representing a unique sample identification number.

Sample Custody - A sample is under custody if:

1. It is in your possession
2. It is in your view, after being in your possession
3. It was in your possession and you locked it up
4. It is in a designated secure area

Chain-of-Custody Record - A chain-of-custody record is a form used to document the transfer of custody of samples from one individual to another.

Custody Seal - A custody seal is a tape-like seal that is part of the chain-of-custody process and is used to detect tampering with samples after they have been packed for shipping.

Sample Label - A sample label is an adhesive label placed on sample containers to designate a sample identification number and other sampling information.

Sample Tag - A sample tag is attached with string to a sample container to designate a sample identification number and other sampling information. Tags may be used when it is difficult to physically place adhesive labels on the container (e.g., in the case of small air sampling tubes). Check with your EPA regional Contract Laboratory Program (CLP) coordinator as not all Regions require sample tags.

3.0 General Responsibilities

Sampler - The sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

Field Team Leader - The field team leader (FTL) is responsible for ensuring that strict chain-of-custody procedures are maintained during all sampling events. The FTL is also responsible for coordinating with the subcontractor laboratory to ensure that adequate information is recorded on custody records. The FTL determines whether proper custody procedures were followed during the fieldwork.

Field Sample Custodian - The field sample custodian, when designated by the FTL, is responsible for accepting custody of samples from the sampler(s) and properly packing and shipping the samples to the laboratory assigned to do the analyses. A field sample custodian is typically designated only for large and complex field efforts.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Supplies

- Chain-of-custody records (applicable client or CDM Smith forms)
- Sample labels and/or tags
- Scribe software (if required)
- Custody seals
- Clear tape
- Computer
- Printer and paper

5.0 Procedures

5.1 Chain-of-Custody Record

This procedure establishes a method for maintaining custody of samples through use of a chain-of-custody record. This procedure will be followed for all samples collected or split samples accepted.

Field Custody

1. Collect only the number of samples needed to represent the media being sampled. To the extent possible, determine the quantity and types of samples and sample locations before the actual fieldwork. As few people as possible shall handle samples.

2. Complete sample labels or tags for each sample using waterproof ink.

3. Maintain personal custody of the samples (in your possession) at all times until custody is transferred for sample shipment or directly to the analytical laboratory.

Transfer of Custody and Shipment

1. Complete a chain-of-custody record for all samples (see Figure 1 for an example of a chain-of-custody record. Similar forms may be used when requested by the client). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the sample custodian in the appropriate laboratory.

   - The date/time will be the same for both signatures when custody is transferred directly to another person. When samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures. Common carriers are not required to sign the chain-of-custody record.
   - In all cases, it must be readily apparent that the person who received custody is the same person who relinquished custody to the next custodian.
   - If samples are left unattended or a person refuses to sign, this must be documented and explained on the chain-of-custody record.

   Note: If a field sample custodian has been designated, he/she may initiate the chain-of-custody record, sign, and date as the relinquisher. The individual sampler(s) must sign in the appropriate block, but does (do) not need to sign and date as a relinquisher (refer to Figure 1).

2. Package samples properly for shipment and dispatch to the appropriate laboratory for analysis. Each shipment must be accompanied by a separate chain-of-custody record. If a shipment consists of multiple coolers, a chain-of-custody record shall be filled out for each cooler documenting only samples contained in that particular cooler.

3. The original record will accompany the shipment, and the copies will be retained by the FTL and, if applicable, distributed to the appropriate sample coordinators. Freight bills will also be retained by the FTL as part of the permanent documentation. The shipping number from the freight bill shall be recorded on the applicable chain-of-custody record and field logbook in accordance with SOP 4-1, Field Logbook Content and Control.

Procedure for Completing CDM Smith Example Chain-of-Custody Record

The following procedure is to be used to fill out the CDM Smith chain-of-custody record. The record provided herein (Figure 1) is an example chain-of-custody record. If another type of custody record (i.e., provided by the EPA Contract Laboratory Program (CLP) or a subcontract laboratory or generated by Scribe) is used to track the custody of samples, the custody record shall be filled out in its entirety.

1. Record project number.
2. Record FTL for the project (if a field sample custodian has been designated, also record this name in the "Remarks" box).
3. Record the name and address of the laboratory to which samples are being shipped.
4. Enter the project name/location or code number.
5. Record overnight courier's airbill number.
6. Record sample location number.
7. Record sample number.
8. Note preservatives added to the sample.
9. Note media type (matrix) of the sample.
10. Note sample type (grab or composite).
11. Enter date of sample collection.
12. Enter time of sample collection in military time (24 hour clock).
13. When required by the client, enter the names or initials of the samplers next to the sample location number of the sample they collected.
14. List parameters for analysis and the number of containers submitted for each analysis.
15. Enter appropriate designation for laboratory quality control (e.g., matrix spike/matrix spike duplicate [MS/MSD], matrix spike/duplicate [MS/D]), or other remarks (e.g., sample depth).
16. Sign the chain-of-custody record(s) in the space provided. All samplers must sign each record.
17. If sample tags are used, record the sample tag number in the "Remarks" column.
18. The originator checks information entered in Items 1 through 16 and then signs the top left "Relinquished by" box, prints his/her name, and enters the current date and time (military).
19. Send the top two copies (usually white and yellow) with the samples to the laboratory; retain the third copy (usually pink) for the project files. Retain additional copies for the project file or distribute as required to the appropriate sample coordinators.
20. The laboratory sample custodian receiving the sample shipment checks the sample label information against the chain-of-custody record. Sample condition is checked and anything unusual is noted under "Remarks" on the chain-of-custody record. The laboratory custodian receiving custody signs in the adjacent "Received by" box and keeps the copy. The white copy is returned to CDM Smith.

5.2 Sample Labels and Tags

Unless the client directs otherwise, sample labels or tags will be used for all samples collected or accepted for CDM Smith projects.

1. Complete one label or tag with the information required by the client for each sample container collected. A typical label or tag would be completed as follows (see Figure 2 for example of sample tag; labels are completed with the equivalent information):

   - Record the project code (i.e., project or task number).
   - Enter the station number (sample number or EPA CLP identification number) if applicable.
   - Record the date to indicate the month, day, and year of sample collection.
   - Enter the time (military) of sample collection.
   - Place a check to indicate composite or grab sample.

- Record the station (sample) location.
- Sign in the space provided.
- Place a check next to "yes" or "no" to indicate if a preservative was added.
- Place a check under "Analyses" next to the parameters for which the sample is to be analyzed. If the desired analysis is not listed, write it in the empty slot. Note: Do not write in the box for "laboratory sample number."
- Place or write additional relevant information under "Remarks."

2. Place adhesive labels directly on the sample containers. Place clear tape over the label to protect from moisture.

3. Securely attach sample tags to the sample bottle if required. On 2.27 liter (80 oz.) amber bottles, the tag string may be looped through the ring-style handle and tied. On all other containers, it is recommended that the string be looped around the neck of the bottle, then twisted, and relooped around the neck until the slack in the string is removed. In some instances, when the tag cannot be physically attached to the sample container, it is acceptable to simply place the sample tag in the zip lock bag with a sample container.

4. Double-check that the information recorded on the sample label or tag is consistent with the information recorded on the chain-of-custody record.

5.3 Custody Seals

Two custody seals must be placed on opposite corners of all shipping containers (e.g., cooler) before shipment. The seals shall be signed and dated by the shipper.

Custody seals may also be required to be placed on individual sample bottles. Check with the client or refer to EPA regional guidelines for direction. In these instances the custody seal is placed over or in some cases around the lid or cap of the sample container.

5.4 Sample Shipping

SOP 2-1, *Packaging and Shipping Environmental Samples* defines the requirements for packaging and shipping environmental samples.

6.0 Restrictions/Limitations

Check with the EPA region or client for specific guidelines. If no specific guidelines are identified, this procedure shall be followed.

For EPA CLP sampling events, combined chain-of-custody/traffic report forms generated with Scribe or other EPA-specific records may be used. Refer to regional guidelines for completing these forms.

The EPA Scribe software may be used to customize sample labels and custody records when directed by the client or the CDM Smith project manager.

7.0 References

U. S. Army Corps of Engineers. 2001 or current revision. *Requirements for the Preparation of Sampling and Analysis Plan*, EM 200-1-3. Appendix F. February.

U. S. Environmental Protection Agency. Revised March 1992 or current revision. *National Enforcement Investigations Center, Multi-Media Investigation Manual*, EPA-330/9-89-003-R. p.85.

_____. 2015. Scribe Manuals. http://www.ertsupport.org/scribe_home.htm and http://www.epaosc.org/scribe

_____. 2002 or current revision. *EPA Guidance for Quality Assurance Project Plans*, EPA QA/G-5, EPA/240/R-02/009. Section 2.2.3. December.

_____. 2014 or current revision. *Sampler's Guide, Contract Laboratory Program Guidance for Field Samplers*, EPA-540-R-09-03. January.

Figure 1
Example CDM Smith Chain-of-Custody Record

125 Maiden Lane, 5th Floor
New York, NY 10038
(212) 785-9123
Fax: (212) 785-6114

CHAIN OF CUSTODY RECORD

PROJECT ID.	FIELD TEAM LEADER	LABORATORY AND ADDRESS	DATE SHIPPED
PROJECT NAME/LOCATION		LAB CONTRACT:	AIRBILL NO.

MEDIA TYPE
1. Surface Water
2. Groundwater
3. Leachate
4. Field QC
5. Soil/Sediment
6. Oil
7. Waste
8. Other _____

PRESERVATIVES
1. HCl, pH <2
2. HNO3, pH <2
3. NaOH, pH >12
4. H2SO4, pH <2
5. Zinc Acetate, pH >9
6. Ice Only
7. Not Preserved
8. Other _____

SAMPLE TYPE
G = Grab
C = Composite

SAMPLE LOCATION NO.	LABORATORY SAMPLE NUMBER	PRESER-VATIVES ADDED	MEDIA TYPE	SAMPLE TYPE	20__ DATE	TIME SAMPLED	ANALYSES (List no. of containers submitted)								REMARKS (Note if MS/MSD)
1.															
2.															
3.															
4.															
5.															
6.															
7.															
8.															
9.															
10.															

SAMPLER SIGNATURES:

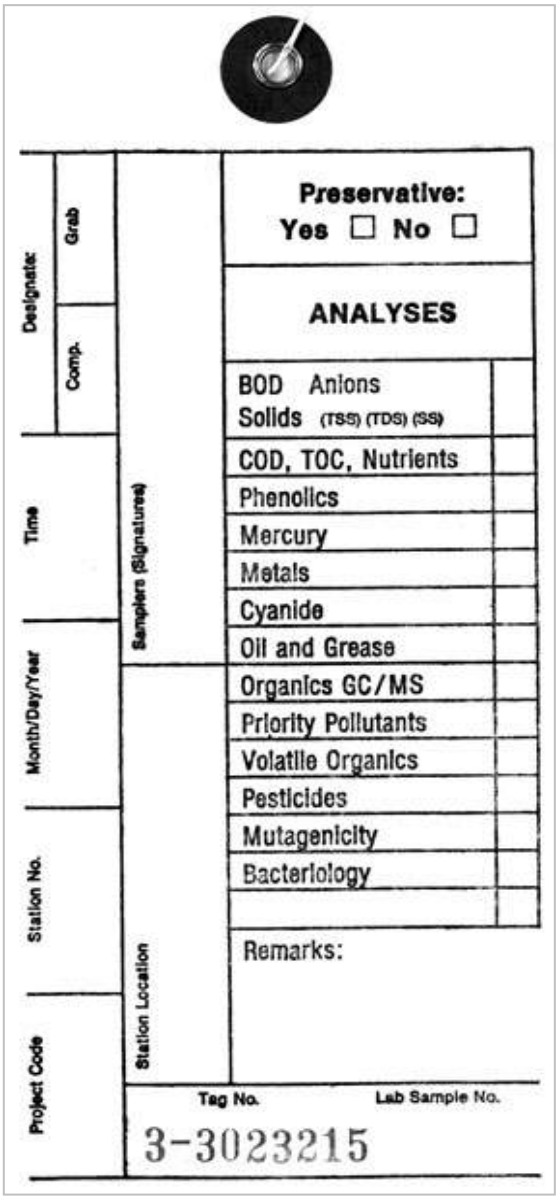
RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)	DATE/TIME	RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)	DATE/TIME
(SIGN)		(SIGN)		(SIGN)		(SIGN)	
RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)	DATE/TIME	RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)	DATE/TIME
(SIGN)		(SIGN)		(SIGN)		(SIGN)	

COMMENTS:

DISTRIBUTION: White and yellow copies accompany sample shipment to laboratory; yellow copy retained by laboratory. Pink copy retained by samplers. 1/98

Note: If requested by the client, different chain-of-custody records may be used. Copies of the template for this record may be obtained from the Chantilly Graphics Department.

Figure 2
Example Sample Tag



Note: Equivalent sample labels or tags may be used.

Surface Soil Sampling	SOP 1-3 Revision: 9 Date: February 2015

Approved: *Signature* **Technical Review:** Mike Valentino

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define the general techniques and requirements for the collection of surface soil samples.

2.0 Background

The techniques and protocols described herein may be used to collect other surface media, including sediment and sludge.

2.1 Definitions

Grab Sample - A discrete portion of sample material or an aliquot taken from a specific sample location at a given point in time.

Spoon/Scoop - A small stainless steel, Teflon®, or Teflon® -lined utensil measuring approximately 15 cm (6 inches) in length with a stem-like handle (for manual operation). Samples are collected using a scooping action.

Surface Soil - Soils generally defined as the soils extending from ground surface to approximately 30 centimeters (cm), or approximately one foot, below ground surface (bgs). Surface soil samples are frequently collected from 0 to 15 cm (0 to 6 inches) bgs. Depending on the soil interval sampled will vary.

Syringe - A hand-held, T-shaped, disposable plastic sampling device used to obtain undisturbed, unconsolidated material samples (e.g., soil or sediment) for laboratory analyses. Samples are collected by pushing the open end of the sampling device into the material to be sampled to retrieve a discrete sample, typically in the amount of 5 or 10 grams.

Trowel - A small stainless steel or Teflon or Teflon® -lined shovel measuring approximately 15 to 20 cm (6 to 8 inches) in length with a slight (approximately 140°) curve across the length. The trowel has a stem-like handle (for manual operation). Samples are collected with a scooping action.

2.2 Associated Procedures

- SOP 1-2, *Sample Custody*
- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.3 Discussion

Surface soil samples are collected to determine the type(s) and level(s) of contamination in soil and often provide information important to the completion of risk assessments for a given site. Surface soil samples may be collected as part of a site investigation or as part of a site-specific sampling plan, and/or as a screen for “hot spots”, which may require more extensive sampling based on the results of the initial surface soil sampling.

Sediment(s) and sludge(s) that have been exposed by evaporation, stream rerouting, or any other means are collected by the same methods as those for surface soil(s). Typically the top 1 to 2 cm of material are carefully removed before collection of the sample. If a thick, matted root zone is encountered at or near the surface, it shall be removed before collecting the representative soil sample. Surface soil, exposed sediment, or sludge is collected using stainless steel and/or Teflon-lined trowels or scoops.

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any other SOPs pertaining to the sampling of specific media. The site manager must also ensure that the quantity and location of surface soil samples collected meet the requirements of the site-specific sampling plan.

Field Team Leader - The field team leader is responsible for ensuring that field personnel collect surface soil samples in accordance with this SOP and other relevant guidance for surface soil sampling.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

- Insulated cooler and clear waterproof sealing tape
- Securely-sealed bags of ice or "blue ice" packs
- Nitrile or other appropriate protective gloves
- Plastic zip-top bags
- Personal protective clothing and equipment
- Stainless steel and/or Teflon-lined spatulas and pans, trays, or bowls
- Plastic sheeting (disposable, protective ground cover)
- Stainless steel and/or Teflon-lined trowels or spoons/scoops (or other equipment as specified in the site-specific sampling plan)
- Appropriate project documents (including sampling or work plan, and health and safety plan)
- Appropriate sample containers
- Field logbook
- Indelible black ink pen and/or marker
- Sample chain-of-custody forms
- Custody seals
- Decontamination supplies
- Paper towels or Kimwipes

Additional equipment is discussed in Section 5.2.2, VOC Field Sampling/Preservation Methods.

5.0 Procedures

5.1 Preparation

The following steps must be followed when preparing for sample collection:

1. Review site-specific health and safety plan and project plans before initiating sampling activity.

2. Don the appropriate personal protective clothing as specified in the site-specific health and safety plan.

3. Locate sampling location(s) in accordance with project documents (e.g., work plan) and document pertinent information in the field logbook. When possible, reference sampling locations back to known, existing site features such as buildings, roads, intersections, etc.

4. Processes for verifying sample collection depth must be specified in the site-specific sampling or work plans.

5. Place clean plastic sheeting on a flat, level surface near the sampling area, if possible, and place sampling equipment on the plastic; place the insulated cooler(s) on separate plastic sheeting to avoid the potential for any cross-contamination.

6. A clean, new or sufficiently decontaminated trowel, scoop, or spoon will be used to obtain sample material from each specified sample location. Other equipment may be used (e.g., shovels) to collect sample material if constructed of stainless steel and decontaminated appropriately prior to use.

5.2 Sample Collection

The following general steps must be followed when collecting surface soil samples.

1. Wear new, clean gloves during handling of all sample containers and sampling devices. Change out gloves at each sampling location, or each time a new sample is to be collected, to avoid cross-contamination.

2. Surface soil samples are typically collected from the areas of least contamination to the areas of the greatest contamination, if known.

3. Document the sampling process by recording applicable information in the designated field logbook. Document any and all deviations from SOPs and the sampling plan in the field logbook and include rationale for changes. See SOP 4-1 for guidance on entering information into field log books.

4. During sample collection, record observations made of the geologic features of the soil or sediments per American Society for Testing and Materials (ASTM) D 2448 (Standard Practice for Description and Identification of Soils (Visual-Manual Procedure) in the field logbook.

5. Carefully remove stones, vegetation, snow, etc. from the ground surface in the sampling location area. Clear the sample location using a new and/or appropriately decontaminated spoon, scoop, or other tool as described to expose a fresh sampling surface.

6. Collect the required sample aliquot for volatile analyses, as appropriate, as well as any other samples that may be degraded by aeration, followed by the collection of samples for other analyses. Note that samples are typically not collected from ground surface to approximately six (6) to twelve (12) inches below ground surface for volatile organic compound analysis because of the potential for volatile loss. Sample collection and preservation techniques, as appropriate, are discussed in the following subsections of this SOP.

7. Store samples at 4° Celsius (C) (±2°C) until samples are delivered to the designated analytical laboratory. An appropriate amount of ice or number of cold packs should be used according to the number of samples and/or the volume of sample material collected in order to ensure that a temperature of 4°C is achieved and maintained for delivery to the analytical laboratory. Sample holding times shall be determined with the appropriate analytical laboratory.

8. Pack all samples as required by the work plan and/or laboratory requirements. Include properly completed documentation and affix signed and dated custody seals to the cooler lid. See SOPs 1-2 and 2-1 for guidance on sample custody procedures and packaging and shipping environmental samples.

9. Decontaminate sampling equipment between sample locations. See SOP 4-5 for guidance on decontamination of field equipment at non-radioactive sites.

5.2.1 Method for Collecting Samples for Nonvolatile Organic or Inorganic Compound Analysis

The requirements for collecting samples of surface soil for nonvolatile organic or inorganic analyses are as follows:

1. Wear new, clean gloves during handling of all sample containers and sampling devices. Change out gloves at each sampling location, or each time a new sample is to be collected, to avoid cross-contamination.

2. Clear the area to be sampled of debris as described in Section 5.2 of this SOP. Determine sample depth as described in the sampling plan.

3. Label each sample container with the appropriate sample collection information.

4. Use a decontaminated stainless steel or Teflon-lined trowel or spoon to obtain sufficient sample material from the required interval and subsampling points, if necessary, to fill the specified sample containers.

5. Empty the contents of the sampling device directly into a clean stainless steel or Teflon-lined tray or bowl.

6. Homogenize the sample by mixing with a spoon, spatula, or trowel.

7. Use the spoon, spatula, or trowel to distribute the mixture into the labeled sample containers. Fill organic sample containers first, then inorganic sample containers.

8. Secure the respective cap on each sample container immediately after filling.

9. Wipe the sample containers with a clean paper towel or Kimwipe to remove any residual soil from the sample container surface.

10. Place sample containers in individual zip-top plastic bags and seal the bags.

11. Store samples at 4° C (±2°C) until samples are delivered to the designated analytical laboratory

12. Pack all samples as required by the work plan and/or laboratory requirements. Include properly completed documentation and affix signed and dated custody seals to the cooler lid. See SOPs 1-2 and 2-1 for guidance on sample custody procedures and packaging and shipping environmental samples.

13. Decontaminate all non-disposable sampling equipment in accordance with SOP 4-5.

5.2.2 Method for Collecting Soil Samples for Volatile Organic Compound Analysis

The requirements for collecting grab samples of surface soil for volatile organic compounds (VOCs) or other samples degraded by aeration are as follows:

1. VOC samples shall be collected with the least disturbance to the soil as possible. When grab sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration, it is necessary to minimize sample disturbance and consequently minimize analyte loss. The representativeness of a VOC grab sample is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed.

2. VOC samples shall be collected as grab samples as discussed in section 5.2.2 of this SOP. Although the method of collection may vary from site to site based on data quality objectives and the degree of known or suspected contamination, collection of samples for VOC analysis should follow the sampling and preservation methodology as described below.

3. Complete the sample label by filling in the appropriate information (e.g., sample identification, date and time of sample collection, and requested analyses) and securing the label to the container.

4. Use a clean stainless steel or Teflon-lined trowel or spoon/scoop to collect sufficient material in one grab to fill the sample containers.

5. With the aid of a clean stainless steel spatula, quickly fill the sample containers directly from the sampling device, removing stones, twigs, grass, etc., from the sample material, as needed. Fill the sample containers, compacting the sample material as much as possible to minimize headspace in each of the containers.

6. Immediately secure the Teflon-lined cap(s) on the sample container(s).

7. Wipe the containers with a clean Kimwipe or paper towel to remove any residual soil from the exterior of the container.

8. Place the sample containers in individual zip-top plastic bag(s) and seal the bag(s).

9. Store samples at 4° Celsius (C) (±2°C) until samples are delivered to the designated analytical laboratory. Determine sample holding times with the appropriate analytical laboratory.

10. Pack all samples as required by the work plan and/or laboratory requirements. Include properly completed documentation and affix signed and dated custody seals to the cooler lid. See SOPs 1-2 and 2-1 for guidance on sample custody procedures and packaging and shipping environmental samples.

11. Decontaminate all non-disposable sampling equipment in accordance with SOP 4-5.

Note: A trip blank shall be included with sample coolers containing VOC samples. QC sample requirements vary from project to project. Consult the project-specific sampling plan for requirements.

5.2.3 VOC Field Sampling/Preservation Methods

The following four sections contain SW-846 test methods for sampling and field preservation of soil samples for VOC analysis. These methods include the EnCore™ Sampler Method for low-level VOC analyses, EnCore Sampler Method for high-level VOC analyses, acid preservation for low-level VOC analyses, and methanol preservation for high-level VOC analyses. Equipment requirements in addition to the equipment specified in Section 4.0 of this SOP for each method are indicated at the beginning of each subsection as follows

When collecting soil samples using the EnCore Sampler Method, collection of soil for moisture content analysis is required. Results of the moisture analysis are used to adjust "wet" concentration results to "dry" concentrations to meet analytical method requirements.

Note: Some variation from these methods may be required depending on the contracted analytical laboratory. For example, sample volume requirements are general requirements. Actual sample volumes, sizes, and quantities may vary depending on client or laboratory requirements.

5.2.3.1 EnCore Sampling Equipment and Collection for Low Level VOC Analyses (<200 µg/kg)

The following equipment is required for low-level analysis:

- Three new 5-gram (g) EnCore samplers
- One 110-milliliter (mL) (4-ounce) wide-mouth glass jar or applicable container for moisture analysis
- One EnCore sampler T-handle

The requirements for collecting samples for low level analysis (<200 µg/kg) of VOCs by the EnCore Sampler Method are as follows:

1. Wear new, clean gloves during handling of all sample containers and sampling devices. Change out gloves at each sampling location, or each time a new sample is to be collected, to avoid cross-contamination.

2. Clear the area to be sampled of debris as described in Section 5.2 of this SOP. Determine sample depth as described in the sampling plan.

3. Remove EnCore sampler and cap from package and attach T-handle to sampler body.

4. Push the sampler into the freshly-exposed sampling surface until the O-ring is visible within the hole on the side of the T-handle. If the O-ring is not visible within this window, then the sampler is not full.

5. Extract the sampler and wipe the sampler head with a clean paper towel or Kimwipe so that the sampler cap can be tightly attached.

6. Push the sampler cap on the head of the sampler with a twisting motion to secure it to the sampler body.

7. Rotate the sampler stem counterclockwise until the stem locks in place to retain the sample within the sampler body.

8. Fill out the sample label with the appropriate sample information (e.g., sample identification, date/time of sample collection, requested analyses) and attach to sampler.

9. Repeat procedure for each of the remaining two samplers.

10. Collect a representative moisture sample in a 110 milliliter (mL) (4-ounce) wide-mouth jar using a new or clean Teflon-lined stainless steel spoon, scoop, or trowel.

11. Store samples at 4° C (±2°C) until samples are delivered to the designated analytical laboratory Samples must be shipped and delivered to the analytical laboratory for extraction within 48 hours.

12. Pack all samples as required by the work plan and/or laboratory requirements. Include properly completed documentation and affix signed and dated custody seals to the cooler lid. See SOPs 1-2 and 2-1 for guidance on sample custody procedures and packaging and shipping environmental samples.

13. Decontaminate all non-disposable sampling equipment in accordance with SOP 4-5.

Note: Verify analytical laboratory requirements for extraction/holding times.

5.2.3.2 Acid Preservation Equipment and Sampling Requirements for Low Level VOC Analyses (<200 µg/kg)

Note: Although not common, acid preservation may be required and should only be performed if specified in the project specific sampling plans. If required, determine the specific field acid preservation procedure based on the requirements specified in the analytical method to be employed. Variations between analytical methods exist with respect to field acid preservation.

The following equipment and supplies are required if field acid preservation is required:

- One 40-mL VOA vial with acid preservation (for field testing of soil pH)
- Two preweighed 40-mL VOA vials with acid preservative and stir bar (for lab analysis)
- Two preweighed 40-mL VOA vials with water and stir bar (in case samples cannot be pre-preserved)
- One pre-weighed jar containing methanol or a pre-weighed empty jar accompanied by a pre-weighed VOA vial containing methanol (for sample screening and/or high level VOC analysis)
- One 110-mL (4-oz) wide-mouth glass jar or other container appropriate for retaining a representative sample for moisture analysis
- One 55-mL (2-oz) jar containing acid preservative (additional acid may be needed because of high soil pH)
- One appropriately sized, non-reactive scoop or measuring spoon capable of delivering 1 g of solid sodium bisulfate
- pH paper
- Weighing scale capable of reading to 0.01 g
- Set of balance weights used in daily balance calibration
- Sodium bisulfate acid solution ($NaHS0_4$)
- A plastic syringe or other sampling device capable of collecting a sufficient sample volume of approximately 5 g

Testing Effervescing Capacity of Soils

Soils must be tested with acid to determine the amount of effervescing that will occur when preserved with acid. Effervescing will drive off VOCs as well as create a high pressure in a sealed VOA vial that could result in the explosion of the sample container. The following steps provide information on the effervescing capacity of the soil.

1. Wear new, clean gloves during handling of all sample containers and sampling devices. Change out gloves at each sampling location, or each time a new sample is to be collected, to avoid cross-contamination.

2. Clear the area to be sampled of debris as described in Section 5.2 of this SOP. Determine sample depth as described in the sampling plan.

3. Using a new, clean syringe, pace approximately 5 g of soil into a VOA vial that contains acid preservative and no stir bar.

4. Do not cap this vial as it may EXPLODE upon interaction with the soil.

5. Observe the sample for gas formation, or effervescence (bubbles that form due to the interaction of carbonates in the soil with the acid preservative).

6. If vigorous or sustained effervescence is observed, then acid is not an acceptable preservative for the sample.
   - In this case the samples need to be collected in the VOA vials containing only water as a preservative and a stir bar. The vials with acid preservative CANNOT be used.

7. If a small amount or no effervescence occurs, then acid is acceptable to preserve the sample. Keep this initial testing VOA vial for use in the buffering test as detailed below.
   - In this case the samples need to be collected in the VOA vials containing acid preservative and a stir bar.

Testing Buffering Capacity of Soils

The soils must be tested to determine the quantity of acid that is required to achieve a pH reading of ≤2 standard units (SUs). The following steps will assist in determining this quantity.

1. If acid preservation is acceptable for sampling soils, then the sample vial that was used to test the effervescing capacity of the soils can be used to test the buffering capacity.

2. Wear new, clean gloves during handling of all sample containers and sampling devices. Change out gloves at each sampling location, or each time a new sample is to be collected, to avoid cross-contamination.

3. Clear the area to be sampled of debris as described in Section 5.2 of this SOP. Determine sample depth as described in the sampling plan.

4. Cap the VOA vial containing the 5 g of soil, acid preservative, and no stir bar as retained during Step 7 of the effervescing test as described above.

5. Shake the VOA vial gently to homogenize the contents.

6. Open the VOA vial and test the pH of the acid solution with pH paper by dipping one end of a pH paper strip into the soil/acid solution.
   - If the pH paper indicates a pH below 2, then samples can be collected in the two preweighed 40-mL VOA vials with the acid preservative and stir bar. As the pH reading is below 2, it is not necessary to add additional acid to the VOA vials.
   - If the pH paper indicates a pH above 2, then additional acid needs to be added to the VOA vial.

7. To add acid to a sample with a pH above 2, measure out 1 g of the solid sodium bisulfate acid and add to the appropriate VOA vial.

8. Cap the VOA vial and shake thoroughly.

9. After an additional 1 g of solid sodium bisulfate has been added to the VOA vial containing sample material with a pH above 2, repeat Step 4.
   - If the pH paper reads below 2, then the samples can be collected in the two preweighed 40-mL VOA vials containing acid preservative, a stir bar, and 1 g of sodium bisulfate.
   - On the Chain of Custody and in the field log book, note that one additional gram of acid was added such that the laboratory can analyze the samples accordingly.
   - If the pH paper reads above 2, repeat Steps 5 through 7 until the sample pH is less than or equal to 2 SUs.

After the soil chemistry has been determined, samples can be collected. The procedure summarized below assumes the appropriate acid- or water-preserved VOA vials are used based on the guidance discussed.

Sample Preservation Steps

1. Wear new, clean gloves during handling of all sample containers and sampling devices. Change out gloves at each sampling location, or each time a new sample is to be collected, to avoid cross-contamination.

2. Add more acid to the sample if necessary (based on the buffering capacity testing discussed in the previous section).

3. Collect an approximately 5-g soil sample using a cutoff plastic syringe or other sampling device designed to obtain 5 g of soil from a freshly exposed sampling surface.

4. Carefully wipe exterior of sample collection device with a clean paper towel or Kimwipe.

5. Transfer the sample from the sample collection device to the appropriate VOA vial, using caution when extruding the sample to prevent splashing the acid outside of the vial.

6. Remove any soil from the threads of the VOA vial using a clean paper towel or Kimwipe.

7. Cap the VOA vial and weigh the jar to the nearest 0.01 g.

8. Record the exact weight on the sample label.

9. Repeat this sampling procedure for the duplicate VOA vial.

10. Weigh the VOA vial containing methanol preservative to the nearest 0.01 g. If the weight of the vial with methanol varies by more than 0.01 g from the original weight recorded on the vial, discard the vial. If the weight is within tolerance, it can be used for soil preservation as discussed below.

11. Take a clean, empty sample jar or the jar that contains the methanol preservative and collect a 5-g or 25-g sample using a cutoff plastic syringe or other coring device designed to deliver 5 g or 25 g of soil from a freshly exposed sample surface. The 5-g or 25-g size is dependent on the client or analytical laboratory requirements, or as specified in the sampling plan.

12. Carefully wipe the exterior of the collection device with a clean paper towel or Kimwipe.

13. Transfer the soil to a clean, empty jar or a VOA vial that contains methanol. If extruding into a jar that contains methanol, be careful not to splash the methanol outside of the sample container.

14. If the jar used to collect the soil sample did not contain preservative before the soil was added, immediately preserve with the methanol provided, using only one vial of methanol preservative per sample jar.

15. Remove any soil from the threads of the VOA vial using a clean paper towel or Kimwipe and cap the vial.

16. Weigh the jar with sample to the nearest 0.01 g and record the weight on the sample label.

17. Collect dry weight sample using a clean stainless steel spoon or trowel.

18. Store samples at 4°C ($\pm$2°C) until samples are delivered to the designated analytical laboratory

19. Pack all samples as required by the work plan and/or laboratory requirements. Include properly completed documentation and affix signed and dated custody seals to the cooler lid. See SOPs 1-2 and 2-1 for guidance on sample custody procedures and packaging and shipping environmental samples.

20. Decontaminate all non-disposable sampling equipment in accordance with SOP 4-5.

5.2.3.3 EnCore Sampling Equipment and Sampling Requirements for High Level Analysis (≥200 µg/kg)

The following equipment is required for high-level analysis:

- One 5-g sampler or one 25-g sampler

Note: The volume requirements specified are general requirements. Actual sample volumes, container sizes, and quantities may vary depending on client or laboratory requirements.

- One 110-mL (4-oz) wide-mouth glass jar or applicable container specified for moisture analysis
- One T-handle EnCore sampler

The requirements for collecting high level analysis by the EnCore Sampler Method are as follows:

1. Wear new, clean gloves during handling of all sample containers and sampling devices. Change out gloves at each sampling location, or each time a new sample is to be collected, to avoid cross-contamination.

2. Clear the area to be sampled of debris as described in Section 5.2 of this SOP. Determine sample depth as described in the sampling plan.

3. Remove the EnCore sampler and cap from package and attach the T-handle to sampler body.

4. Push the sampler into freshly exposed soil surface until the O-ring is visible within the hole/window on the side of the T-handle. If the O-ring is not visible within the window/hole, then the sampler is not full.

5. Use a clean paper towel or Kimwipe to wipe the sampler head so that the cap can be tightly attached.

6. Push the sampler cap on the sampler head with a twisting motion to secure it to the sampler body.

7. Fill out the sample label and attach it to sampler.

8. Rotate the sampler stem counterclockwise until the stem locks in place to retain the sample within the sampler body.

9. Collect a representative moisture sample in 110-mL (4-oz) wide-mouth glass jar or designated container using a clean stainless steel spoon or trowel.

10. Store samplers at 4°C (±2°C) until samples are delivered to the designated analytical laboratory. Samples must be shipped and delivered to the analytical laboratory for extraction within 48 hours.

11. Pack all samples as required by the work plan and/or laboratory requirements. Include properly completed documentation and affix signed and dated custody seals to the cooler lid. See SOPs 1-2 and 2-1 for guidance on sample custody procedures and packaging and shipping environmental samples.

12. Decontaminate all non-disposable sampling equipment in accordance with SOP 4-5.

Note: Verify requirements for extraction/holding times.

5.2.3.4 Methanol Preservation Equipment and Sampling Requirements for High Level Analyses (≥200 µg/kg)

The following equipment is required for high-level analysis:

- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied by a pre-weighed VOA via that contains methanol (laboratory grade)
- Cutoff plastic syringe or other sampling device to obtain 5 g or 25 g of soil
- Set of balance weights used in daily balance calibration
- One dry weight cup
- Weighing balance that accurately weighs to 0.01 g

The requirements for sampling and preservation are as follows:

1. Wear new, clean gloves during handling of all sample containers and sampling devices. Change out gloves at each sampling location, or each time a new sample is to be collected, to avoid cross-contamination.

2. Clear the area to be sampled of debris as described in Section 5.2 of this SOP. Determine sample depth as described in the sampling plan.

3. Weigh the VOA vial containing methanol preservative to the nearest 0.01 g. If the weight of the VOA vial containing methanol varies by more than 0.01 g from the original weight recorded on the vial, discard the vial. If the weight is within tolerance, it can be used for soil preservation/collection as described below. (Commercial sources are available which supply pre-preserved and tared vials which eliminates the need to transport and handle larger quantities of methanol and eliminates the need for a precision scale vials with preservative).

4. Quickly collect a 5-g or 25-g sample using a plastic syringe or other sampling device designed to obtain 5 g or 25 g of soil from a freshly exposed sampling surface.

5. Carefully wipe the exterior of the collection device with a clean paper towel or Kimwipe.

6. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol, be careful not to splash the methanol outside of the vial. The type of jar used and the sample volume needed is dependent on the client or laboratory requirements.

7. If the jar used to collect the soil sample was empty before the soil was added, immediately preserve with the methanol provided, using only one vial of methanol preservative per sample jar.

8. Remove any soil from the exterior of the vial using a clean paper towel or Kimwipe and cap the sample container.

9. Weigh the jar containing the soil to the nearest 0.01 g and record the weight on the sample label.

10 Collect a dry weight sample in a clean, unpreserved sample container using a clean stainless steel spoon or trowel.

11. Store samples at 4°C ($\pm$2°C) until samples are delivered to the designated analytical laboratory.

12. Pack all samples as required by the work plan and/or laboratory requirements. Include properly completed documentation and affix signed and dated custody seals to the cooler lid. See SOPs 1-2 and 2-1 for guidance on sample custody procedures and packaging and shipping environmental samples.

13. Decontaminate all non-disposable sampling equipment in accordance with SOP 4-5.

14. If dropping the samples off at the analytical laboratory or requesting a sample pick-up is not an option, sample containers may need to be shipped to the analytical laboratory. Samples should be packed with ice packs sufficient to maintain a temperature of 4° C in the cooler, and shall be shipped in accordance with Department of Transportation (DOT) regulations. Consult CDM Smith's Health and Safety website (http://cdmweblegacy/h&s/hazmat_transport.html) for guidance on shipping hazardous materials.

6.0 Restrictions/Limitations

As presented in Section 5.2 of this SOP, when grab sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration, it is necessary to minimize sample disturbance and consequently minimize analyte loss. The representativeness of a VOC grab sample is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed.

7.0 References

U. S. Environmental Protection Agency. *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001. December 1987 or current revision.

_____. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846)*, Third Edition, November 1986, (as amended by Updates I, II, IIA, IIB, III, and IIIA, IIIB, and IVA, IVB). Method 5035 (***Note***: § 6.2.1.8 of this method says samples stored in EnCore™ samplers shall be analyzed within 48 hours or transferred to soil sample vials in the laboratory within 48 hours): December 1996, Revision O, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples.

_____. 2014. Region 4. *The Field Branches Quality System and Technical Procedures, Soil Sampling. SESDPROC-300-R3*. August.

Subsurface Soil Sampling	SOP 1-4 Revision: 8 Date: February 2015

Approved: Signature **Technical Review:** David Schroeder

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to define the techniques and requirements for collecting soil samples for environmental or geotechnical characterization purposes from the unconsolidated subsurface zone. General techniques discussed in this SOP include use of hand augers, split-barrel samplers, Shelby tubes, direct-push rig samplers, and backhoes, as well as field sampling and preservation methods.

2.0 Background

2.1 Definitions

Auger Flight - A steel section length attached to the auger length to extend the augers and remove additional unconsolidated material as drilling depth increases.

Backhoe - An excavator to which a shovel bucket is attached to a hinged boom and is drawn backward to move materials.

Direct Push Rig Sampler - A sampler with a locking tip that keeps the device closed during the sampling push. The tip is released at the desired depth, and the push is continued. During the push, the soil is pushed up into the sampler.

Grab Sample - A discrete portion or aliquot of material taken from a specific location at a given point in time.

Hand Auger - A stainless steel cylinder (bucket) approximately 7 to 10 centimeters (cm), or 3 to 4 inches (in) in diameter and 30 cm, or 1 foot (ft) in length, open at both ends with the bottom edge designed to advance perpendicular to the ground surface with a twisting motion into unconsolidated subsurface material to obtain a soil sample. The auger has a T-shaped handle (used for manual operation) attached to the top of the bucket by extendable stainless steel rods.

Liner - A cylindrical sleeve generally made of brass, stainless steel, or Teflon® that is placed inside a split-barrel sampler, direct-push rig sampler, or hand auger bucket to collect samples for Volatile Organic Compound (VOC) or other analyses or to prevent sample contamination.

Shelby Tube - A cylindrical sampling device which is generally made of steel, and which is driven into the subsurface soil through a hollow-stem auger using a drill rig. The tube, once retrieved, is capped on both ends. The undisturbed soil sample is extruded in the laboratory before soil analysis.

Slide Hammer - A device consisting of a drive weight (hammer) and a drive weight fall guide.

Split-Barrel Sampler - A cylindrical sampling device generally made of carbon steel that fits into a hollow-stem auger. The sampler is opened lengthwise, which allows the sample to be retrieved by "splitting" the barrel sampler. Also referred to as a split-spoon.

Subsurface Soil - The unconsolidated, or non-lithified, material that exists deeper than approximately 30 cm (1 foot) below the ground surface (bgs).

Unconsolidated Zone - A layer of non-lithified earth material (soil or sediment) that has no mineral cement or matrix binding its grains.

2.2 Associated Procedures

- SOP 1-2, *Sample Custody*
- SOP 1-3, Surface Soil Sampling

- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 3-5, *Lithologic Logging*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.3 Discussion

Shallow subsurface soil samples, or those taken from depths between 0.15 cm to 3 meters (m), or between 6 in and 10 ft bgs, may be collected using hand augers. However, soil samples collected with a hand auger are commonly of poorer quality than those samples collected by split-barrel or Shelby tube samplers because the soil sample is disturbed during the augering process. Split-barrel and Shelby tube samplers are generally used during collection of soil samples using hollow-stem auger drilling methods. Barrel-type samplers may also be used to collect soil samples from hand auger borings using a slide hammer device. For environmental sampling programs, liners are used to minimize the loss of volatile organic compounds (VOCs) and to prevent sample cross-contamination. Collecting samples using a backhoe enables the collector to correlate the precise vertical and horizontal interval of the sample collected relative to adjacent, visible subsurface materials.

The size and material of sampling devices used shall be selected based on project and analytical objectives and as defined in the site-specific sampling and/or work plans. Note that operation and collection of samples via drill rig (split spoon or Shelby tubes), direct-push methods, or backhoe is typically performed by subconsultants to CDM Smith, with field oversight provided by a CDM Smith field representative (engineer, geologist, scientist, or similar) as further discussed in Section 5.2 of this SOP.

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that field personnel are trained in the use of this procedure and the required equipment, and for ensuring that subsurface soil samples are collected in accordance with this procedure and any other SOPs pertaining to specific media sampling. The site manager must also ensure that the quantity and location of subsurface soil samples collected meet the requirements of the site-specific sampling and/or work plans.

Field Team Leader - The field team leader is responsible for ensuring that field personnel collect subsurface soil samples in accordance with this SOP and other relevant procedures.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

4.0 Required Equipment

4.1 General

- Site-specific plans (e.g., sampling, work, health and safety)
- Field logbook
- Indelible black ink pens and markers
- Clear, waterproof tape
- Appropriate sample containers
- Labels and appropriate forms/documentation for sample shipment
- Insulated cooler(s) and waterproof sealing tape
- Bags of ice or “blue ice” packs
- Nitrile or appropriate gloves
- Plastic zip-top bags
- Personal protective clothing and equipment
- Plastic sheeting
- Stainless steel and/or Teflon-lined spatulas and pans, trays, bowls, trowels, or spoons
- Decontamination supplies
- Sample chain-of-custody forms
- Custody seals
- Kimwipes or paper towels

Additional equipment is discussed in Section 5.2.7, Field Sampling/Preservation Methods.

4.2 Manual (Hand) Augering

- T-handle
- Hand auger: flighted-, bucket-, or tube-type auger as required by the site-specific plans
- Extension rods
- Wrench(es), pliers
- Slide hammer with extension rods

4.3 Split-Barrel and Shelby Tube Sampling

- Drill rig equipped with a 63-kilogram (kg) (140-lb) drop hammer and sufficient hollow-stem auger flights to drill to the depths required by the site-specific work/sampling plans.
- Sufficient numbers of split-barrel samplers so that at least one sampler is always decontaminated and available for sampling. Three split-barrel samplers are generally the minimum necessary (Shelby tubes are used only once).
- Split-barrel liners (as appropriate).
- Wrench(es), hammer.

4.4 Direct Push Rig Sampling

- Direct push rig with sufficient probe rods to extend to sample depths required by the site-specific work/sampling plans
- Sufficient number of samplers (in case of malfunction) and appropriate liners to collect adequate number of samples
- Extension rods
- Wrench(es), pliers, other specific tools

4.5 Backhoe Sampling

- Backhoe with a sufficient length boom to extend to planned depths
- Sufficient number of trowels or scoops
- Extension rods
- Tape, utility knife, other specific tools as needed

5.0 Procedures

5.1 Preparation

1. Review site-specific health and safety plan and project plans before initiating sampling activity.

2. Don the appropriate personal protective clothing as indicated in the site-specific health and safety plan.

3. Locate sampling location(s) in accordance with project documents (e.g., work plan) and document pertinent information in the appropriate field logbook. When possible, reference locations back to existing site features such as buildings, roads, intersections, etc.

4. Processes for verifying depth of sampling must be specified in the site-specific plans.

5. Clear away vegetation and debris from the ground surface at the boring location.

6. If decontamination of equipment and/or personnel is required, set up a decontamination zone in accordance with SOP 4-5.

7. Prepare an area near the sampling location to perform sample collection activities by placing plastic sheeting on the ground, or, if required by the site-specific health and safety plan and or work/sampling plans, place plastic sheeting over the area immediately surrounding the borehole, as applicable.. Sample collection should be performed at a safe distance from all heavy equipment, or as determined by heavy equipment operator(s) and/or the CDM Smith field representative.

5.2 Sample Collection

The following general steps must be followed when collecting all subsurface soil samples. Refer to section 5.3 of this SOP and SOP 1-3 (Surface Soil Sampling) for additional guidance on field sampling and preservation methods.

1. Wear clean gloves during handling of all sample containers and sampling devices.

2. VOC samples or samples that may be degraded by aeration shall be collected first and with the least disturbance possible. When sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration, it is necessary to minimize sample disturbance and consequently minimize analyte loss. The representativeness of a VOC grab sample is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed. Sample containers containing samples for VOC analysis shall be filled completely to minimize headspace (see section 5.3 of this SOP).

3. All sampling information, including environmental and/or geotechnical soil characterization, sample depth, sample volume, and requisite geotechnical or environmental analyses shall be recorded in the field logbook and on any associated forms as specified in the site-specific sampling/work plans. Sample lithology shall be described according to SOP 3-5.

4. Specific sampling devices to be used shall be identified in the site-specific work/sampling plans and shall be recorded in the field logbook.

5. Care must be taken to prevent cross-contamination and misidentification of samples as described in subsequent subsections of this SOP.

5.2.1 Manual (Hand) Augering

The following steps must be followed when collecting environmental soil samples using hand-auger techniques:

1. Advance the auger to the depth specified in the site-specific sampling plan for sample collection Place cuttings on plastic sheeting or as specified in the site-specific work/sampling plans. If possible, lay out the cuttings in stratigraphic order, or from the shallowest cuttings collected to the deepest cuttings collected.

2. During auger advancement and sample collection, record , observations made of the geologic features of the soil or sediments per American Society for Testing and Materials (ASTM) D 2448 (Standard Practice for Description and Identification of Soils (Visual-Manual Procedure) in the field logbook.

3. Stop advancing the auger when the top of the specified sampling depth has been reached. If required by the site-specific sampling plan, remove the auger from the hole and decontaminate the auger or use a separate decontaminated auger, then obtain the sample.

4. Collect a grab sample for VOC analyses (or samples that may be degraded by aeration) immediately and place in sample container. Sample container(s) shall be filled completely to minimize headspace.

5. Remaining sample material for other analyses shall be homogenized before placing samples in the appropriate containers.

6. Wipe container(s)with a clean Kimwipe or paper towel to remove residual soil from the exterior of the container(s).

7. Label the sample container with the appropriate information. Secure the label by covering it with a piece of clear tape.

8. Place the containers in zip-top plastic bags and seal the bags. Pack samples in a cooler with ice or cold packs (to maintain a temperature of 4°C)

9. Proceed with additional sampling as required by the site-specific plans.

10. When sample collection is complete, dispose of cuttings, plastic sheeting, etc., as specified in the site-specific plans.

11. Complete the field logbook entry and other appropriate forms, being sure to record all relevant information before leaving the site.
12. Properly package all samples for shipment and complete all necessary sample shipment documentation. Remand custody of samples to the appropriate personnel. Refer to SOPs 1-2 and 2-1 and site-specific plans.

5.2.2 Manual (Hand) Augering Using a Tube Sampler with Liner or Slide Hammer

The following steps must be followed when collecting environmental soil samples using a hand-auger and a tube sampler with liner or slide hammer:

1. Auger to the depth required for sampling. Place cuttings on the plastic sheeting as specified in the site-specific plans. If possible, lay out the cuttings in stratigraphic order.

2. During auger advancement and sample collection, record observations made of the geologic features of the soil or sediments per ASTM D 2448 (Standard Practice for Description and Identification of Soils (Visual-Manual Procedure) in the field logbook.

3. Stop advancing the auger when the top of the specified sampling depth has been reached. If required by the site-specific sampling plan, remove the auger from the hole and decontaminate the auger per the site-specific work/sampling plan (see line item 11 below).

4. Prepare a clean, new tube sampler by installing a decontaminated liner in the auger tube.

5. Obtain the sample by driving the sample tube through the sample interval with the slide hammer. Remove the liner from the tube and immediately cover the ends with Teflon tape and cap the ends of the tube. Seal the caps with waterproof tape.

6. Wipe sealed liners with a clean Kimwipe or paper towel.

7. Label the sealed liners as required in the site-specific plans. Mark the top and bottom of the sample on the outside of the liner.

8. Place sealed liners in zip-top plastic bags and seal the bags. Pack samples in a cooler with ice or cold packs (to maintain a temperature of 4°C).

9. Proceed with additional sample collection as required by the site-specific sampling plans.

10. When sampling is complete, dispose of cuttings, plastic sheeting, etc., as specified in the site-specific work/sampling plans.

11. Decontaminate all equipment according to SOP 4-5 between each sample.

12. Complete the field logbook entry and other forms, being sure to record all relevant information before leaving the site.

13. Properly package all samples for shipment and complete all necessary sample shipment documentation. Remand custody of samples to the appropriate personnel. See SOPs 1-2 and 2-1 or site-specific plans.

5.2.3 Split-Barrel Sampling

Note: Steps 1 through 12 describe the general activities to be performed by a licensed drilling contractor, not by CDM Smith personnel.

Subsurface Soil Sampling

SOP 1-4
Revision: 8
Date: February 2015

The following steps must be followed when collecting split-barrel samples for environmental and/or geotechnical purposes:

1. Remove any pavement and subbase material from an area of twice the bit diameter, if necessary.
2. The drilling rig will be decontaminated at a separate location before drilling, per SOP 4-5 or the site-specific decontamination procedures.
3. Attach the hollow-stem auger with the cutting head, plug, and center rod(s) to the drill rig.
4. Begin drilling and proceed to the first designated sample depth, adding auger flights as necessary.
5. Upon reaching the designated sample depth, slightly raise the auger(s) to disengage the cutting head, and rotate the auger without advancement to clean cuttings from the bottom of the hole.
6. Remove the plug and center rods, if applicable.
7. If required by the site-specific sampling plan, install decontaminated liners in the split barrel sampler.
8. Install a decontaminated split-barrel on the center rod(s) and insert it into the hollow-stem auger. Connect the hammer assembly and lightly tap the rods to seat the drive shoe at the top of undisturbed soil or sediment.
9. Mark the center rod in 15-cm (6-inch) increments from the top of the auger(s).
10. Drive the split-barrel using the hammer. Use a full 76-cm (30-inch) drop as specified by ASTM D 1586. Record the number of blows required to drive the sampler through each 15-cm (6-inch) increment.
11. Stop driving the split-barrel sampler when the full length of the spoon (24 inches) has been driven or if refusal is encountered. Refusal occurs when little or no progress is made for 50 blows of the hammer. ASTM D1586-11 § 7.2.1 and 7.2.2 defines “refusal” as >50 blows per 6-inches advanced or a total of 100 blows.
12. Pull the sampler free by using upswings of the hammer to loosen the sampler. Pull out the center rod and sampler.
13. Unscrew the sampler assembly from the center rod and place the sampler on the plastic sheeting.
14. Remove the drive shoe and head assembly. If necessary, tap the sampler assembly with a hammer to loosen threaded couplings.
15. With the drive shoe and head assembly off, open (split) the sampler, being careful not to disturb the sample.
16. Label sample containers with appropriate information. Secure the label, covering it with a piece of clear tape. If liners were used, immediately install Teflon tape over the ends of the liners, cap the liners, and seal the caps over the ends of the liner with waterproof tape. Label the samples as required by the site-specific plans. Mark the top and bottom of each sample on the outside of each liner. Indicate boring/well number and depth on the outside of the liner, as required.
17. If samples are to be collected from the soil sample for VOC analyses and liners were not used, place sample material in the appropriate sample container immediately after opening the split-barrel, filling the sample bottle as completely as possible to minimize headspace. Seal the container immediately, then describe the sample material in the field logbook and/or associated forms per ASTM D 2488.
18. Remaining sample material shall be homogenized before placing samples in appropriate containers.
19. Record the sample identification number, depth from which the sample was taken, sample recovery and the analyses to be performed on the samples in the field logbook and on the appropriate forms.

20. Wipe containers with a clean Kimwipe or paper towel. Label sample containers as required when liners are not used.

21. Place containers and/or sealed liners in zip-top plastic bags and seal the bags. Pack samples in a cooler with ice or cold packs (to maintain a temperature of 4°C)

22. In the field logbook and on the boring log, describe sample lithology by observing cuttings and/or the bottom end of the liner.
23. Continue to advance the borehole to the next sampling point. Collect samples as outlined above.

24. When sampling is complete, remove the drilling rig to the heavy equipment decontamination area.

25. Dispose of cuttings, plastic sheeting, etc., as specified in the site-specific plans. Backfill borehole as specified in project- and /or site-specific work/sampling plans.

26. Decontaminate samplers and other small sampling equipment according to SOP 4-5 before proceeding to other sampling locations.

27. Complete the field logbook entry and other forms, being sure to record all relevant information before leaving the site.

28. Properly package all samples for shipment to laboratories and complete all necessary sample shipment documentation. Remand custody of the samples to appropriate personnel. See SOPs 1-2 and 2-1 or site-specific plans.

5.2.4 Shelby Tube Sampling

Note: Steps 1 through 11 describe activities to be performed by a licensed drilling contractor, not by CDM Smith personnel. ASTM D1586-11 provides additional details pertaining to this sampling methodology.

The following steps must be followed when collecting geotechnical samples using Shelby tubes:

1. Remove any pavement and subbase material from an area of twice the bit diameter, if necessary.

2. The drilling rig will be decontaminated at a separate location before drilling.

3. Attach the hollow-stem auger with the cutting head, plug, and center rod(s).

4. Begin drilling and proceed to the first designated sample depth, adding auger(s) as necessary.

5. Upon reaching the designated sample depth, slightly raise the auger(s) to disengage the cutting head, and rotate the auger without advancement to clean cuttings from the bottom of the hole.

6. Remove the plug and center rods, if applicable.

7. Attach a head assembly to a decontaminated Shelby tube sampler assembly. Attach the Shelby tube assembly to the center rods.

8. Lower the Shelby tube and center rods into the hollow-stem augers and seat it at the bottom. Be sure to leave 30 inches or more of center rod above the lowest point to the hydraulic piston's extension.

9. Use the rig's hydraulic drive to push the Shelby tube into undisturbed soil. The tube shall be pushed with a slow, steady force. The pressure used by the driller to push the Shelby tube shall be noted in the field logbook.

10. When the Shelby tube has been advanced to its full length or to refusal, back off the hydraulic pistons. Attach a hoisting plug to the upper end of the center rod, slightly twist to break off the sample, and pull the apparatus out of the hole with the rig winch.

11. Retrieve the Shelby tube to ground surface, detach it from the center rod, and remove the head assembly.

12. Since the typical intent of Shelby tube sampling is for engineering purposes and an undisturbed sample is required, the tube ends shall be sealed immediately. Sealing is accomplished by filling any void space in the tube with melted beeswax, then placing caps on the ends of the tube and taping caps into place. The top and bottom ends of the tube shall be marked and the tube transported to the laboratory in an upright position. ***It is extremely important that the Shelby tube samples are not disturbed in any way (dropped, rolled, subjected to extreme temperatures, etc.)***.

13. Wipe sealed tubes with a clean Kimwipe or paper towel.

14. Indicate boring/well number and depth on outside of the tube.

15. Place sealed tubes in zip-top plastic bags, seal bags, and pack samples in a chilled cooler, if applicable.

16. Continue to advance the borehole to the next sampling point. Collect additional samples per the site-specific sampling plan as outlined above.

17. When sampling is complete, remove the drilling rig to the heavy equipment decontamination area.

18. Dispose of cuttings, plastic sheeting, etc., as specified in the site-specific work/sampling plans. Backfill borehole as specified in project- and /or site-specific work/sampling plans.

19. Complete the field logbook entry, being sure to record all relevant information before leaving the site.
20. Properly package all samples for shipment to laboratories and complete all necessary sample shipment documentation. Remand custody of the samples to appropriate personnel. See SOPs 1-2 and 2-1 or site-specific plans.

5.2.5 Direct Push Rig Sampling

Note: Steps 1 through 11 describe activities to be performed by a licensed drilling contractor, not CDM Smith personnel.

The following steps must be followed when collecting environmental samples using a direct push rig sampler:

1. Verify that the direct-push rig has been decontaminated at a separate location before drilling.

2. Attach the properly assembled sampler with appropriate liner to the end of the probe rod.

3. Attach drive cap and probe to the first designated sample depth, adding rod(s) as necessary.

4. Upon reaching the designated sample depth, remove the drive cap to access the inside of the probe rods.

5. Insert extension rods into probe rod; turn extension rod to release tip.

6. Retrieve extension rods, replace drive cap, add additional push rod if required, and push probe rod to the planned sample interval.

7. Attach pull cap and retrieve push rods and sampler.

8. Remove the sampler from the probe rod, and then remove the cutting shoe from the sampler.

9. Once the cutting shoe is removed, the liner containing the sample material can be removed from the sampler. Analytical samples can now be collected by CDM Smith personnel per site-specific plans and per Section 5.2.2 of this SOP.

10. When sample collection is complete, remove the push rig to the heavy equipment decontamination area.

11. Dispose of excess sample cuttings, plastic sheeting, etc., as specified in the site-specific plans.

12. Complete the field logbook entry, being sure to record all relevant information before leaving the site.
13. Properly package all samples for shipment to laboratories and complete all necessary sample shipment documentation. Remand custody of the samples to appropriate personnel. See SOPs 1-2 and 2-1 or site-specific plans.

5.2.6 Backhoe Sampling

Note: Steps 1, 2, 7, and 8 describe activities to be performed by a licensed heavy equipment operator, not CDM Smith personnel.

The following steps must be followed when collecting environmental samples using a backhoe:

1. Verify that the parts of the backhoe that will come in contact with the soil to be sampled have been decontaminated before excavation begins.

2. Excavate to the depth required in the site-specific plans.

3. Use a stainless steel trowel or scoop to obtain the sample material

4. Attach the trowel to a steel rod, wooden handle, or other similar device.

5. Remove the surface layer of soil "smeared" on the trench wall.

6. Replace the trowel with a clean trowel to collect a representative sample.

7. Analytical samples shall be collected by CDM Smith personnel per site-specific plans and per Section 5.2.2 of this SOP.

8. When sample collection is complete in the trench, backfill the trench with the excavated material, as appropriate.

9. Once the trench has been backfilled, move the backhoe to the heavy equipment decontamination area.

10. Dispose of excess sample cuttings, plastic sheeting, etc., as specified in the site-specific plans.

11. Complete the field logbook entry, being sure to record all relevant information before leaving the site.
12. Properly package all samples for shipment to laboratories and complete all necessary sample shipment documentation. Remand custody of the samples to appropriate personnel. See SOPs 1-2 and 2-1 or site-specific plans.

5.3 Field Sampling/Preservation Methods

The following three sections contain SW 846 Methods for sampling and field preservation. These methods include EnCore™ Sampler Method for low-level detection limits, EnCore Sampler Method for high-level limits/screening, and methanol preservation. Use of these methods may be required by the governing EPA Region, the client, or if required by the site-specific sampling plan. These methods are very detailed and contain equipment requirements at the beginning of each section.

When collecting soil samples using the EnCore Sampler Method, collection of soil for moisture content analysis is required. Results of this analysis are used to adjust "wet" concentration results to "dry" concentrations to meet analytical method requirements.

Note: Some variations from these methods, (e.g., sample volume) may be required depending on the contracted analytical laboratory.

5.3.1.1 EnCore Sampler Equipment and Collection Requirements for Low-Level Analyses (<200 µg/kg)

The following equipment is required for low-level analysis:

- Three 5 grams (g) samplers

Note: The sample volume requirements specified are general requirements. Actual sample volume and/or container sizes may vary depending on client or laboratory requirements.

- One 110-milliliter (mL) (4-ounce [oz.]) wide-mouth glass jar or applicable container for moisture analysis
- One T-handle
- Paper towels

The requirements for collecting low level analysis by the EnCore Sampler Method are as follows:

1. Wear clean gloves during handling of all sample containers and sampling devices.
2. Remove sampler and cap from package and attach T-handle to sampler body.
3. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full. The O-ring will be visible within the hole on the side of the T-handle. If the O-ring is not visible within this window, then the sampler is not full.
4. Extract sampler and wipe the sampler head with a paper towel so that the cap can be tightly attached.
5. Push cap on with a twisting motion to secure to the sampler body.
6. Rotate the sampler stem counterclockwise until stem locks in place to retain sample within the sampler body.
7. Fill out sample label and attach to sampler.
8. Repeat procedure for the remaining two samplers.
9. Collect moisture sample in 110-mL (4-oz.) wide-mouth jar using a clean stainless steel spoon or trowel.
10. Store samplers at 4°C, ±2°C. Samples must be shipped and delivered to the analytical laboratory for extraction within 48 hours.

Note: Verify requirements for extraction/holding times.

5.3.1.2 EnCore Sampler Equipment and Collection Requirements for High-Level Analyses (≥200 µg/kg)

The following equipment is required for high-level analysis:

- One 5-g sampler or one 25-g sampler (the sampler size used will be dependent on client and laboratory requirements
- One 110-mL (4-oz.) wide-mouth glass jar or applicable container specified for moisture analysis
- One T-handle
- Paper towels

The requirements for collecting high-level analysis by the EnCore Sampler Method are as follows:

1. Wear clean gloves during handling of all sample containers and sampling devices.
2. Remove sample and cap from package and attach T-handle to sampler body.
3. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full. The O-ring will be visible within the hole on the side of the T-handle. If the O-ring is not visible within this window, then the sampler is not full.
4. Use clean paper toweling to quickly wipe the sampler head so that the cap can be tightly attached.
5. Push cap on with a twisting motion to attach cap.

6. Fill out a sample label and attach to sampler.

7. Rotate sampler stem counterclockwise until the stem locks in place to retain the sample within the sampler body.

8. Collect moisture sample in 110-mL (4-oz.) wide-mouth jar or designated container using a clean stainless steel spoon or trowel.

9. Store samplers at 4°C, ±2°C. Samples must be shipped and delivered to the analytical laboratory for extraction within 48 hours.

Note: Verify requirements for extraction/holding times.

5.3.1.3 Methanol Preservation Equipment and Sampling Requirements for High-Level Analyses (≥200 µg/kg)

The following equipment is required for methanol preservation sampling:

- One preweighed jar that contains methanol or a preweighed empty jar accompanied with a preweighed vial that contains methanol (laboratory grade)
- One dry weight cup
- Weighing balance that accurately weighs to 0.01 g (with accuracy of ±0.1 g)
- Set of balance weights used in daily balance calibration
- Latex gloves
- Paper towels
- Cutoff plastic syringe or other coring device to deliver 5 g or 25 g of soil

The requirements for sampling and preservation are as follows:

1. Wear clean gloves during all handling of preweighed vials.

2. Weigh the vial containing methanol preservative to the nearest 0.01 g. If the weight of the vial with methanol varies by more than 0.01 g from the original weight recorded on the vial, discard the vial. If the weight is within tolerance, it can be used for soil preservation/collection below. (Commercial sources are available which supply pre-preserved and tared vials which eliminates the need to transport and handle larger quantities of methanol and eliminates the need for a precision scale vials with preservative).

3. Quickly collect a 5-g or 25-g sample using a cutoff plastic syringe or other coring device designed to deliver 5 g or 25 g of soil from a freshly exposed surface of soil. The 5-g or 25-g size used is dependent on client and laboratory requirements.

4. Carefully wipe the exterior of the collection device with a clean paper towel.

5. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol, be careful not to splash the methanol outside of the vial. Again, the type of jar used is dependent on the client or laboratory requirements.

6. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided, using only one vial of methanol preservative per sample jar.

7. Using the paper toweling, remove any soil off of the vial threads and cap the jar.

8. Weigh the jar with the soil in it to the nearest 0.01 g and record the weight on the sample label.

9. Collect dry weight sample using a clean stainless steel spoon or trowel.

10. Store samples at 4°, ±2°C.

11. Ship sample containers with plenty of ice in accordance with DOT regulations (CORROSIVE. FLAMMABLE LIQUID. POISON) to the laboratory.

6.0 Restrictions/Limitations

- Basket or spring retainers may be needed for split-barrel sampling in loose, sandy soils.
- A larger-diameter split spoon sampler assembly in addition to the standard split spoon assembly is recommended for all projects on which environmental sample collection from discrete intervals may be required. This enables additional sample material to be recovered in the event the initial split spoon sample does not yield adequate sample material. This method is not recommended for the collection of samples that are to be analyzed for VOCs.
- Shelby tubes are most appropriately used to sample cohesive materials, and may not retain sample material in loose, sandy soils.
- When grab sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration, it is necessary to minimize sample disturbance and consequently minimize analyte loss. The representativeness of a VOC grab sample is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed.

7.0 References

American Society for Testing and Materials. 2011. *Standard Test Method for Penetration Test (SPT) and Split Barrel Sampling of Soils*. Standard Method D1586-11.

_____. 2000. *Standard Test Method for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes*. Standard Method D1587-08(2012)e1.

U. S. Environmental Protection Agency. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846)*, Third Edition, November 1986, (as amended by Updates I, II, IIA, IIB, III, and IIIA, IIIB, and IVA, IVB). Method 5035 (***Note***: § 6.2.1.8 of this method says samples stored in EnCore™ samplers shall be analyzed within 48 hours or transferred to soil sample vials in the laboratory within 48 hours): December 1996, Revision O, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples.

_____. 2014. Region 4. *The Field Branches Quality System and Technical Procedures, Soil Sampling. SESDPROC-300-R3*. August.

Groundwater Sampling Using Bailers	SOP 1-5 Revision: 9 Date: February 2015
Approved: *Signature*	**Technical Review:** David Schroeder

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define requirements for the collection of groundwater samples with bailers.

2.0 Background

Collection of groundwater samples from monitoring wells on or near a hazardous waste site may be required to characterize the nature and extent of contamination.

Methods used for the collection of groundwater samples include bailing and a variety of pumping techniques. Bailers are hollow cylinders with unidirectional (open up) check valves at the bottom end. Some bailers may also be closed or valved at the upper end. Bailers used in environmental applications are typically constructed of polyvinyl chloride (PVC), polyethylene, stainless steel, or Teflon®. Disposable polyethylene, PVC, and Teflon bailers are commonly used and eliminate the possibility of cross contamination as a result of poor decontamination. The bailer line typically consists of disposable nylon cord, disposable polypropylene cord, or Teflon-coated stainless steel wire. The bailer is slowly lowered into the well using an acceptable type of line until submerged. The bailer is then retrieved to the surface for sample collection. For the best results, the sequence of sampling is from least to most contaminated wells. It is preferable to have bailers dedicated to each monitoring well.

2.1 Associated Procedures

- SOP 1-2, *Sample Custody*
- SOP 1-6, *Water Level Measurement*
- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-3, *Well Development and Purging*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that field personnel are trained in the use of this procedure and for verifying that groundwater samples are collected in accordance with this procedure.

Field Team Leader - The field team leader is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any associated SOPs.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

- Site-specific plans
- Field logbook
- Indelible black ink pens and markers
- Labels and appropriate forms/documentation for sample shipment
- Sample chain-of-custody forms
- Insulated cooler and waterproof sealing tape (strapping tape)
- Plastic zip-top bags

- Ice double-bagged in plastic zip-top bags
- Bailer of the appropriate design and construction for the sampling application
- Clean bailer line of sufficient length for well condition
- Water level meter and/or other water level measuring device
- Clean beaker(s) or other container for measurement of water quality parameters
- Plastic sheeting (4-mil thickness)
- Latex or appropriate gloves
- Filtering apparatus (e.g., peristaltic pump), if required
- Appropriate sample containers with labels and preservatives, as required
- Temperature, conductivity, pH, dissolved oxygen, turbidity, and other meters as required by the site-specific field sampling plan
- Photoionization detector (PID) or equivalent and other instruments as required by the site-specific health and safety plan
- Decontamination supplies, as required by SOP 4-5
- Personal protective clothing and equipment as required by the site-specific health and safety plan

5.0 Procedures

1. Review site-specific health and safety plan and project plans before initiating sampling activity.

2. Don personal protective clothing and equipment as specified in the site-specific health and safety plan. All field equipment will be calibrated, tested, or checked for proper functioning before use per the manufacturer's instructions.

3. Prepare the site for sample acquisition. If required, cover the ground surface around the wellhead with plastic sheeting. Arrange the required decontaminated sampling and monitoring equipment for convenient use. If onsite decontamination is required, arrange the necessary supplies in a nearby but separate location, away from the wellhead (i.e., in an exclusion zone).

4. Open the well and note the condition of the casing and cap. Immediately check for organic vapors using a PID or flame ionization detector as appropriate. Refer to the site health and safety plan for the required monitoring and frequencies.

5. Determine the static water level and depth to well bottom in accordance with SOP 1-6. Record this information in the field logbook and/or on the appropriate form.

6. Purge the well according to SOP 4-3. Allow the water level in the well to recover to 75 percent of its static level so that a representative sample of the screened portion of the aquifer can be obtained. The bailer shall be completely submerged so that it does not contact the bottom of the well which may introduce sediment into the groundwater sample. Samples shall be collected within 3 hours of purging if recharge is sufficient. Wells with a low recharge rate must be collected within 24 hours of purging.

7. Securely attach the bailer to the line. The opposite end of the line shall be secured to prevent loss of the bailer into the well.

8. Arrange the sample containers in the order of use. Samples to be analyzed for volatile organic compounds (VOCs), if required, shall be obtained first, followed in order by other organic samples, then inorganic samples and other parameters. For example:

   a) VOCs
   b) Purgeable organic carbon (POC)
   c) Purgeable organic halogens (POX)
   d) Total organic halogens (TOX)
   e) Total organic carbon (TOC)
   f) Extractable organics*
   g) Total metals
   h) Dissolved metals
   i) Cyanide
   j) Sulfate and chloride
   k) Nitrate and ammonia
   l) Radionuclides

   *Extractable organics include semivolatile organic compounds, pesticides, and PCBs.

9. Don clean sampling gloves; lower the decontaminated or disposable bailer into the well. The bailer shall enter the water slowly to prevent aeration, particularly when VOC samples are being collected. Do not allow the bailer to come in contact with the well bottom.

10. Retrieve the filled bailer to the surface. To prevent contamination of the bailer line, do not allow line to contact the ground, rather keep line in a clean pail or other clean container. Hang the bailer from a bailer stand or other support, if available, or have an assistant hold it off the ground. Immediately obtain any required volatile samples (VOC, POC, POX, TOX, or TOC) by gently transferring water from the bailer to the sample bottle through a VOC sampling device inserted into the bottom of the bailer. Care shall be taken to adjust the flow of the water into the vial so that it is not too fast. The vial shall also be tilted so that the stream of water is directed down the side of the vial to reduce nonlaminar flow into the vial and to prevent aeration. Check the filled VOC vials for bubbles. If bubbles are present in a vial, discard it and fill another vial from the bailer. After collecting volatile samples, lower the bailer to collect additional water for the remaining parameters. If sample filtration is required for metals, it shall be performed immediately following sample retrieval, and before sample preservation. Organic samples generally do not require filtration; VOC samples are never filtered. Preservation of samples shall be performed according to the applicable field plan. Check the pH on samples (other than VOCs) that require preservation. Collect additional quality assurance/quality control samples (i.e., duplicate samples) as required by the applicable field plan.

11. Wipe the outer surfaces of the sample containers clean with a Kim-wipe or clean paper towel. Additional sample bottle decontamination may be appropriate in some cases.

12. Properly label all containers according to SOP 2-1.

13. Place sample containers in individual zip-top plastic bags and seal the bags (if required by site-specific plans).

14. Immediately pack all sample containers that require a 4ºC preservation on ice in coolers (refer to SOP 2-1 and site-specific plans).

15. Record the parameters to be analyzed and volumes collected, and time and date of collection in the field logbook. Prepare chain-of-custody forms according to site-specific plans and SOP 1-2.

16. Decontaminate sampling equipment as needed, according to SOP 4-5.

17. Close and lock the well cover. Clean up the area and place disposable materials (plastic sheeting, gloves, Tyvek®) in the designated waste receptacle.

18. If required by the site-specific field sampling plan, obtain required field measurements such as temperature, conductivity, pH, oxidation potential (Eh), turbidity, salinity, or dissolved oxygen measurements immediately after samples have been collected and record them in the field logbook.

6.0 Restrictions/Limitations

Careful sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration is necessary to minimize sample disturbance and, hence, analyte loss. The representativeness of this sample, however, is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed.

Use of nondisposable bailers may contribute to cross contamination if proper and thorough decontamination procedures are not followed.

7.0 References

Office of Solid Waste and Emergency Response. 1992. *RCRA Ground-water Monitoring: Draft Technical Guidance*, EPA/530/R-93/001. November.

Office of Solid Waste and Emergency Response. 1986. *RCRA Groundwater Monitoring Technical Guidance Enforcement Document*, OSWER-9950.1. September.

ASTM International. 2001. Standard Practice for Sampling Liquids Using Bailers. Standard Method D6699-01 (2010).

Groundwater Level Measurement	SOP 1-6 Revision: 9 Date: February 2015
Approved: Signature	**Technical Review:** David Schroeder

1.0 Objective

Groundwater level measurements are fundamental to groundwater and solute transport studies and are conducted during groundwater sampling events to calculate the amount of groundwater to be purged from the well. This technical standard operating procedure (SOP) defines the techniques and requirements for obtaining depth to groundwater (or groundwater level) measurements.

2.0 Background

2.1 Definitions

Water Level Indicator - A portable device for measuring the depth from a fixed point (which could be below, at, or above the ground surface) to groundwater inside a well, borehole, or other underground opening.

Measurement Point - An easily located and clearly defined mark at the top of a well from which all water level measurements from that particular well are made. The measurement point shall be as permanent as possible to provide consistency in measurements.

Electrical Tape - A graduated plastic tape onto which a water-sensitive electrode is connected that will electronically signal the presence of water (as a result of circuit closure).

Immiscible Fluids - Two or more fluid substances that will not mix and, therefore, will exist together in a layered form. The fluid with the highest density will exist as the bottom layer, the fluid with the lowest density will exist as the top layer, and any other fluid layers will be distributed relative to their respective densities.

Discharge - The removal/release of water from the zone of saturation.

Recharge - The addition of water to the zone of saturation.

Static Water Level - The level of water in a well, borehole, or other underground opening that is not influenced by discharge or recharge.

Well Casing - A steel, stainless steel, or polyvinyl chloride pipe that extends into a borehole and is connected to the well screen or sealed at the bedrock surface in open-hole wells. The upper portion (approximately 3 to 4 feet) of the well casing is normally enclosed by an outer steel protective casing.

Protective Casing - A steel cylinder or square protective sleeve extending approximately 3 to 5 feet into the ground, surrounding the well casing. For flush-mounted wells, the protective casing will extend only high enough so that the well and protective casing can be enclosed by a Christy box or equivalent vault. In above-grade wells, the protective casing will extend above the ground surface approximately 2 to 3 feet. The protective casing protects the well casing.

2.2 Associated Procedures

- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.3 Discussion

The most common uses of static water level data are to: determine the elevation of groundwater, the direction of groundwater flow, identify areas of recharge and discharge, evaluate the effects of manmade and natural stresses on the groundwater system, define the hydraulic characteristics of aquifers, and evaluate stream-aquifer relationships. Specific uses for water level data may include:

- Determining the change in water level due to distribution and rate of regional groundwater withdrawal
- Showing the relationship of groundwater to surface water
- Estimating the amount, source, and area of recharge and discharge
- Determining rate and direction of groundwater movement

Static water level measurements shall be obtained from each well before purging, sampling, or other disturbance of the water table.

3.0 General Responsibilities

Project Manager - The project manager is responsible for ensuring that measurements are conducted in accordance with this procedure and any other SOP pertaining to site activities related to obtaining groundwater level measurements.

Field Team Leader - The field team leader is responsible for ensuring that field personnel obtain water level measurements in accordance with this and other relevant procedures.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field sampling plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

4.1 General

- Site-specific plans
- Field logbook
- Indelible black ink pens
- Permanent felt-tip marker (e.g., Sharpie)
- Personal protective equipment
- Decontamination equipment and supplies, including rinse bottles and deionized water
- Tap water and large beaker or bucket
- Water level meter

4.2 Measuring Devices

The equipment required to obtain water level measurements is dependent on the type of procedure chosen. Measurements may be made with a number of different devices and procedures. Measurements are taken relevant to a permanent measurement point on the well riser.

Electrical tapes are preferred over other devices such as steel tape because of the electrical tape's simplicity and ability to make measurements in a short period of time. Many types of electrical instruments have been devised for measuring water levels; most operate on the principle that a circuit is completed when two electrodes are immersed in water. Examples of electrical tapes that are frequently used include the Slope Indicator Co.® and Solinst® electronic water level indicators. These instruments are powered by batteries that shall be checked before mobilization to the field.

Electrical tapes are coiled on a hand-cranked reel unit that contains the batteries and a signaling device that indicates when the circuit is closed (i.e., when the probe reaches the water). Electrodes are generally contained in a weighted probe that keeps the tape taut in addition to providing some shielding of the electrodes against false indications as the probe is being lowered into the hole. The electrical tapes are marked with 0.01-foot increments. Caution shall be exercised when using electrical tapes when the water contains elevated amounts of dissolved solids. Under these conditions, the signaling device will remain activated after the probe is removed from the water. When the water being measured contains very low amounts of dissolved solids, it is possible for the probe to extend several inches below the water level before activating the signaling device. Both of these conditions are related to the conductivity of the water and in some cases may be compensated for by the sensitivity control, if the device has this option. In groundwater with high conductivity the sensitivity control may need to be turned down, and in groundwater with low conductivity the sensitivity control may need to be turned up to obtain a proper depth to groundwater measurement.

5.0 Procedures

5.1 Preparation

The following steps must be taken when preparing to obtain a water level measurement:

1. Assign a designated field logbook to record all field events and measurements according to SOP 4-1. Document any and all deviations from SOPs and site-specific plans in the field logbook and include rationale for the changes.

2. Always exercise caution to prevent inappropriate or contaminated materials from entering an environmental well.

3. Standing upwind from the well, open the groundwater well. Monitor the well with a photoionization detector, flame ionization detector, or equivalent vapor analyzer as soon as the cap is opened, as dictated by the site-specific health and safety plan.

For comparability, water level measurements shall always be referenced to the same vertical (elevation) datum marker, such as a U. S. Geological Survey (USGS) vertical and horizontal control point monument. The elevations calculated from the measurement of static water levels shall be referenced to mean sea level unless otherwise specified in the site-specific plans.

The measurement point must be as permanent as possible, clearly defined, marked, and easily located. Frequently, the top of the PVC riser is designated as the measurement point. However, since the top of the riser is seldom smooth and horizontal, one particular point on the riser pipe shall be designated and clearly marked. This can be accomplished by marking a point on the top of the riser pipe with a permanent marker. To avoid spilling liquids into the well, paints or other liquid marking materials shall not be used.

5.2 Water Level Measurement Using Electrical Water Level Indicators

The following steps must be followed when taking water level measurements using electrical tapes:

1. Before lowering the probe into the well, the circuitry shall be checked by dipping the probe in tap water and checking to ensure that the signaling device responds to probe submergence. The probe shall then be lowered slowly into the well until contact with the water surface is indicated. The electrical tape reading is made at the measuring point. Take a second and third check reading to verify the measurement before completely withdrawing the tape from the well. All three measurements shall be recorded in the field logbook.

2. Independent electrical tape measurements of static water levels using the tape shall agree within ±0.01 foot for depths of less than about 200 feet. At greater depths, independent measurements may not be this close. For a depth of about 500 feet, the maximum difference of independent measurement using the same tape shall be within ±0.1 foot.

3. Decontaminate the electrical tape according to SOP 4-5 before proceeding to the next well to minimize cross contamination.

It may be necessary to check the electrical tape length with a graduated steel tape after the line has been used for a long period of time (at least annually) or after it has been pulled hard in attempting to free the line. Some electrical tapes, especially the single line wire, are subject to becoming permanently stretched.

5.3 Other Water Level Measurement Methods

Although the method cited above (electrical water level indicator) for measuring water levels predominates in the environmental sector, there are a number of other methods available that may be well suited for a particular purpose.

5.3.1 Ultrasonic Method

The ultrasonic method electronically measures the amount of time it takes a sound wave to reach and reflect off the water surface and return to the ground surface. These instruments contain electronic microprocessors, capable of performing this measurement many times each second. The actual depth to water, as calculated by the microprocessor, is an average of many individual readings.

5.3.2 Pressure Gauge Method

This method, also called the air-line submergence method, uses a pressure gauge and is the preferred method for obtaining water level measurements in pumping wells. An air line constructed of semi-rigid tubing is inserted into the well below the water table. The tube end at the surface is connected to an air tank or compressor and pressure gauge. Filtered air is then forced through the tube and the resultant pressure is read in pounds per square inch (psi). This reading is converted to feet of water in the column and subtracted from the total tube length to give depth to water. Readings are then converted to groundwater elevation. Results are plotted on a field logging form. Calibration records and the exact procedures used must be maintained.

5.3.3 Acoustic Probe Method

The acoustic probe is an electronic device containing two electrodes and a battery-powered transducer. The probe is attached to a tape. The probe is lowered into the well until a sound is detected, indicating the electrodes in the probe have contacted the water surface. This method is similar to the electrical probe method discussed in Section 5.2.

5.3.4 Continuous Recording Method

The measurement of groundwater elevations within pumping or monitoring wells can be accomplished by the use of a mechanical or digital analog computerized continuous recording system and shall be performed according to specifications given by the manufacturer of each unit. In general, when using the mechanical or digital system, the pressure or electrical transducer is lowered into the well until it intersects the water surface. The actual depth to water is then measured by one of the methods described above and used to calibrate the continuous recorder.

The necessary adjustments and preparations are then completed according to the specifications given for each type of continuous recorder. Proper maintenance of continuous recording devices during water level monitoring shall be performed such that continuous, permanent records are developed for the specified period of time. Records shall be stored on mechanical graph paper or on a microprocessor. Frequent calibrations of equipment shall also be made during monitoring periods of long duration in accordance with the manufacturers' specifications.

6.0 Restrictions/Limitations

6.1 Groundwater and Miscible Fluids

Where water is rapidly dripping or flowing into a well, either from the top of the well or from fractures, obtaining an accurate reading may not be possible.

The effect of the water flowing into the well may interfere with an electronic water level measuring device, resulting in a false water level measurement. If water levels must be recorded in wells completed in aquifers that are recharging or discharging, the electronic water level indicator is the preferred measuring device, but shall be used with the awareness of possible false measurements. To minimize the effects of "splashing," a 1-inch pipe (decontaminated for environmental wells) may be lowered into the pumping well into which the water level indicator is then inserted. This will minimize the effect of "splashing" until the probe contacts the groundwater. It will also protect the probe from becoming tangled in pump wiring or well spacers associated with downhole equipment such as submersible pumps.

6.2 Immiscible Fluids

For wells containing immiscible contaminants, the field personnel will need to use special procedures for the measurement of fluid levels. The procedure to follow will depend on whether layers are light immiscibles that form lenses floating on the top of the water table, or dense immiscibles that sink through the aquifer and form lenses over less permeable layers.

In the case of light immiscibles, measurements of immiscible fluid and water levels cannot be accomplished by using normal techniques. A conventional electrical tape often will not respond to nonconducting immiscible fluids.

Techniques have been specially developed to measure fluid levels in wells containing immiscible fluids, particularly petroleum products. A special paste or gel applied to the end of the steel tape and submerged in the well will show the top of the oil as a wet line and the top of the water as a distinct color change, or an interface probe can be used that will detect the presence of conducting and nonconducting fluids. Thus, if a well is contaminated with low density, nonconducting immiscible fluids such as gasoline, the probe will first detect the surface of the gasoline, but it will not register electrical conduction. However, when the probe is lowered deeper to contact water, it will detect electrical conduction. Normally, a variation in an audible signal indicates the difference between phases.

Both of these methods have disadvantages. They are less effective with heavier and less refined petroleum products because the product tends to stick to the tape or probe, giving a greater product thickness measurement than is present. Paste or gel cannot be used when sampling groundwater for the same constituents present in the paste or gel product.

Note that water levels obtained in this situation are not suitable for determining hydraulic gradients without further interpretation. To use such data for determining hydraulic gradients, the difference in density between the light immiscible phase and water has to be considered.

Measuring fluid levels in wells screened in lenses of dense immiscible fluids resting on a low permeability formation is somewhat easier, provided the immiscible fluid is nonconducting. The top of the dense layer can be identified by simply using an electrical sounder. As an electrical sounder passes from groundwater into the immiscible phase, the detection unit will deactivate because the fluid will no longer conduct electricity. A better method would be to use an interface probe as described above. The variation in the audible signal associated with the detection of differing phase liquids will also allow the user to obtain a groundwater depth and dense immiscible thickness measurement.

7.0 References

U. S. Environmental Protection Agency. 1987. *A Compendium of Superfund Field Operations Methods*. EPA/540/P-87/001. December.

_____. 2013. Region 4. The Field Branches Quality System and Technical Procedures, Groundwater Level and Well Depth Measurement. SESDPROC-105-R2. January

Weight, Willis D. and Sonderegger, John L., 2001. *Manual of Applied Hydrogeology*. Lewis Publishing Company. 187-190.

Vapor Sampling Using a SUMMA® Canister

TSOP 1-8
Revision: 8
Date: February 2015

Approved: *Signature* **Technical Review:** John Eisenbeis

1.0 Objective

The purpose of this technical standard operating procedure (TSOP) is to define procedures and requirements for collection of vapor using evacuated SUMMA® canister samplers.

2.0 Background

Collection of discrete or temporally composited vapor samples from designated locations on or near a hazardous waste site may be required to characterize and model the nature and extent of ambient, indoor or subsurface contamination. The sampling method described in this TSOP is adapted from U.S. Environmental Protection Agency Method TO-15 Second Edition 1999.

Two types of vapor samples can be collected with SUMMA canisters. The canister can be opened and allowed to fill over a short period to obtain a grab sample or filled slowly by using a flow controller to collect a time-integrated sample.

2.1 Definitions

Canister - Leak-free stainless steel pressure vessels of desired volume (e.g., less than 1 liter (L) to greater than 6 L) are available, with valves and specially prepared (SUMMA) nonreactive interior surfaces. The canister is initially evacuated (under high vacuum) at the laboratory to approximately -30 inches of mercury (Hg). The canisters must be properly cleaned at the laboratory before each use by either ultra-pure humidified air in a series of evacuation/pressurization cycles or in a high temperature oven. The canister is then analyzed by gas chromatograph/ mass spectrophotometer (GC/MS) to verify cleanliness. ***Note***: Canisters previously used for the measurement of high-level contamination may not be suitable for indoor air monitoring, since they may not be able to be adequately decontaminated. A 100 percent certification (individual certification) process is appropriate for ambient and indoor air SUMMA canister sampling applications.

Particulate Matter Filter – 2 or 7-micron filters are built into the flow controllers. The 7-micron filter is primarily used with grab samples. The 2-micron filter is standard for integrated samples. A separate laboratory-cleaned filter is used for each canister sample.

Stainless Steel Vacuum/Pressure Gauges - A gauge capable of measuring vacuum (0 to -30 inches Hg or -100 to 0 kilo pascals [kPa]) and pressure (0 to 30 pounds per square inch [psi] or 0 to 206 kPa) is needed to verify the initial canister vacuum and to measure sampling progress in the sampling system. Gauges shall be tested clean and leak tight.

Sampling Inlet Line/Connectors - Stainless steel or Teflon® tubing (all usually ¼-inch diameter) and appropriate air-tight connectors (e.g., Swagelok® tube fittings) connect the canister to the sample inlet. A Swagelok fitting has four parts, the connector body, two ferrules, and a nut. The larger (cone-shaped) ferrule fits into the connector body, small diameter end first. The smaller end of the smaller ferrule fits on top of the cone ferrule, and the nut is screwed on top. The tubing fits through the nut and the two ferrules, and seats firmly in the connector. On newly created fittings, the nut shall be tightened with two wrenches, 1¼ turns past finger tight to compress the ferrules onto the tubing. Swagelok makes an inspection gauge that fits between the nut and the connector body if a large number of critical connections are to be made. Connections that have been previously made up will only need a short tighten with the wrenches past finger tight.

Stainless Steel Shut-Off Valve - Leak free, for vacuum/pressure gauge.

Electronic Mass Flow Controller - This controller, used to collect composite samples, must be capable of maintaining a constant flow rate (±10 percent) over a sampling period of up to 24 hours and under conditions of changing temperature (20 to 40 degrees Celsius [°C]) and humidity.

Auxiliary Vacuum Pump – The pump continuously draws air through the inlet manifold at 10 L/minute or higher flow rate. The canister vacuum extracts a sample from the manifold at a lower flow rate, and excess air is exhausted. ***Note***: The use of higher inlet flow rates dilutes any contamination present in the inlet and reduces the possibility of sample contamination as a result of contact with active adsorption sites on inlet walls. The auxiliary pump and sampling manifold can also be used to purge vapor monitoring wells.

Elapsed Time Meter/Stopwatch - Measures duration of sampling.

2.2 Associated Procedures

- TSOP 1-2, *Sample Custody*
- TSOP 4-1, *Field Logbook Content and Control*
- TSOP 1-10, *Field Measurement of Organic Vapors*

2.3 Discussion

Ambient air, emission source, or vapor monitoring well samples are collected to determine the type(s) and level(s) of contamination from airborne toxic organic compounds (often important to risk assessment), characterize subsurface vapor contamination, or evaluate vapor intrusion into buildings. These samples may be collected as part of an investigative plan, site-specific sampling plan, and/or as a screen for "hot spots," which may require more extensive sampling. Sampling will include necessary quality assurance/quality control (QA/QC) samples as documented in the project-specific Quality Assurance Program Plan (QAPP), and a field blank and trip blank shall accompany all sampled media.

3.0 Responsibilities

Site Manager - The site manager is responsible for ensuring that SUMMA canister sampling efforts are performed consistently with this procedure and other project-specific documents such as the QAPP, work plan, sampling plan, and the health and safety plan.

Field Team Leader - The field team leader is responsible for ensuring that field personnel collect vapor samples in accordance with this or the project-specific procedure in the field plan and that appropriate documentation is collected.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Required Equipment/Supplies

- Site-specific plans
- Adjustable wrenches (two 9/16-inch opened end wrenches for Swagelok fittings)
- Labels/canister tags (shall be attached to canister) and appropriate laboratory-supplied, individual canister-specific, chain-of-custody forms
- Field logbook or field forms
- Documentation for sample shipment
- Items listed in Section 2.1, Definitions, as applicable
- Sturdy shipping container(s)

5.0 Procedures

5.1 Preparation

1. The analysis of vapor samples collected in canisters will have very low detection limits. Canister preparation shall therefore be performed in clean air environments, away from any type of volatile organic contamination. The canisters are expensive, and, although relatively sturdy, valves and connections can be easily damaged. Do not over tighten valves. Use two wrenches to remove and reconnect line caps and attachments. Using a single wrench may cause excessive torque on the fitting/canister connection.

2. Check the number on the permanent label/tag attached to the canister against the laboratory-supplied chain-of-custody form. Verify that the canister number on the form agrees with the label. Note the date the canister was cleaned, preevacuated canister vacuum, and laboratory analysis verification, etc. The supplied paperwork will vary depending on the laboratory supplying the canister. The objective is to verify that the canister is the correct one, has been cleaned, and has been evacuated to a vacuum approximately -30 inches Hg.

3. In a clean environment, use two wrenches to remove the Swagelok cap from the end of the canister and attach a clean vacuum gauge. Verify that the gauge reads zero. Tighten the gauge fitting tightly with the wrenches. Open the canister valve, read the gauge, and record the reading on the chain-of-custody. SHUT THE VALVE TIGHTLY (turn clockwise). The vacuum reading shall agree closely (the field gauge may not be as accurate as the gauge in the laboratory) with the vacuum noted by the laboratory. If the field vacuum is more than 1 inch of Hg less than the laboratory vacuum, then the canister valve has leaked during the time since the canister was evacuated, and a portion of the canister has been filled with unknown vapor. If the vacuum is not sufficient, replace the canister and document the change. If there are questions, call the laboratory that supplied the canister to discuss further action.

4. If the two vacuum readings agree and the canister is suitable for use, verify that the valve is closed tightly, and remove the vacuum gauge. Attach a clean sample filter (supplied by the laboratory) and replace the Swagelok cap on the filter.

5. If required, build a sampling apparatus using appropriate tubing, connectors, or valves to connect the canister to the sampling location.

6. If a vacuum pump is to be used to purge a soil vapor monitoring well, insert a “tee” fitting between the well and the vacuum pump (upstream of the pump), and a line valve between the tee fitting and the pump. During purging, the line valve is opened and the third leg of the tee is capped, allowing the pump to pull air from the well without short-circuiting through the tee. If a portable photoionization detector (PID), flame ionization detector (FID), or other real-time instrument is to be used to take a field screening reading from the well, a second tee fitting shall be installed on the downstream side of the pump exhaust port. The probe of the instrument is inserted into one leg of the tee, while the other leg is allowed to vent, to prevent pressurizing the instrument and creating false readings.

5.2 Using a Flow Controller for Time-Integrated (Temporally Composited) Sampling

1. To ensure the correct time-integrated sampling rate, the flow controller must be calibrated by a certified laboratory to maintain a constant flow into the canister over the desired sample period. In general, flow controllers are set to standard atmospheric conditions. If the air sample is collected at an elevation or under vacuum, the flow controller needs to be calibrated accordingly. Under standard atmospheric conditions, if a 6-L canister is selected, the flow controllers are usually set to collect a 5-L sample over the sampling interval The flow rate can be calculated by -
   Flow Rate (ml/min) = Target fill volume (ml)/Sampling interval (min).

2. The evacuated canister, flow controller (if required), vacuum gauge, particulate matter filter, and sample inlet need to be assembled (if not already pre-assembled by the offsite laboratory) using two open-end wrenches to tighten the tubing and sampling system components to the canister valve stem.

3. Data entries on the daily sample event data sheet (or equivalent) shall include serial numbers for all numbered sampling components, sample initiation time, sample identification number, sample collection time, separate entries for sub-atmospheric SUMMA canister pressures at sample initiation and completion, sampler initial(s), initiation date, collection date, and sample tag custody number.

5.3 Sample Collection

1. The sample system assembly is transported to the required sample location. Before collecting each sample, confirm that the canister number and sample identification corresponds to the correct sample location.

2. If using a vacuum pump to purge a vapor monitoring well or other space, begin preparing the canister when sufficient purging of the well has been accomplished. Attach a canister with the valve closed to the third leg of the tee upstream of the pump (discussed in Section 5.1), while the pump continues to pull vapor from the well. Do not connect the canister to the downstream side of the pump since the pump may contain contaminants that may be introduced into the sample. The sample is drawn into the canister by the vacuum. When it is time to collect the sample, the valve between the canister and the pump is shut off, preventing backflow through the pump if the canister vacuum happens to be stronger than the pump.

3. When collecting a grab (not a temporally composited) sample, check the tightness of all fittings on the sampling apparatus and the connections on the canister. If sampling ambient air or an emission source, position the canister inlet in the intended environment with the inlet line pointed downward to prevent rainwater from entering the canister.

4. To initiate a sample event, gently open (counter clockwise) the canister valve until a hissing noise is heard. Note the initial vacuum reading on the gauge (attached to the canister with a tee) and that the vacuum level begins to drop. There are differing opinions on how long to allow for the canister to fill, and whether there is any negative effect of opening the valve rapidly. A good compromise is to adjust the valve so there is a slow hiss, allowing the canister to fill in about a half minute. The canister valve can be closed before the hissing stops and the vacuum gauge reaches 0 inches Hg. Record the final vacuum level on the chain-of-custody. Close the valve firmly, without overtightening.

5. Remove the canister from the sampling apparatus, remove the gauge and filter, and replace the canister cap. Do not use the same filter to collect another sample.

6. Record the final vacuum level, duration of sample collection, and any other pertinent sampling information of the chain-of-custody. The final vacuum of a 6-L canister should be between 5 to 10 inches Hg. Complete documentation on the daily event datasheet and the field logbook before leaving each sample location.

7. Place the canister in a sturdy container for shipping. Place all of the used filters in a plastic baggie and return them to the laboratory with the canisters.

6.0 Restrictions/Limitations

The nonreactive inner surface of the SUMMA canister may not be compatible for sampling atmospheres with high levels of chlorine or sulfur. Contact the analytical laboratory for guidance when these elements are suspected.

For 24-hour time-integrated sampling, sample flow rates through the vacuum gauge of the sample system may not remain stable (within the method flow rate tolerance) at vacuums less than 9 inches Hg (e.g., 8 inches Hg). Caution shall be taken in using 24-hour time-integrated samples where final vacuum is less than 4 inches Hg. Flow rates at this vacuum and below are not constant, yielding potentially nonrepresentative results during the latter portion of the sampling period. Any canister that has reached 0 inches Hg vacuum may not be a representative 24-hour sample.

Samples collected at vacuums above 10 inches Hg (e.g., when the canister valve is closed before the gauge reaches 10 inches Hg) may not contain enough sample volume to meet sample volume requirements for high-resolution GC/MS analysis detection limits.

Summa canisters do not require any special storage procedures (e.g., no chilling). Ice crystals can block the flow controller or sampling apparatus at temperatures at or below freezing, Frequent monitoring of canister pressure is recommended during cold periods.

7.0 References

U.S. Environmental Protection Agency. 1999. *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, Second Edition, *Compendium Method TO-15, Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)*. January.

Eurofins/Air Toxics, 2014. Guide to Air Sampling - Canisters and Bags. June.

Tap Water Sampling

SOP 1-9
Revision: 7
Date: February 2015

Approved: Signature **Technical Review:** David Sembrot

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to define the requirements for collecting tap water samples for the purpose of assessing water quality. General guidelines for purging the water supply system before sample collection are also provided. Depending on the objective of the sampling event as defined in the site-specific sampling plan, the water source may be from a private or public potable water supply, such as a groundwater well or a surface water reservoir.

2.0 Background

2.1 Definitions

Holding Tank - An in-house water reservoir that provides a limited reserve water supply and equalizes water pressure throughout the plumbing system. Most domestic well holding tanks have a storage capacity of approximately 30 gallons.

Onsite Water Supply - A source of potable water located on the property to be sampled. The water source could be a groundwater aquifer (i.e., a residential groundwater well) or a surface water body (i.e., a water intake from a lake).

Potable Water - Water considered safe for human consumption.

Tap Water Samples - Samples of water collected from a faucet or spigot at a residence, business, or industrial plant. Usually, samples are collected from the tap(s) nearest the water supply source or area of interest along the distribution system.

Water Filter - A device used to remove suspended particulate matter and/or various compounds from the water source. One type of common filter is a water softener that uses a calcium-salt filter to remove calcium and magnesium ions from potable water to reduce the hardness.

2.2 Associated Procedures

- SOP 1-2, *Sample Custody*
- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-3, *Well Development and Purging*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.3 Discussion

Tap water sampling may be conducted in residential, commercial, or industrial areas. Consequently, sampling personnel will interface with the general public (i.e., homeowners, business owners, or concerned citizens) and must present themselves in the utmost professional manner. Permission to access the property must be obtained before conducting the tap water sampling event; the client shall be consulted as to the proper notification procedures. At the time of the sampling, it is recommended that a letter of introduction be presented to the property owner or representative, explaining the purpose of the tap water sampling and indicating the name of the person and phone number to contact if the property owner has questions. At no time shall the sampling team enter a home or business without the approval of the property owner; the property owner or representative must be present to enter a building.

Generally, water supply sources and distribution systems can be categorized into two types:

- Onsite water supplies such as private, groundwater wells or surface water intakes for single residences, businesses, or industrial plants with limited distribution systems

- Large distribution systems from public or municipal groundwater or surface water supplies with extensive distribution systems for multiple users

The site-specific sampling plan shall describe the source of the potable water supply, the water distribution system, and other site-specific factors that may affect the water quality (well construction details, local hydrogeology, the presence of filters or holding tanks within the distribution system, pipe age, and composition, etc.). It is preferable to collect the samples from a tap located prior to a filtering device or a holding tank so that contaminants will be less likely to have been removed or allowed to settle out. The sampling objectives and sampling requirements, including analytical parameters, preservatives, and sample handling procedures must also be specified. Depending on the water source and distribution system, the site-specific sampling plan shall describe the requirements for purging the system before collecting the tap water sample and for disposing of the purged water.

The procedures described in this SOP provide guidelines to obtain representative tap water samples from water supplies/distribution systems ranging from small onsite water supplies to large multiuser distribution systems.

3.0 General Responsibilities

Field Team Leader - The field team leader is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any associated SOPs.

Sampling Personnel - Field team members are responsible for conducting tap water sampling events in accordance with this procedure, all associated SOPs, and requirements as described in the site-specific plans.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

All or part of the equipment listed may be required at any specific site, depending on the plan(s) for that site.

- Site-specific plans including letter(s) of introduction
- Field logbook and indelible black ink pens and markers
- Forms and other documentation for sample shipment
- Sample containers, labels, and preservatives, as required
- Insulated cooler and waterproof sealing tape
- Ice bags or “blue ice”
- Plastic zip-top bags
- 5-gallon bucket and stopwatch
- Temperature, conductivity, pH, dissolved oxygen, and turbidity meters (with clean beakers or other appropriate containers), as required by the site-specific plans
- Photoionization detector (PID) and/or other monitoring/screening instruments as required by the site-specific health and safety plan or sampling plan
- Decontamination supplies, as required by SOP 4-5
- Personal protective equipment (PPE), as required by the site-specific health and safety plan
- Latex or appropriate gloves

5.0 Procedures

1. Obtain the name(s) of the resident(s) or water supply owner/operator, the exact mailing address, and telephone numbers. This information is required to obtain access to the property to be sampled and to submit a letter of introduction to the owner/representative.

2. Determine the location of the tap to be sampled based on its proximity to the water source. It is preferable that the tap being sampled be prior to any holding or pressure tanks, filters, water softeners, or other treatment devices that may be present.

3. If the sample must be collected at a point in the water line beyond a pressurization or holding tank, a sufficient volume of water shall be purged to provide a complete exchange of fresh water into the tank and at the location where the sample is collected. If the sample is collected from a tap or spigot located just before a storage tank, spigots located inside the building or structure shall be turned on to prevent any backflow from the storage tank to the sample tap or spigot. It is generally advisable to open as many taps as possible during the purge, to ensure a rapid and complete exchange of water in the tanks.

4. Samples collected to determine if system related variables (e.g., transmission pipes, water coolers/heaters, holding/ pressurization tanks) are contributing to the quality of potable water shall be collected after a specific time interval (e.g., weekend, holiday). Sample collection shall consist of an initial flush, a sample after several minutes, and another sample after the system has been purged.

5. Devices such as hoses, filters, or aerators attached to the tap may harbor a bacterial population and therefore shall be removed before sampling.

6. Potable water samples which have been chlorinated must be treated with sodium thiosulfate to dechlorinate the sample prior to other preservation and shipping for analysis.

7. Sample containers shall not be rinsed before use when sampling for bacterial content, and precautions shall be taken to avoid splashing drops of water from the ground or sink into either the bottle or cap.

8. Samples of the raw water supply and the treated water after chlorination shall be collected when sampling at a water treatment plant.

9. In the logbook, record the location and describe the general condition of the tap selected for sampling. The rationale used in selecting the tap sampling location, including any discussions with the property owner, shall also be recorded. Provide a sketch of the water supply/distribution system noting the location of any filters or holding tanks and the water supply source (i.e., an onsite groundwater well or surface water intake or a water service line from a public water main). If an onsite water supply is present, observe and record the surrounding site features that may provide potential sources of contamination to the water supply.

10. Don the appropriate personal protective clothing as dictated by the site-specific health and safety plan. Latex gloves shall be changed between sampling locations to avoid possible cross contamination of the tap water samples.

11. Before sample collection, the supply system shall be purged by turning the cold water tap on. The following general guidelines shall be followed to determine when the system is adequately purged (refer to the site-specific sampling plans for any other requirements):

    - **Onsite Water Supply**. A minimum of three standing volumes of water (i.e., the static volume of water in the well and holding tank, if present) shall be purged. Obtain water temperature, conductivity, and pH measurements after each volume of water is purged. If the standing volume of water in the supply system is unknown, the tap shall be allowed to run for a minimum of 15 minutes and temperature, conductivity, and pH measurements, or other parameters as specified by the project plan, shall be collected at approximately 3- to 5-minute intervals. (In general, well construction details and holding tank volumes shall be obtained before conducting the sampling event to estimate the standing volume of the water supply system.) The system is considered adequately purged when the temperature, conductivity, and pH stabilize within 10 percent for three consecutive readings. If these parameters do not stabilize within 15 minutes, then purging shall be discontinued and tap water samples may be collected as discussed in Section 6.0.

- **Large Distribution Systems**. Because it is impractical to purge the entire volume of standing water in a large distribution network, a tap shall be run for a minimum of 5 minutes, which shall be adequate to purge the water service line. Obtain temperature, conductivity, and pH measurements at approximately 1-minute intervals. The system is considered adequately purged when the temperature, conductivity, and pH readings, or other parameters as specified

  by the project plan, stabilize within 10 percent for three consecutive readings. If these parameters do not stabilize within 5 minutes, then purging shall be discontinued and tap water samples may be collected as discussed in Section 6.0.

  During purging, a 5-gallon bucket and stopwatch may be used to estimate the flow rate if required by the site-specific plans. Dispose of the purged water according to the site-specific plans. Record the temperature/conductivity/pH readings, or other parameters as specified by the project plan, the volume of water purged, the flow rate if measured, and the method of disposal in the field logbook.

12. After purging the supply system, collect the samples directly from the tap (i.e., if a hose was used for purging, the hose shall be disconnected before sampling). Any fittings on the end of the faucet that might introduce air into the sample (i.e., a fine mesh screen that is commonly screwed onto the faucet) shall be removed before sample collection also.

13. Obtain a smooth-flowing water stream at moderate pressure with no splashing. Samples for volatile organic compound (VOC) analyses shall be collected using a reduced flow rate (see below). Hold the sample bottle in one hand and the cap in the other; do not touch the inside of the cap; do not allow the faucet to touch the inside of the bottle; do not allow splashing water from the ground or sink to enter the bottle or cap. VOC samples shall be filled first, followed by other organic analyses, inorganic analyses, and then other water quality parameters. Refer to the site-specific plans for the required sample parameters, preservatives, and sample handling procedures. The following general guidelines shall be followed when collecting samples:

    - **VOC**. Reduce the flow rate to a minimum to reduce aeration of the VOC sample. Use a pre-preserved "test" vial to determine the appropriate amount of hydrochloric acid (HCl) needed to reduce the pH of the sample to less than 2. Dispose of this test vial after the appropriate amount of HCl is determined. Add the required amount of HCl to the sample vials and then fill the vials with the sample water. Quickly replace the cap and check for air bubbles. If air bubbles are present, the vial will be discarded and a new vial will be filled as detailed above.

    - **Semivolatile Organic Compounds (SVOCs), Pesticides, and Polychlorinated Biphenyls (PCBs)**. Generally, aqueous samples for SVOCs and pesticides/PCBs require no preservative. Sample containers may be filled directly from the tap.

    - **Total (unfiltered) Metals**. Generally, tap water samples are not collected for filtered (dissolved) metals because risk assessment data needs require total metals analyses (check the site-specific plans to determine filtering requirements). The sample container for total metals may be filled directly from the tap. Nitric acid (HNO_3) shall then be added to the filled container to preserve the sample to a pH less than 2.

    - **Other Sample Parameters**. Other water quality parameters, such as cyanide dissolved oxygen, hardness, nitrate/nitrite, etc., shall be collected and preserved as required by the site-specific sampling plans.

14. Label all sample containers as required and place them in a cooler with ice. Record all appropriate data in the field logbook and on the chain-of-custody forms.

6.0 Restrictions/Limitations

To protect the sample from contamination on the exterior of a tap, a tap shall not be chosen for sampling if any of the following conditions exist:

- A leaky tap allowing water to flow out from around the stem of the valve handle and down the outside of the faucet.
- A tap located too close to the bottom of the sink or the ground surface.
- A tap that allows water to run up on the outside of the lip.

- A tap that does not deliver a steady stream of water. A temporary fluctuation in line pressure may cause sheets of microbial growth, lodged in some pipe sections or faucet connections, to break loose.

Careful sampling for VOC analysis, or for any other compound(s) that may be degraded by aeration, is necessary to minimize sample disturbance and, hence, analyte loss.

7.0 References

U. S. Environmental Protection Agency. 2013. Region 4. The Field Branches Quality System and Technical Procedures, Potable Water Sampling. SESDPROC-305-R3. May.

Field Measurement of Total Organic Vapors

SOP 1-10
Revision: 7
Date: February 2015

Approved: *Signature* **Technical Review:** Stuart Barden

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to define the techniques and the requirements for the measurement of total organic vapors in the field.

2.0 Background

2.1 Definitions

Photoionization Detector (PID) - A portable, hand-held instrument that measures the concentration of gaseous organic compounds through the photoionization of organic vapors.

Flame Ionization Detector (FID) - A portable, hand-held instrument that measures the concentration of gaseous organic compounds through the flame ionization of organic vapors.

2.2 Associated Procedures

- SOP 1-4, *Subsurface Soil Sampling*
- SOP 1-5, *Groundwater Sampling Using Bailers*
- SOP 1-6, *Water Level Measurement*
- SOP 1-8, *Volatile Organic Compound Air Sampling Using USEPA Method TO-15 with SUMMA Canister*
- SOP 1-12 *Low-Stress (Low-Flow) Groundwater Sampling*
- SOP 3-1, *Geoprobe® Sampling*
- SOP 3-5, *Lithologic Logging*
- SOP 4-3, *Well Development and Purging*

2.3 Discussion

The measurement of organic vapors is a required step during numerous field activities. The primary purpose of such measurements is health and safety monitoring to determine if the breathing zone in a work area is acceptable or if personal protective equipment such as a respirator or a supplied air device is necessary for field personnel. At the perimeter of a work area, measurements can be taken as part of a perimeter air monitoring plan to document protection of surrounding community. In addition to health and safety monitoring, total organic vapor measurement is also used in conjunction with sampling activities, including screening subsurface soil samples, soil vapor and indoor air sampling, and groundwater sampling, where measurements are useful for establishing approximate contaminant levels or ranges.

The two types of instruments most commonly used to measure total organic vapors are PIDs and FIDs. Both instruments first ionize the gaseous compound and then measure the response, which is proportional to the concentration.

2.3.1 PID Operation

The PID is preferred when the compound of interest is an aromatic or halogenated volatile organic compound (VOC). The PID ionizes the sampled vapors using an ultraviolet lamp that emits light energy at a specific electron voltage (eV - labeled on the lamp). The ultraviolet lamp produces photons that are absorbed by the sampled vapor molecule. The molecule becomes excited, producing a positively charged ion and emitting an electron. The number of electrons emitted is proportional to the concentration of the sampled gases. Every organic compound has a specific ionization potential in electron volts. The energy emitted by the lamp must be higher than the ionization potential of the compound for the compound to become ionized and emit an electron. If the ionization potential of the compound is higher than the eV of the lamp, there will be no response on the instrument. Therefore, the ionization potential of the known or suspected compounds shall be checked against the energy of the ultraviolet lamp to verify that the energy provided by the lamp is greater. The manufacturer's manual shall be consulted to determine the appropriate ultraviolent lamp to be used for the known or suspected compounds. Additionally, manufacturer's manuals shall be consulted to obtain the appropriate correction factors for known or suspected contaminants.

Water vapor in the vapor sample can interfere with the PID detector and cause the instrument to stop responding or cause the zero baseline to drift. This can occur using the PID on a rainy day or when sampling headspace samples that have been in the sun. If moisture interference is suspected, the calibration gas shall be used to check the instrument response by inserting the gas as a check sample, not by recalibrating. If the response is lower than the gas level, then the probe and the ionization chamber shall be dried out before reusing the instrument.

The sampling probe shall not be inserted directly into soil samples or dusty areas, as the instrument vacuum will pull dirt into the ionization chamber. Under particularly dirty or dusty conditions, the lamp may become covered with a layer of dust. If dirty conditions are encountered, or if the instrument response seems to have decreased, then the lamp shall be cleaned. The instrument manual provides instructions on how to remove the instrument cover to access the lamp, and how to clean the screen in the ionization chamber and the surface of the lamp.

The instrument manual may provide instruction on use of disposable dust and/or moisture filters for minimizing effects from dust and/or moisture.

The ultraviolet lamp in the PID is sensitive to shock, especially when using the higher eV lamps. Therefore, they shall be handled and transported carefully.

2.3.2 FID Operation

The FID is preferred when sampling for petroleum hydrocarbons and methane (landfill gases). It responds well to aromatic hydrocarbons but is not as convenient to use as the PID. The FID allows measurement of a wide variety of compounds, but in general its sensitivity is not as high as the PID for compounds where the PID is applicable. The FID is virtually unaffected by ambient levels of water vapor.

The FID ionizes the vapor sample by burning it in a hydrogen/air flame, and measuring the response beyond what is caused by the hydrogen alone. This instrument requires a hydrogen supply, contained in a small tank in the instrument. This hydrogen, including the gas in the instrument tank, is considered a flammable gas and appropriate requirements must be adhered to when shipping. The instrument shall be emptied of hydrogen before shipping. Federal Express Hazardous Material shipping manifests must be completed when shipping the gas.

The hydrogen gas in the FID combustion chamber is ignited by pressing a red button on the side of the instrument, which sends electrical current to a small resistance coil igniter in the combustion chamber. This igniter is very sensitive, and if the red button is pressed for longer than 5 seconds, the coil will burn out and the instrument will be unusable unless another igniter is available. If the instrument will not light, check the electrical connections and switches for proper settings. Check that the pump is pumping, and allow fresh air to flow through the combustion chamber for several minutes before lighting. Check to see if the exhaust port of the combustion chamber is dirty.

3.0 Responsibilities

Site Manager - The site manager is responsible for ensuring that field activities are conducted in accordance with this procedure and any other SOPs pertaining to the specific activity.

Field Team Leader - The field team leader is responsible for ensuring that field personnel conduct field activities in accordance with this and other relevant procedures.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

Field Measurement of Total Organic Vapors

SOP 1-10
Revision: 7
Date: February 2015

4.0 Required Equipment

- Site-specific plans (i.e., scope of work)
- Health and safety plan
- Field logbook
- Waterproof black ink pen
- Personal protective clothing and equipment
- Photoionization detector or flame ionization detector
- Calibration gases in a range appropriate for the expected use
- 0.5 liter (16-ounce) or "Mason" type glass jar or Ziploc-type plastic bags
- Hydrogen canister and fill valve and hose (if using FID for a period of more than 1 day)

5.0 Procedures

5.1 Direct Reading Measurement

1. Connect the measurement probe to the instrument and make necessary operational checks (e.g., battery check, etc.) as outlined in the manufacturer's manual.

2. Calibrate the instrument following the applicable manufacturer's manual

3. Make sure the instrument is reading zero and all function and range switches are set appropriately.

4. Insert the end of the probe directly into the atmosphere to be measured (e.g., breathing zone, monitoring well casing, split spoon, etc.) and read the total organic vapor concentration in parts per million (ppm) from the instrument display. Apply the appropriate correction factor if necessary. Record the highest instrument response.

5. Immediately document the reading in the field logbook or on the appropriate field form.

5.2 Headspace Measurement

1. Connect the measurement probe to the instrument and make necessary operational checks (e.g., battery check, fan check, etc.) as outlined in the manufacturer's manual.

2. Calibrate the instrument following the appropriate manufacturer's manual.

3. Make sure the instrument is reading zero and all function and range switches are set appropriately.

4. Fill a clean glass jar or Ziploc-type plastic bag approximately half-full of the sample to be measured. For a jar, quickly cover the top of the jar with one or two sheets of clean aluminum foil and apply cap to seal the jar. For a bag, quickly seal the bag minimizing volume of air in bag.

5. Allow headspace to develop for approximately 10 minutes. It is generally preferable to shake the sealed jar for 10 to 15 seconds at the beginning and end of headspace development. For a bag, kneed the bag to break apart the sample and maximize sample surface area.

Note: When the ambient temperature is below 0°C (32°F), the headspace development and subsequent measurement shall occur within a heated vehicle or building.

6. For a jar, remove the jar cap and quickly puncture the foil and insert the instrument probe to a point approximately one-half of the headspace depth. Do not let the probe contact the soil. For a bag, quickly puncture the bag wall and insert the probe, wrapping the bag wall around the probe stem to minimize loss of vapors. If using a PID and there is condensation on the inside of the jar or bag, only leave the probe in the jar or bag long enough to obtain a reading. Remove the probe and allow fresh air to flow through the instrument to avoid excess water vapor to build up.

7. Read the total organic vapor concentration in ppm from the instrument display. Apply the appropriate correction factor if necessary. Record the highest instrument response.

8. Immediately record the reading in the field logbook or on the appropriate field form.

6.0 Restrictions/Limitations

The two methods outlined above are the most commonly used for field measurement of total organic vapors but do not apply to all circumstances. Consult project- or program-specific procedures and guidelines for deviations. Both the PID and FID provide quantitative measurement of total organic vapors, but generally neither instrument is compound-specific. The typical reading range of the PID is 0 to 2,000 ppm, and the typical reading range of the FID is 0 to 10,000 ppm. The FID will measure methane while the PID will not. ***Note***: The presence of methane will cause erratic PID measurements. In methane rich environments, toxic organic vapors shall be monitored with an FID. If desired, a charcoal filter can be placed temporarily on the FID inlet probe, which will trap all organic vapors except methane. The filtered (methane only) reading can be subtracted from unfiltered (total organic vapors) to provide an estimate of non-methane organic vapors. The reading accuracy of both instruments can be affected by ambient temperature, barometric pressure, humidity, lithology, etc.

7.0 References

Department of Defense. *Environmental Field Sampling Handbook, Revision 1*. April 2013 or current revision.

Sediment and/or Sludge Sampling	SOP 1-11 Revision: 10 Date: February 2015

Approved: *Signature* **Technical Review:** Stuart Barden

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define requirements for collection and containment of samples collected from freshwater or marine sediment, and/or sludge samples.

2.0 Background

2.1 Definitions

Sediment - Bottom substrate underlying a body of surface water, whether freshwater or marine, such as a lake pond, harbor, river, bay or other surface water bodies.

Sludge - Bottom substrate underlying an engineered wastewater pond or solid material removed from a wastewater treatment stream or effluent. Sludge materials range in type from dewatered solids to high viscosity liquids. Sludge particles may be suspended throughout the water column or settled to the bottom as the bottom substrate.

Grab Sample - A surface sample from the sediment or sludge taken from a specific location at a given point in time. Used for horizontal characterization of sediment or sludge.

Core Sample - A subsurface sample taken from the sediment or sludge using a coring device that allows for penetration to a greater vertical depth; used for more comprehensive, vertical characterization of sediment or sludge.

Composite - Two or more subsamples taken from the sediment or sludge at a specific location at a specific point in time, which are then combined and homogenized (mixed) to form a single sample when removed from the homogenate.

Discrete - An individual sample that is taken from the sediment or sludge and analyzed as an individual sample, rather than combined or homogenized to create a composite sample.

2.2 Associated Procedures

- SOP 1-2, *Sample Custody*
- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-2, *Photographic Documentation of Field Activities*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.2 Discussion

Sediment/sludge samples are collected to physically, chemically, and/or biologically characterize the nature of the substrate within a given surface water body. Sediment and sludge samples offer the advantage over surface water samples that they provide a more stable, site-specific, and possibly historical account of contamination or other features than surface water samples, as surface water may be flowing, is transitory, and thus more difficult to ascribe specific characteristics to any given specific location.

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that field personnel are trained in the use of this and related SOPs and the required equipment.

Field Team Leader - The field team leader (FTL) is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any other specific SOPs pertaining to specific sample collection requirements. The FTL must also ensure that the quantity, locations, and procedures for collecting sediment/sludge sampling meet the requirements of any approved site-specific plans such as sampling and analysis plans (SAPs), field sampling plans (FSPs), and quality assurance project plans (QAPPs).

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field sampling plan and/or site quality assurance project plan (QAPP).

4.0 Required Equipment

All or part of the equipment listed under the "as needed" category may be required at any specific site, depending on the plan(s) for that site.

- Site-specific plans
- Field logbook
- Indelible black ink pens and markers
- Labels and appropriate forms/documentation for sample shipment
- Appropriate sample containers
- Insulated cooler and waterproof sealing tape
- Ice bags or "blue ice"
- Plastic zip-top bags
- Clear waterproof tape
- Personal protective clothing and equipment (e.g. hard hat, etc.)
- Latex or other appropriate gloves, boots
- Rubber boots and/or rubberized waders
- Stainless steel or Teflon® spoons, spatulas, or scoops
- Teflon or stainless steel mixing bowls or trays
- Aluminum foil
- Kimwipe or paper towels
- ½- to ¾-inch (12- to 19-mm) braided nylon line or Teflon-coated wire rope
- Clean plastic sheeting
- Tap and deionized water with spray bottles
- Appropriate photographic equipment and supplies
- Appropriate decontamination equipment and supplies (e.g. detergent, scrub brushes, buckets for capturing investigation-derived waste)
- Eckman, Ponar, Van Veen, or other grab sampling device for depositional area (e.g. stream) sediment or lake sampling
- Chain of Custody forms from laboratory(ies) as appropriate

As needed:

- Global Positioning System (GPS) unit
- Hand or gravity corer with extensions or stainless steel hand auger
- Core liners of Teflon, stainless steel, brass, aluminum, or polybutyrate, as specified in the site-specific plan(s)
- Stainless steel push tubes
- Dredge with 15- to 20-foot (4.5- to 6.0-meter) sampling pole (hollow) and insert (e.g., Peterson, Eckman, Ponar)
- Motorized or other coring device
- Boat with depth finder for deep water or inaccessible shorelines
- Any personal protective equipment specified in the site-specific health and safety plan
- Spare parts for all equipment
- Tape measure

Sediment and/or Sludge Sampling

SOP 1-11
Revision: 10
Date: February 2015

5.0 Procedures

5.1 Preparation

The following steps shall be taken when preparing for sampling sediment/sludge:

1. Review site-specific health and safety plan (HSP) and project plans (FSP, QAPP) before initiating sampling activity.
2. Don the appropriate personal protective clothing as described by the HSP.
3. Use field GPS unit to identify selected sediment or sludge samples within the specified tolerance (e.g. within 2 meters, etc.).
4. Where specific locations are unspecified in the project plans, within a stream or river, avoid areas where the channel has been scoured or where bedrock is present, as it may not be possible to collect samples at these locations.
5. Where excessive organic materials (e.g., root mass, leaf litter) or large grain sizes (e.g. medium to coarse gravel) are present, these material should be minimized in the collected sample.
6. Prepare sampling site by laying out clean plastic sheeting on the ground or any flat, level surfaces near the sampling area and place equipment to be used on the plastic.
7. If surface water is present at the sample location, make field measurements in physical, chemical, and biological characteristics of the water (e.g., temperature, dissolved oxygen, conductivity, pH, etc.), as described by the project-specific plans.
8. The samples shall be collected from areas of least to greatest contamination (when known) and, when collecting several samples in one day in a flowing water body, should be always collected from downstream to upstream.
9. When sampling sediment and surface water from the same surface water body, collect surface water samples before sediment samples to avoid collection of resuspended sediment or sludge particulates.
10. Document the sampling events, recording all information in the designated field logbook and take photographs (as appropriate). Document any and all deviations from this SOP in the field notebook and include rationale for changes.
11. The sample collection points should be shown on a site map and described in the field logbook; any deviations from the site plans should be documented, as well as the rationale for this deviation.
12. Label each sample container with the appropriate information. Secure the label by covering it with waterproof clear tape.
13. Decontaminate reusable sampling equipment after sample collection according to SOP 4-5.
14. Procedures for verifying depths of samples must be included in site-specific project plans.
15. Check that a trip blank/temperature blank, when necessary, is included in the chilled cooler. Quality assurance/quality control requirements vary from project to project. Consult the project-specific work plan for quality requirements.

5.2 Sediment/Sludge Sample Collection from Shallow Waters

5.2.1 Method for Collecting Samples for Volatile Organic Compound (VOC) Analysis

The following steps must be followed when collecting shallow water sediment/sludge VOC samples:

1. Use a decontaminated stainless steel or Teflon, long-handled scoop, corer, push tube, or dredge to collect the entire sample in one grab. If wading is necessary, approach the sample location from downstream. Do not enter the actual sample area.

2. Retrieve the sampling device and slowly decant off any liquid phase.

3. Immediately fill the specified sample container(s) with the solid. Use a clean stainless steel or Teflon spoon or spatula to completely fill the container(s), ensuring no headspace.

   Note: Samples to be analyzed for VOC or other compounds degraded by aeration shall be taken as grab samples. Do not homogenize or composite these samples.

4. Once each container is filled, close the container with the Teflon-lined cap. Wipe the outside of the container clean with a Kimwipe or clean paper towel. Affix a completed sample label.

5. Place the sample container(s) in individual zip-top plastic bags and seal the bags.

6. Immediately pack all samples into a chilled cooler.

5.2.2 Method for Collecting Samples for Non-volatile, Semi-Volatile Organic and Inorganic Compound Analysis

The following steps must be taken when collecting shallow water sediment or sludge samples for analytes not immediately degraded by aeration:

1. Collect sufficient volume to fill specified sample containers using decontaminated stainless steel or Teflon-lined equipment (scoops, corer, dredge sampler, etc.). If wading is necessary, approach the sample location from downstream. Do not enter the actual sample area.

2. Retrieve the sampling device with the sample and slowly decant off any liquid phase.

3. Pool and homogenize samples in a stainless steel, Teflon, or appropriate pan or mixing bowl, using stainless steel spatula or spoon.

4. Fill each sample container with the homogenized sample to approximately 75 to 90 percent capacity, filling sample containers for organics analyses first.

5. Once each container is filled, close the container with a Teflon-lined cap. Wipe the outside of sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.

6. Place the sample container(s) in individual zip-top plastic bags and seal the bags.

7. Immediately pack all samples into a chilled cooler, if required for preservation.

5.3 Subsurface Sediment/Sludge Sample Collection Using a Corer or Auger from Shallow Waters

5.3.1 Method for Collecting Samples for Volatile Organic Compound Analysis Using an Unlined Corer (also applies to augers)

The following steps must be taken when collecting subsurface sediment or sludge VOC samples that underlie shallow water:

1. At the specified sampling location, force or drive the corer to the specified depth.

2. Twist and withdraw the corer in a smooth motion.

3. Retrieve the sampling device, remove the corer nosepiece (if possible), and extrude the sample into the specified sampling container(s). Use a clean stainless steel or Teflon spoon or spatula to completely fill the container(s), ensuring no headspace.

4. Once each container is filled, close the container with the Teflon-lined cap. Wipe the outside of the sample container clean with a Kimwipe or clean paper towel. Affix a completed sample label.

5. Place the sample container(s) in individual zip-top plastic bags and seal the bags.

6. Immediately pack all samples into a chilled cooler.

5.3.2 Method for Collecting Samples for Volatile Organic Compound Analysis Using a Lined Corer

The following steps must be followed when collecting shallow water subsurface sediment/sludge VOC samples that underlie shallow water:

1. Install decontaminated liner(s) in the corer barrel.

2. At the specified sampling location, force or drive the corer to the specified depth.

3. Twist and withdraw the corer in a smooth motion.

4. Retrieve the sampling device, remove the corer nosepiece (if possible) and remove the liner(s), cap the liner(s), and seal the caps with Teflon tape.

5. Wipe the outside of the liner clean with a Kimwipe or clean paper towel. Label the top and bottom ends of the liner(s). Affix a completed sample label.

6. Place capped and sealed liners in individual zip-top plastic bags and seal the bags.

7. Immediately pack all samples into a chilled cooler.

5.3.3 Method for Collecting Samples for Non-volatile, Semi-Volatile Organic and Inorganic Compound Analysis Using a Corer (also applies to augers)

The following steps must be followed when collecting subsurface sediment/sludge samples that underlie shallow water for analytes not degraded by aeration:

1. At the specified sampling location, force or drive the corer to the specified depth.

2. Twist and withdraw the corer in a smooth motion.

3. Retrieve the sampling device. Remove the corer nosepiece (if possible) and extrude the sample into a stainless steel or Teflon-lined pan or bowl. Collect sufficient sample volume to fill all containers.

4. Use a stainless steel or Teflon spoon or spatula to homogenize and then divide the sample material into the appropriate number of sample containers.

5. Fill each container to approximately 75 to 90 percent capacity, filling containers for organics analyses first. Close the container with a Teflon-lined cap. Wipe the outside of sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.

6. Place the sample container(s) in individual zip-top plastic bags and seal the bags.

7. Immediately pack all samples into a chilled cooler.

5.4 Sediment or Sludge Sample Collection Using a Dredge (Grab) Sampler from Deeper Waters

5.4.1 Method for Collecting Samples for Volatile Organic Compound Analysis

The following steps must be followed when collecting deep-water sediment/sludge VOC samples:

1. Attach a clean piece of ½- to ¾-inch (12- to 19-mm) braided nylon line or Teflon-coated wire rope to the top of the dredge sampler. The line must be of sufficient length to reach the sediment or sludge and have enough slack to release the mechanism. Mark the distance to the bottom on the line.

2. Attach the free end of the sampling line to a fixed support to prevent loss of the sampler.

3. At the specified sampling location, open the sampler jaws and slowly lower the sampler until contact with the bottom (sediment/sludge) is felt.

4. Release tension on the line; allow sufficient slack for the mechanism (latch) to release. Slowly raise the sampler to reduce disturbance of the sampler to the extent possible.

5. Once the sampler is above the water surface, place the sampler in a stainless steel or Teflon-lined tray or pan. Open the sampler. Immediately collect the sample for VOC analysis, using a stainless steel or Teflon spoon or spatula. Fill each container completely to minimize headspace.
6. Once each container is filled, close the container with the Teflon-lined cap. Wipe the outside of sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.

7. Place the sample container(s) in individual zip-top plastic bags and seal the bags.

8. Immediately pack all samples into a chilled cooler.

5.4.2 Method for Collecting Samples for Non-volatile, Semi-volatile Organic and Inorganic Compounds

The following steps must be followed when collecting deep-water sediment/sludge samples for analytes not degraded by aeration:

1. Attach a clean piece of ½- to ¾-inch (12- to 19-mm) braided nylon line or Teflon-coated wire rope to the top of the sampler. The line must be of sufficient length to reach sediment or sludge and have enough slack to release the mechanism. Mark the distance to the bottom on the line.

2. Attach the free end of the sampling line to a fixed support to prevent loss of the sampler.

3. At the specified sampling location, open the sampler jaws and slowly lower the sampler until contact with the bottom (sediments/sludge) is felt.

4. Release tension on the line; allow sufficient slack for the mechanism (latch) to release. Slowly raise the sampler to reduce disturbance of the sampler to the extent possible.

5. Once the sampler is above the water surface, place the sampler in a stainless steel or Teflon-lined tray or pan. Open the sampler.

6. Collect sufficient volume of sample to fill the specified sampler containers, and place the material into a clean stainless steel bowl or other container to homogenize the sample. If compositing is required, pool the grab samples in a tray or other container, and homogenize the pooled samples by mixing them together with a stainless steel or Teflon spoon or spatula.

7. Fill the specified sample containers to approximately 75 to 90 percent capacity with the homogenized sample using the stainless steel or Teflon spoon or spatula. Fill sample containers for organics analyses first.

8. Once each container is filled, close the container with the Teflon-lined cap. Wipe the outside of sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.

9. Place sample container(s) in individual zip-top plastic bags and seal the bags.

10. Immediately pack all samples into a chilled cooler.

6.0 Restrictions/Limitations

Core sampling devices may not be usable if cobbles exist in the sediment/sludge. Bumping of core sampling devices and Ponar dredge samplers may result in the loss of some of the sample.

Grab sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration is necessary to minimize sample disturbance and, hence, analyte loss. The representativeness of this sample, however, is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed.

7.0 References

Department of Defense. *Environmental Field Sampling Handbook, Revision 1*. April 2013 or current revision.

Mudroch, A. and S.D. MacKnight. 1991. Handbook of Techniques for Aquatic Sediment Sampling. CRC Press, Inc. 210 p.

U.S. Environmental Protection Agency, Region IV. *Science and Ecosystem Support Division, Operating Procedure SESDPROC-300-R3*. August 2014 or current revision.

U. S. Geological Survey. *National Field Manual for the Collection of Water-Quality Data*, Chapter A8. October 1997.

Low- Stress (Low-Flow) Groundwater Sampling	SOP 1-12 Revision: 2 Date: February 2015
Approved: Joyell Mullins	**Technical Review:** Lynne France

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define the procedural requirements for low-flow (minimal drawdown) groundwater sampling.

2.0 Background

Low-flow groundwater sampling is a method of collecting samples from a well that, unlike traditional purging methods, does not require the removal of large volumes of water from the well. The objective of low-flow groundwater sampling is to collect samples with minimal alterations to water chemistry through pumping the well at a rate low enough to minimize drawdown and to avoid disturbance in the well. Low-flow groundwater sampling refers to the velocity with which water enters the pump intake, and that which is imparted to the formation pore water in the immediate vicinity of the well screen. It does not necessarily refer to the flow rate of water discharged at the surface of the well which can be affected by flow regulators or restrictions.

Water level drawdown provides the best indication of the stress imparted by a given flow-rate for a given hydrological situation. The objective of low-flow groundwater sampling is to pump the well in a manner that minimizes stress (drawdown) to the system. Minimal drawdown must be stabilized such that the water to be sampled is representative of the formation surrounding the screened interval, and is not from the stagnant water column above the screened interval. Minimal drawdown is achieved to the extent practical taking site sampling objectives into account. Typically flow rates on the order of 0.1 to 0.5 liter per minute (L/min) are used. However, achieving flow rates of 0.1 to 0.5 L/min can be dependent on site-specific hydrogeology. Some very coarse-textured formations have successfully been sampled via low-flow techniques at flow rates up to 1 L/min. The effectiveness of using low-flow purging is intimately linked with proper well screen location, well screen length, and well construction and development techniques.

Low-flow groundwater sampling can be used to collect samples for all categories of aqueous-phase contaminants and naturally occurring analytes, including volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), and other organic compounds; metals and other inorganics; pesticides; polychlorinated biphenyls (PCBs); radionuclides; and microbiological constituents. Low-flow groundwater sampling techniques are particularly well-suited in applications where it is desirable to sample aqueous-phase constituents that may sorb or partition to particulate matter. It is not applicable to sampling wells that contain either light or dense non-aqueous-phase liquids (LNAPLs or DNAPLs).

A variety of sampling devices are available for low-flow groundwater sampling, including peristaltic pumps, bladder pumps, electrical submersible pumps, and gas-driven pumps. Pump type should be selected based on known site conditions, including well depth, well diameter, water level, and anticipated well volume, as well as sampling objectives. Note that peristaltic pumps (suction pumps) cannot be used under conditions in which the water table is greater than 25 feet below ground surface. Additionally, in most instances, peristaltic pumps may not be used for collecting VOC samples because they create a vacuum that potentially contributes bias to sampling for VOC's via low flow technicques. Bailers, too, are generally inappropriate devices for low-flow sampling. Gas-driven pumps are generally not advisable for VOC or SVOC sample collection due to the potential for sample contamination.

Dedicated pumps (those which are permanently installed in the well –e.g., bladder pumps) are preferred over portable pumps because they eliminate disturbance to the water column in the well during pump insertion, thus providing lower turbidity values, shorter purge times, and lower purge volumes to achieve stabilized indicator parameter measurements. However, portable pumps can be used if care is taken to minimize disturbance to the water column during pump insertion, and if adequate time is allowed following pump insertion and prior to pump operation for any particulates agitated in the water column to settle. Both dedicated and portable pumps should be easily adjustable, and should operate reliably at lower flow rates. All pumps typically have some limitations which should be evaluated with respect to site-specific considerations and data quality objectives on a case-by-case basis.

Low- Stress (Low-Flow) Groundwater Sampling	SOP 1-12 Revision: 2 Date: February 2015

Water quality indicator parameters should be continuously monitored during low-flow purging using a flow-through cell, or in-line parameter monitoring techniques. Continuous indicator parameter monitoring is a critical component to low-flow groundwater sampling. Water quality indicator parameters include temperature, pH, oxidation-reduction potential (ORP), specific conductivity, dissolved oxygen (DO), and turbidity. The flow-through cell enables continuous collection of real-time parameters during low-flow purging. Stabilization is achieved after all parameters fall within established limits for three successive readings as discussed in Section 5. Stabilization of low-flow parameters is further discussed in Section 5.0 (Procedure) of this SOP.

Advantages of low-flow groundwater sampling are:
- Improved sample quality (e.g., less turbid and more representative of the aquifer)
- Potentially reduced purging and sampling times
- Reduced purge water volume

2.1 Associated Procedures
- SOP 1-6, *Water Level Measurement*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*
- SOP 2-2, *Guide to Handing of Investigation Derived Waste*

3.0 General Responsibilities
Site Manager - The site manager is responsible for ensuring that field personnel are trained in the use of this procedure and for verifying that well development and purging are carried out in accordance with this procedure.

Field Team Leader - The field team leader is responsible for complying with this procedure.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Required Equipment
- Pump (including peristaltic pumps, bladder pumps, electrical submersible pumps, and gas-driven pumps as discussed in Section 2.0 of this SOP)
- Appropriate controller for selected pump type
- For bladder pumps: Compressor and controller for the system (compressed non-reactive gas may also be used in lieu of a compressor)
- Power source (e.g., battery or generator), as required
- Pump tubing (typically polyethylene with Teflon® lining).Note that portable bladder pumps require combination tubing (for air and water); therefore the correct tubing sizes for the portable bladder pump should be verified. Additionally, peristaltic pumps require flexible tubing (silicone or Tygon) tubing for the pump head.
- Electronic water level meter or oil-water interface probe (according to SOP 1-6)
- Water quality meter(e.g., YSI 600 Series) with a closed flow-through cell for continuous in-line measurement of temperature, pH, conductivity, ORP, and DO prior to sample collection
- Turbidity meter (Reporting in nephelometric turbidity units [NTUs])
- Standards for calibration and field check, as needed, of water quality and turbidity meters (as determined by anticipated field conditions)
- Volume measuring device to determine flow (e.g., graduated cylinder)
- Stop watch
- Tape measure
- Personal protective equipment as specified in the site-specific health and safety plan
- Polyethylene sheeting

- Sample containers, including packaging supplies and all associated paperwork (e.g., chain of custody forms) as required in the sampling plan and/pr SOP 2-1, Packaging and Shipping Environmental Samples
- Decontamination supplies, as required, according to SOP 4-5
- Disposal drums (e.g., 55-gallon Department of Transportation-approved) or other purge water storage container, if required by the site-specific sampling plan
- Photoionization detector (PID)/organic vapor monitor (OVM) or equivalent as specified in site-specific health and safety plan

Note: All sampling devices (bladders, pumps, and tubing) should be constructed of stainless steel, polyethylene, Teflon®, glass, or similar non-reactive materials.

5.0 Procedure

The following steps must be followed for low-flow groundwater sampling activities:

1. Review site-specific health and safety plan, and site-specific project and sampling plans before initiating sampling activities.

2. Review available existing data for site to evaluate approach to sampling site wells: prepare to sample site wells in the order of least contaminated to most contaminated. Additionally, existing site data should be reviewed to determine anticipated hydrogeologic conditions and well completion details.

3. Prior to sampling, all sampling devices and monitoring equipment shall be calibrated according to manufacturer's recommendations and the site-specific sampling plan. Calibration of pH should be performed with at least two buffers which bracket the expected pH range. DO calibrations should be corrected for local barometric pressure readings and altitude.

4. Put on personal protective clothing and equipment as specified in the site-specific health and safety plan.

5. Open the well cover and check condition of the wellhead, including the condition of the surveyed reference mark, if any. If no reference mark exists, create a reference mark on the north side of the well riser using a permanent marker or equivalent. Record the location of the reference mark in the field notes.

6. Monitor the air space at the wellhead for VOCs using a PID/OVM or equivalent immediately upon removal of the well plug and as according to health and safety requirements.

7. Determine the depth to static water level in accordance with SOP 1-6, taking precautions to minimize disturbance of the stagnant water column above the screened interval during water level measurement. Well depth should be obtained from review of the well completion logs or from previous work. Insertion of a water level measuring device to the bottom of the well casing will result in resuspension of settled solids from the formation surrounding the screened interval, thus requiring longer purging times for turbidity and other field parameter equilibration.

8. Dedicated sampling devices (those permanently installed in the well) capable of pumping and sampling are preferred over any other type of device. Any portable sampling device should be slowly and carefully lowered to the middle of the screened interval or slightly above the middle to minimize excessive mixing of the stagnant water in the casing above the screen with the screened interval zone water, and to minimize resuspension of solids collected at the bottom of the well or in the surrounding formation within the screened interval.

9. New polyethylene tubing shall be used for each sample when using non-dedicated sampling equipment. Prepare the pump and tubing for insertion into the well. Lower the pump intake down into the well casing. Connect the flow-though cell in-line with the pump effluent tubing.

10. Generally, the pump intake should be placed in the mid-point of the screened interval. This provides consistency between sampling rounds. However, if the geology of the screened interval consists of heterogeneous materials with layers of contrasting hydraulic conductivity, the pump intake should be positioned adjacent to the zone of highest hydraulic conductivity (as determined via review of the existing site hydrogeologic conditions/well completion logs). Also, the sampling plan should be consulted to determine if particular zones (e.g., known zones of contamination) are targeted for sampling per DQOs).

11. To achieve low-flow purging conditions, the purge rate should generally not exceed 0.5 L/min. Adjust the pump control to stabilize the flow rate, and therefore minimize drawdown (less than 0.3 foot during purging activities). The water level in the well should be measured throughout the purging process to monitor drawdown. Flow rate can be measured from the discharge tube using a volumetric measuring device (e.g., a graduated cylinder) and a stop watch (Note: determine flow rate by measuring volume in 0.5-minute or 1-minute increments.)

12. Record water level measurements, and field parameters including pH, temperature, specific conductivity, oxidation reduction potential (ORP), DO, turbidity, and flow rate every three to five minutes during the purging process. Record all measurements and observations in the log book or on a Groundwater Purging and Sampling Form (Attachment 1). Purging shall continue until the field parameters have stabilized. Parameters are considered stable when three consecutive readings are within the limits of the criteria defined in Table 5.1 and/or in accordance with the site-specific sampling plan. Turbidity ideally should stabilize below 10 NTU prior to sample collection, particularly if groundwater samples are to be collected for metals or PCB analyses.

TABLE 5.1 Stabilization of Water Quality Indicator Parameters

Parameter	Units	Stabilization Criteria
Water Level	Feet/meters	< 0.3 foot (< 0.1 meter)
Temperature	°F/°C	± 3 percent, or ±1.8 degrees Fahrenheit (°F) /±1 degree Celsius (°C)
pH	(n/a)	± 10 percent, or ±0.1 standards units (SU)
Specific Conductivity	µm/cm	±3 percent (microsiemens per centimeter, or µm/cm)
ORP	mV	±10 millivolts (mV)
Dissolved Oxygen	mg/L	±10 percent, or 0.2 milligram per liter (mg/L) - whichever is greater
Turbidity	NTU	± 1 Nephelometric Turbidity Unit (NTU) (±10 percent for turbidity if greater than 10 NTU)
Flow Rate	L/min	0.1 to 0.5 Liters per minute (L/min) (< 1 L/min), Specific flow rates and sampling rates to be identified in the sampling plan if project/contract required.

13. In low recharge aquifers, the following steps shall be followed:
    (1) If the initial water level is less than 10 feet above the top of the well screen, then purge the well until dry and allow sufficient recharge to collect samples.
    (2) If the initial water level in the well is greater than 10 feet above the top of the screen, then care shall be taken to prevent the dewatering of the screened interval during purging of the well.
        (2a) Continue purging until the water level is between 1 foot (0.3 meter) and 5 feet (1.5 meters) above the top of the screened interval.
        (2b) Allow the well to recharge, then continue purging until at least one full initial well volume has been purged.
    (3) Record all data, measurements, and observations in the log book.

14. After field parameters have stabilized, disconnect the flow through cell, and collect groundwater samples directly from the discharge tubing into an appropriate sample container. If using a peristaltic pump to collect VOC samples, refer to item 16 of this SOP for the correct procedure for sampling VOCs with a peristaltic pump. If an in-line, flow-through cell is used to continuously monitor indicator parameters, it should be disconnected or bypassed during sample collection. During sample collection, maintain the pump rate at the same rate used during purging (unless specified in the sampling plan). The pump rate used during sample collection may need to be lowered to minimize aeration, bubble formation, or turbulent flow of water into sample bottles, or to prevent sample preservatives from being washed out of the sample container.

15. Groundwater sampling (including the collection of all required quality assurance/quality control samples specified in the sampling plan) shall be performed immediately upon completion of purging (unless time for recharge is required for low-recharge wells) using the same equipment used for purging. Sampling should occur in a progression from the least to the most contaminated well, if this is known. Generally, volatile (e.g., solvents and fuel constituents) and gas sensitive (e.g., Fe^{2+}, CH_4, H_2S/HS^-, and alkalinity) analytes should be sampled first. The sequence in which samples for most inorganic parameters are collected is immaterial unless filtered (dissolved) samples are required. Filtered samples should be collected last and in-line filters should be used. After all unfiltered samples have been collected, a 0.45 micron (µm) in-line filter shall be inserted in the discharge line for collection of filtered samples, as required.

16. VOC samples should not be collected directly from the discharge end of a peristaltic pump. After field parameters have stabilized, and all other samples have been collected as required, stop the pump and simultaneously pinch the discharge end of the tubing shut. Disconnect the flow-through cell. Remove the tubing from the well and fill the VOC sample containers from the influent end of the sample tubing, (the end of the tubing that was located down-well during purging activities). The flow rate when filling sample vials may be controlled by setting the peristaltic pump in reverse.

17. Place all samples in a cooler with ice or ice packs to comply with project, laboratory, and/or regulatory requirements.

18. After sampling activities have been completed, remove the portable pump assembly from the well, if used, and decontaminate all non-disposable components. Replace the well plug. Secure the well plug and well cover. Clean up the work area: containerize and/or dispose of purge water as required by the site-specific sampling plan, and dispose of tubing and all other disposable sampling equipment as investigation derived waste (IDW) after each use as described in the site-specific sampling plan.

6.0 Restrictions/Limitations

Only grounded electrical devices should be used for low-flow sampling activities. If a gasoline-powered electrical source is used, place portable power sources (e.g., generators) 50 feet (15 meters) or farther from the wellhead to prevent potential contamination of samples. Additionally, it should be clearly noted in the field notes or on the Groundwater Sampling Log (Attachment 1) if a well has been pumped dry and allowed to recharge prior to sample collection, as low-flow sampling data is no longer applicable.

7.0 References

ASTM D6452-99(2012)e1, Standard Guide for Purging Methods for Wells Used for Groundwater Quality Investigations, ASTM International, West Conshohocken, PA, 2012.

ASTM D4448-01(2013), Standard Guide for Sampling Ground-Water Monitoring Wells, ASTM International, West Conshohocken, PA, 2013.

Puls, R.W. and M.J. Barcelona. April 1996. *Low-Flow (Minimal Drawdown) Ground Water Sampling Procedures*. U.S. EPA, Ground Water Issue, Publication Number EPA/540/S-95/504.

U. S. Environmental Protection Agency. May 2002. *Groundwater Sampling Guidelines for Superfund and RCRA Project Managers.* Ground Water Forum Issue Paper, EPA 542-S-02-001, OSWER, Technology Innovative Office, Washington, D.C.

Low- Stress (Low-Flow) Groundwater Sampling

SOP 1-12
Revision: 2
Date: February 2015

Attachment 1

EXAMPLE LOW-FLOW GROUNDWATER SAMPLING PURGING DATA SHEET

Site Name: _____ **Date:** _____ **OVM:** **FID** ☐ **PID** ☐ **In Casing (ppm): (Initial)** _____ **(Vented to)** _____

Well ID: _____ **Purging/Sampling Device:** _____

Initial Static Water Level (feet btoc): _____ **Analytical Parameters:** _____

Final Water Level (feet btoc): _____ **QC Samples Collected:** _____

Purge Start Time: _____ **Sample Number:** _____

Sample Time: _____ **Samplers' Signatures:** _____

Time	Water Level (ft btoc)	Temperature (ºC)	pH (SU)	Conductivity (µs/cm)	Dissolved Oxygen (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (mL/min)	Comments (e.g., depth of pump intake, screened interval)

Parameter	Units	Stabilization Criterion
Water Level	Feet/meters	< 0.3 foot (< 0.1 meter)
Temperature	°F/°C	± 3 percent, or ±1.8 degrees Fahrenheit (°F) /±1 degree Celsius (°C)
pH	(n/a)	± 10 percent, or ±0.1 standards units (SU)
Specific Conductivity	µm/cm	±3 percent (microsiemens per centimeter, or µm/cm)
ORP	mV	±10 millivolts (mV)
Dissolved Oxygen	mg/L	±10 percent, or 0.2 milligram per liter (mg/L) - whichever is greater
Turbidity	NTU	± 1 Nephelometric Turbidity Unit (NTU) (±10 percent for turbidity if greater than 10 NTU)
Flow Rate	L/min	0.1 to 0.5 Liters per minute (L/min) (< 1 L/min)

Drum Sampling	SOP 1-13 Revision: 2 Date: February 2015
Approved: Signature	**Technical Review:** Dave Sembrot/Shawn Oliveria

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define requirements for drum sampling at hazardous waste sites for disposal, bulking, recycling, segregation, and classification purposes.

2.0 Background

2.1 Definitions

Drum- A portable container, in which material is stored, transported, treated, disposed, or otherwise handled. The most frequent type of containers sampled by field investigators for chemical analysis and/or physical testing are 55 gallon drums.

Hazardous waste – Before a material can be classified as a hazardous waste, it must first be a *solid waste.* Hazardous wastes are regulated by 40 Code of Federal Regulation (CFR) Parts 260-268. S*olid waste* is any discarded material that is not excluded under §261.4(a) or that is not excluded by a variance granted under CFR §§260.30 and 260.31 or that is not excluded by a non-waste determination under §§260.30 and 260.34

Photoionization Detector (PID) - A portable, hand-held instrument that measures the concentration of gaseous organic compounds through the photoionization of organic vapors.

4 Gas Multi-Gas Detector – Real time gas detection systems capable of measuring for LEL, H2S, and %O2.

Bung wrench – A common tool for manually opening drums. Bung wrenches are usually constructed of non-sparking metal alloy (e.g. brass, bronze/manganese, aluminum, etc.) formulated to reduce the likelihood of sparks.

Drum deheader - A tool for manually opening a drum if a bung is not removable with a bung wrench. Drum deheaders are constructed of forged steel with an alloy steel blade and designed to cut off the lid of a drum by means of a scissor-like cutting action. This tool can only be used for closed head drums.

Backhoe spike – Tool used to open drums remotely for sampling. A backhoe spike is made of non-sparking metal that is attached or welded to a backhoe bucket. It is used mostly used in large-scale operations.

Pneumatic devices – A pneumatic bung remover consists of a compressed air supply that is controlled by a two-stage regulator. This device has a high pressure air line of desired length which delivers compressed air to a pneumatic drill, which is adapted to turn the bung fitting selected to fit the bung to be removed. The pneumatic drill has an adjustable bracketing system that allows the drill to be positioned and aligned over the drum. The adjustable bracketing system must be removed prior to sampling the drum. These tools require the drum to be upright and relatively level. Bungs that are rusted shut cannot be removed with this device.

Hydraulic Drum Opener – Tool used to open drums remotely for sampling by hydraulic pressure.

Glass drum thief sampler – Cost effective, quick and disposable tool used to sample drums. Glass drum thieves are typically 6mm to 16mm inner diameter (I.D) and 48 inches long.

COLIWASA sampler - A composite liquid waste sampler (COLIWASA) is used to collect a multiphase waste sample from the full depth of a drum and maintains the sample in a transfer tube until delivery to the sample bottle. COLIWASA samplers usually consist of a 152 cm by 4 cm I.D. section of tubing with a neoprene stopper at one end attached by a rod running the length of the tube to a locking mechanism at the other end.

Core device – Tool used to sample drum solids. This sampler consists of a series of extensions, a T-handle, and the coring device.

2.2 Associated Procedures

- SOP 1-2, *Sample Custody*
- SOP 1-10, *Field Measurement of Organic Vapors*
- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-2, *Photographic Documentation of Field Activities*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.3 Discussion

Drum sampling is necessary to characterize or categorize the contents of drums to obtain a quick, preliminary assessment of the types and layers of materials contained in them. The data can be used to make decisions regarding potential chemicals or contaminants present and the types of waste or material hazard characteristics of each drum. This information is then utilized to determine drum staging or restaging, bulking, transferring or compositing of the drum contents.

The Site manager shall ensure that appropriate H&S planning is performed prior to executing any planned activity of this type. That planning shall include a thorough hazard analysis along with atmospheric monitoring, personnel training, PPE, and emergency response requirements.

Prior to performing activities of this type, CDM Smith employees shall ensure that they have read and understood the applicable H&S planning and procedures related to the planned activity..

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that field personnel are trained in the use of this and related SOPs and the required equipment. The site manager is responsible for ensuring that all necessary health, safety, and security hazards are identified and addressed.

Field Team Leader - The field team leader (FTL) is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any other SOPs pertaining to specific media sampling. The FTL must also ensure that the quantity and location of drum samples collected meet the requirements of the site-specific plans. The FTL must ensure that all drum sampling logs are properly and completely filled out at the time of sampling. Each sampling log should detail the number, thickness and type of physical layers present in the drums and that all drum sampling log labels clearly identify and describe which samples belong to which layers of each drum sampled.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

All or part of the equipment listed under the “as needed” category may be required at any specific site, depending on the plan(s) for that site.

- Site-specific plans
- Field logbook
- Site related MSDSs (if available)
- Indelible black ink pens and markers
- Labels and appropriate forms/documentation for sample shipment
- Chain of custody records
- Drum overpacks
- Chemical reference guides
- Kimwipe or paper towels
- Clean plastic sheeting
- Absorbent material for spills

- Appropriate sample containers (e.g. wide mouth amber glass jars with Teflon cap liner, approximately 500 mL volume)
- Insulated cooler and waterproof sealing tape
- Ice bags or "blue ice"
- Any personal protective equipment specified in the site-specific health and safety plan
- Drum opening devices
- Appropriate decontamination equipment and supplies
- Plastic zip-top bags
- Tap and deionized water
- Water spray bottle
- Appropriate photographic equipment and supplies
- Clear waterproof tape

As needed:

- Global Positioning System (GPS) unit
- Spare parts for all equipment
- Tape measure

5.0 Procedures

5.1 Preparation

The following steps shall be taken when preparing for sampling drums:

1. Review site-specific health and safety plan and project plans before initiating sampling activity. Air monitoring requirements, and action levels should be identified and implemented. Appropriate PPE and various training requirements such as HAZWOPER and/or respiratory protection shall be in place

2. Determine the extent of the sampling effort, sampling methods to be employed, and the types and amounts of equipment and supplies needed.

3. Assess potential or likely materials contained in the drums to be sampled (i.e. visible labels, container material, construction, size and type). Assess the physical state of the drums and its current staged position to determine if any hazards may exist and what safety precautions need to be taken prior to approaching the drums.

4. Don the appropriate personal protective clothing as dictated by the site-specific health and safety plan. Decontaminate or preclean equipment, and ensure it is in working order.

5. Identify and mark all drums to be restaged in preparation for sampling and which drums will be sampled in place.

5.2 Drum Excavation (if required)

Note: If it is presumed that buried drums are on-site, then geophysical investigation techniques should be used to approximate the location and depth of the drums prior to excavation or digging activities.

1. Drum identification and inventory should be conducted during excavation and recorded in the sampling logbook (e.g. location, date of removal, drum ID number, overpack status, etc.) Any information regarding buried drums should also be taken into consideration prior to excavation.

2. Excavation of drums must be accomplished by a qualified conventional heavy equipment operator.

3. Excavate soil around the drum with non-sparking hand tools.

4. Perform a visual inspection to determine the condition of the drums.

Warning: Ensure that all excavation areas have been cleared of underground or overhead utility lines.

5.3 Drum Inspection and Staging

Note: All personnel should assume that unmarked drums contain hazardous materials until their contents have been identified.

1. Drums should be inspected for the following:
    a. Drum condition: pressurization (bulging/dimples) corrosion, rust, punctures, bungs, crystallization round the drum opening, holes, stains, and leaking contents
    b. Symbols, words, or other markings on the drum indicating hazards (e.g. explosive, radioactive, toxic, flammable)
    c. Signs that the drum is under pressure
    d. Shock sensitivity
2. Ambient air monitoring should be done to determine presence of unsafe levels of volatile organics, explosives, or radioactive materials, such as with a photoionization detector (PID), 4 gas multi-meter, and radioactivity detection monitor as specified in the site-specific health and safety plan. Depending on site conditions, cyanide fumes, pH, and halogen vapors should also be screened.
3. If necessary, classify the drum(s) into categories such as: radioactive, leaking/deteriorating, bulging, lab packs, explosive/shock sensitive, and empty.
4. Use color coded tags, labels, or bands to identify the drum's category based on visual inspection.
5. Record the identification and description of each drum in the field logbook (e.g. condition, unusual markings, burial or storage location, and field monitoring information.
6. Stage drums in an isolated area due to potentially suspect radioactive, explosive, or shock-sensitive materials.
7. Physically separate drums by the following categories: containing liquids, solids, containing lab packs, and empty.
8. A qualified forklift operator should move the drums by a forklift equipped with drum grabbers or a barrel grappler to an approved staging area.

Note: Refer to state and federal regulations for specific inspection and staging area requirements.

5.4 Drum Opening

Note: The drum opening area should be physically separated from the drum removal and drum staging operations. (Only non-sparking tools should be used.)

5.4.1 Manual Drum Opening with a Bung Wrench (Appendix A – Figure 1)

1. Positions drums upright with the bung up, or, for drums with bungs on the side, laid on their sides with the bung plugs up.
2. The wrenching motion should be a slow, steady pull across the drum. If the length of the bung wrench handle provides inadequate leverage for unscrewing the plug, a "cheater bar" can be attached to the handle to improve leverage.

5.4.2 Manual Drum Opening with a Drum Deheader (Appendix A – Figure 2)

1. Position the cutting edge just inside the top chime and tighten the adjustment screw so that the deheader is held against the side of the drum.
2. Move the handle of the deheader up and down while sliding the deheader along the chime to enable the entire top to be cut off.
3. Make the initial cut very slowly to allow for gradual release of any built-up pressure.

4. Decontaminate the deheader after each drum use to avoid cross contamination and/or adverse chemical reactions from incompatible materials.

5.4.3 Remote Drum Opening with a Backhoe Spike/Beryllium Punch (Appendix A – Figure 3)

1. Place drums in rows with adequate aisle space to allow ease in backhoe maneuvering.
2. After the drum has been staged, punch a hole in the drum head or lid with the spike.
3. Decontaminate the spike after each drum is opened to prevent cross contamination and/or adverse chemical reactions from incompatible materials.

Note: Remotely operated drum opening tools are the safest available means of drum opening compared to manual methods of opening.

5.4.4 Remote Drum Opening with Hydraulic Devices (Appendix A – Figure 4)

1. Push the piercing device (non-sparking, metal point attached to a hydraulic line) using hydraulic pressure.
2. Attach the piercing device so that a hole for sampling can be made in either the side or head of the drum.
3. Use the piercer to establish a tight seal after penetrating the drum.

Note: Some metal piercers are hollow or tube-like, and therefore, can be left in place to serve as a permanent tap or sampling port.

5.4.5 Remote Drum Opening with Pneumatic Devices (Appendix A – Figure 5)

1. Set the container upright and relatively level.
2. Select a bung fitting to fit the bung to be removed.
3. Attach the adjusting bracketing system to the drum and align the pneumatic drill over the bung. This must be done prior to operating the drill.
4. Operator must step away from the drum to operate the equipment.
5. Once the bung has been loosened, remove the bracketing system prior to sampling the drum.

Note: This device cannot be used to remove rusted bungs. This device also does not permit the slow venting of the drum, therefore, appropriate precautions are necessary.

5.5 Liquid Drum Sampling

Note: Samples collected from drums are considered waste samples and as such, adding preservatives is not required due to the potential reaction of the sample with the preservative. Samples should, however, be cooled to 4^{o}C and protected from sunlight in order to minimize any potential reaction due to the light sensitivity of the sample.

After the drum has been opened, preliminary monitoring of headspace gases should be performed with a PID or 4 gas multi-meter. Also, radioactivity readings should be monitored using an approved detection device. The following techniques should be used in order to take a sample that represents the entire depth of the drum, to check the presence of sludge at the bottom of the drum, and to sample solids:

5.5.1 Glass Drum Thief Sampler (Appendix A – Figure 6)

1. Remove the cover from the sample container.

2. Insert glass tubing almost to the bottom of the drum or until the solid layer is encountered. **Note:** About one foot of tubing should extend above the drum. Lift the clear glass sampler to inspect the liquid for any signs of layering which may indicate a non-aqueous phase either floating or sinking within an aqueous layer. Be aware of any solid or semi-solid layers within the drum.

3. Allow the waste in the drum to reach its natural level in the tube.

4. Cap the top of the sampling tube with a tapered stopper or thumb, ensuring liquid does not come into contact with stopper.

5. Carefully remove the capped tube from the drum and insert the uncapped end into the appropriate sample container.

6. Release the stopper and allow the glass thief to drain until the container is approximately two-thirds full.

7. Remove tube from the sample container, break it into pieces and place the pieces in the drum.

8. Cap the sample container tightly and label it. Place the sample container into a cooler with ice.

9. Replace the bung or clean plastic sheeting over the drum.

10. Record all samples in the field logbook. Ensure every layer present in the drum is represented in separate sampling containers and that the thickness of each layer is noted so that volumes present in the drum can be estimated.

11. Relocate the samples into the decontamination area and package them in accordance with approved field plans.

12. Complete the chain of custody.

5.5.2 COLIWASA Sampler (Appendix A – Figure 7)

1. Open the sampler by placing a stopper rod handle in the T-position and pushing the rod down until the handle sits against the sampler's locking block.

2. Slowly lower the sampler into the liquid waste. Lower the sampler at a rate that permits the levels of the liquid inside and outside the sampler tube to be about the same.

Note: If the level of the liquid in the sample tube is lower than that outside the sampler, the sampling rate is too fast and will result in non-representative sample.

3. When the sampler stopper hits the bottom of the waste container, push the sampler tube downward against the stopper to close the sampler. Lock the sampler in the closed position by turning the T-handle until it is upright and one end rests tightly on the locking block.

4. Slowly withdraw the sampler from the waste container with one hand while wiping the sampler tube with a disposable cloth with the other hand. Inspect the liquid in the COLIWASA for any signs of layering which may indicate a non-aqueous phase either floating or sinking within an aqueous layer. Be aware of any solid or semi-solid layers within the drum.

5. Carefully discharge the sample into the appropriate sample container by slowly pulling the lower end of the T-handle away from the locking block while the lower end of the sampler is positioned in the sample container.
6. Cap the sample container tightly and label it. Place the sample into a container and place it into a cooler with ice.

7. Replace the bung or place clean plastic sheeting over the drum.

8. Record all samples in the field logbook. Ensure every layer present in the drum is represented in separate sampling containers and that the thickness of each layer is noted so that volumes present in the drum can be estimated.

9. Relocate the samples into the decontamination area and package them in accordance with approved field plans.

10. Complete the chain of custody.

5.6 Soil or Solid Drum Sampling

5.6.1 Coring Device

Note: When sampling drum solids, a composite sample should be taken from different areas within the drum.

1. Assemble the sampling equipment.

2. Remove the cover from the sample container.

3. Insert the sampling device to the bottom of the drum. The extensions and the "T" handle should extend above the drum.

4. Rotate the coring device to cut a core of material.

5. Slowly withdraw the sampling device so that as much sample material as possible is retained within it.

6. Transfer the sample to the appropriate sample container, and label it.

Note: A stainless steel spoon or scoop may be used.

7. Replace the bung or place clean plastic sheeting over the drum.

8. Record all sample in the field logbook.

9. Relocate the samples into the decontamination area and package them in accordance with approved field plans.

10. Complete the chain of custody.

6.0 Restrictions/Limitations

Drum sampling is one of the most hazardous site activities. The use of remote drum opening and sampling equipment whenever feasible is highly recommended in regards to health and safety.

Glass drum thief samplers are cost effective and efficient tools for quickly obtaining representative samples within drums containing liquid materials. When solids or semi-solids are encountered the glass drum thief sampler may shatter causing a the potential for hand injuries. Caution must be exercised when utilizing these samplers within drums with multi-phase layers in drums. Consideration for alternative or multiple sampling devices must be given to sample planning to account for liquid, semi-solid and solid layers may be present in drums.

Air monitoring action levels must be defined and understood prior to conducting any site activity

When using the COLIWASA sampler, major drawbacks make it an impractical disposable item such as difficulty in field decontamination in the field and relatively high costs in relation to alternative procedures. It still has applications, however, especially in instances where a true representation of a multiphase waste is absolutely necessary.

7.0 References

40 Code of Federal Regulations Parts 260-268.

ASTM D5680-14 – Standard Practice for Sampling Unconsolidated Solids in Drums or Similar Containers

ASTM D6063-11 – Guide for Sampling of Drums and Similar Containers by Field Personnel

U.S. Environmental Protection Agency. November 1994. *Drum Sampling.* SOP#: 2009.

U.S. Environmental Protection Agency. January 1986. *Drum Handling Practices at Hazardous Waste Sites*, EPA-600/2-86-013.

U.S. Environmental Protection Agency, Region IV. November 2007. Science and Ecosystem Support Division Operating Procedure. *Waste Sampling,* SESDPROC-302-R1.

Martin Marietta Energy Systems, Inc. 1998. *Environmental Surveillance Procedures Quality Control Program*, ESH/Sub/87-21706/1.

Hackman et al. 2001. *Hazardous Waste Operations and Emergency Response Manual and Desk Reference.*

Appendix A

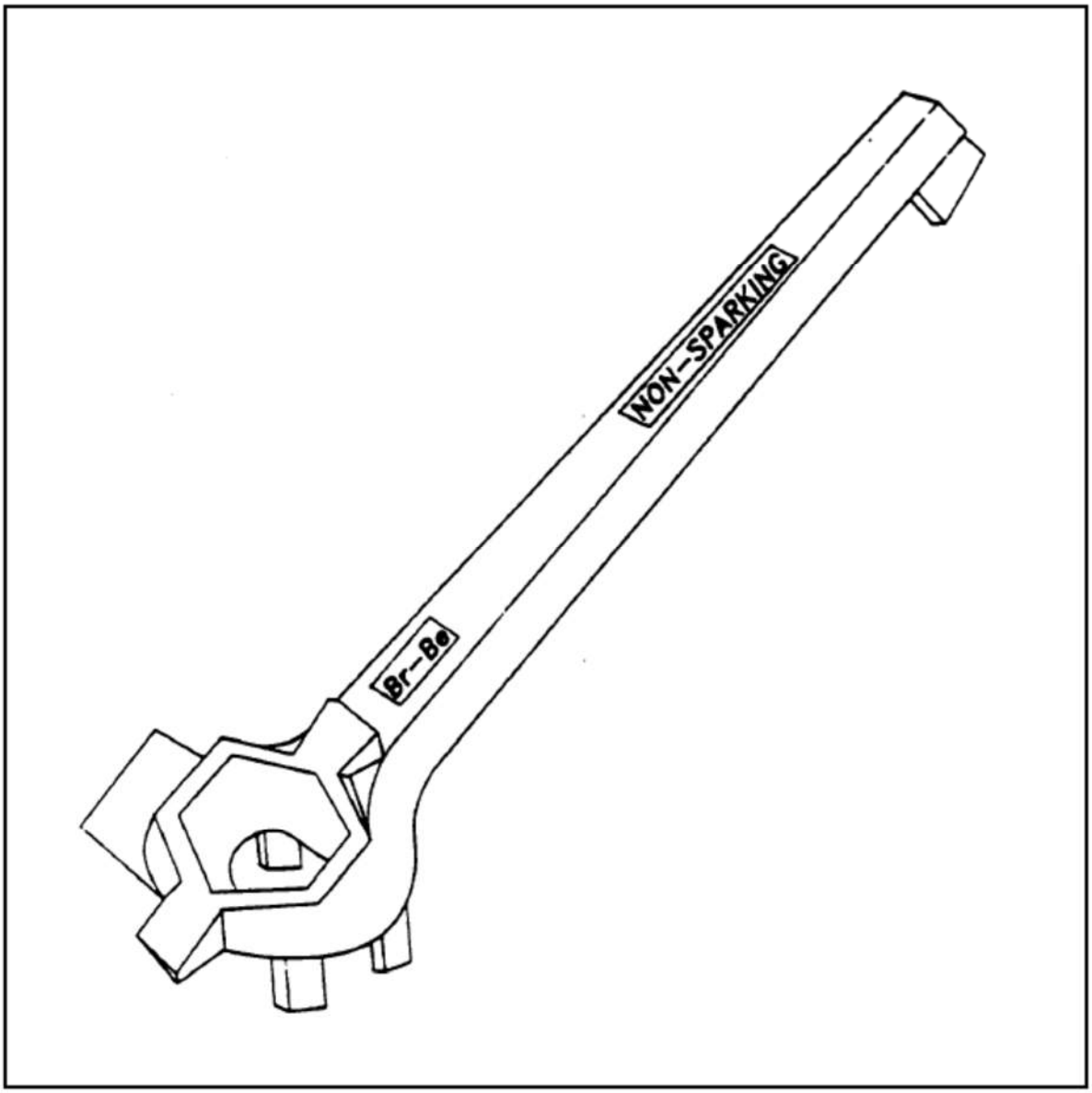


Figure 1. Universal Bung Wrench

Appendix A

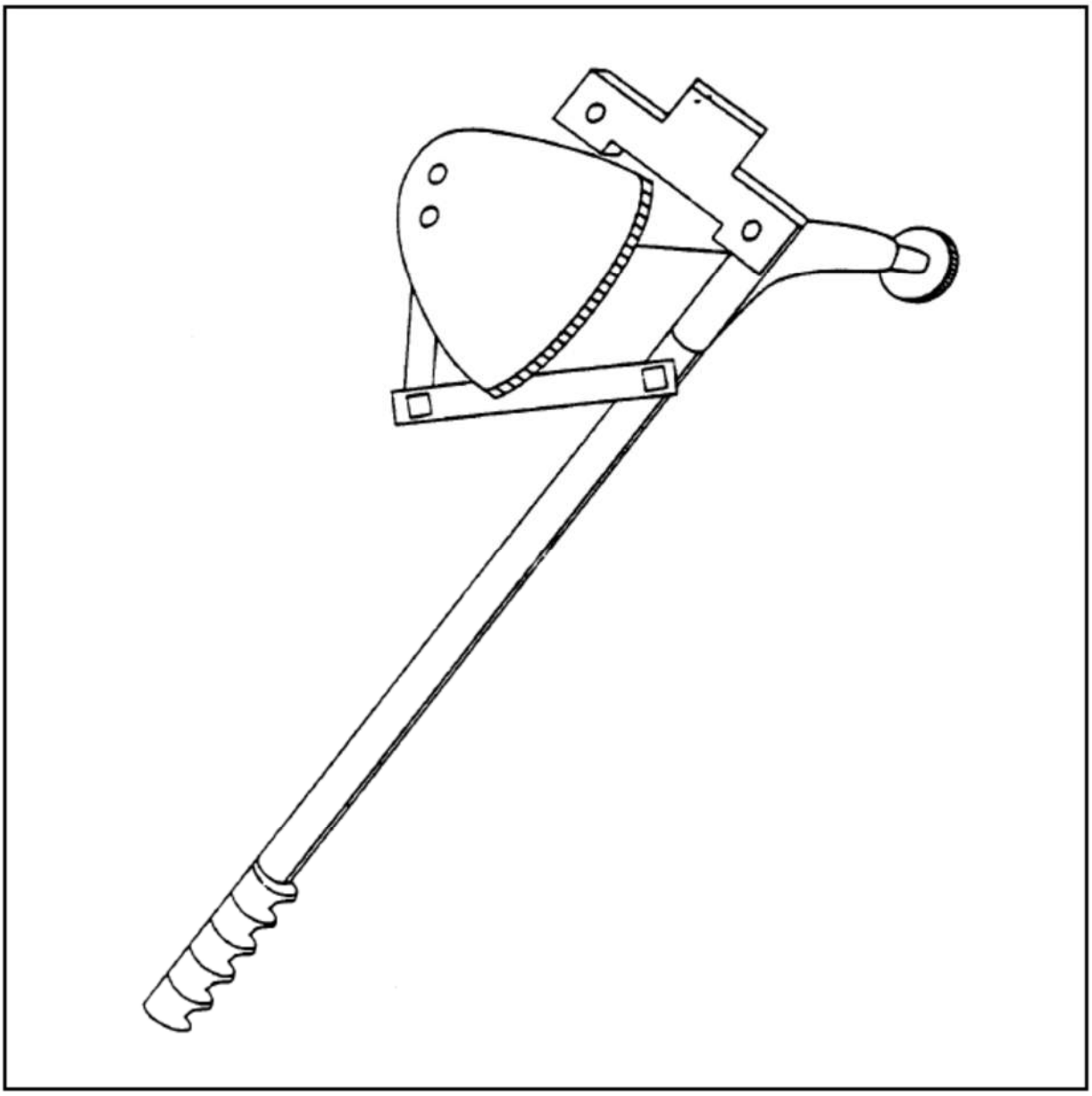


Figure 2. Drum Deheader

Appendix A

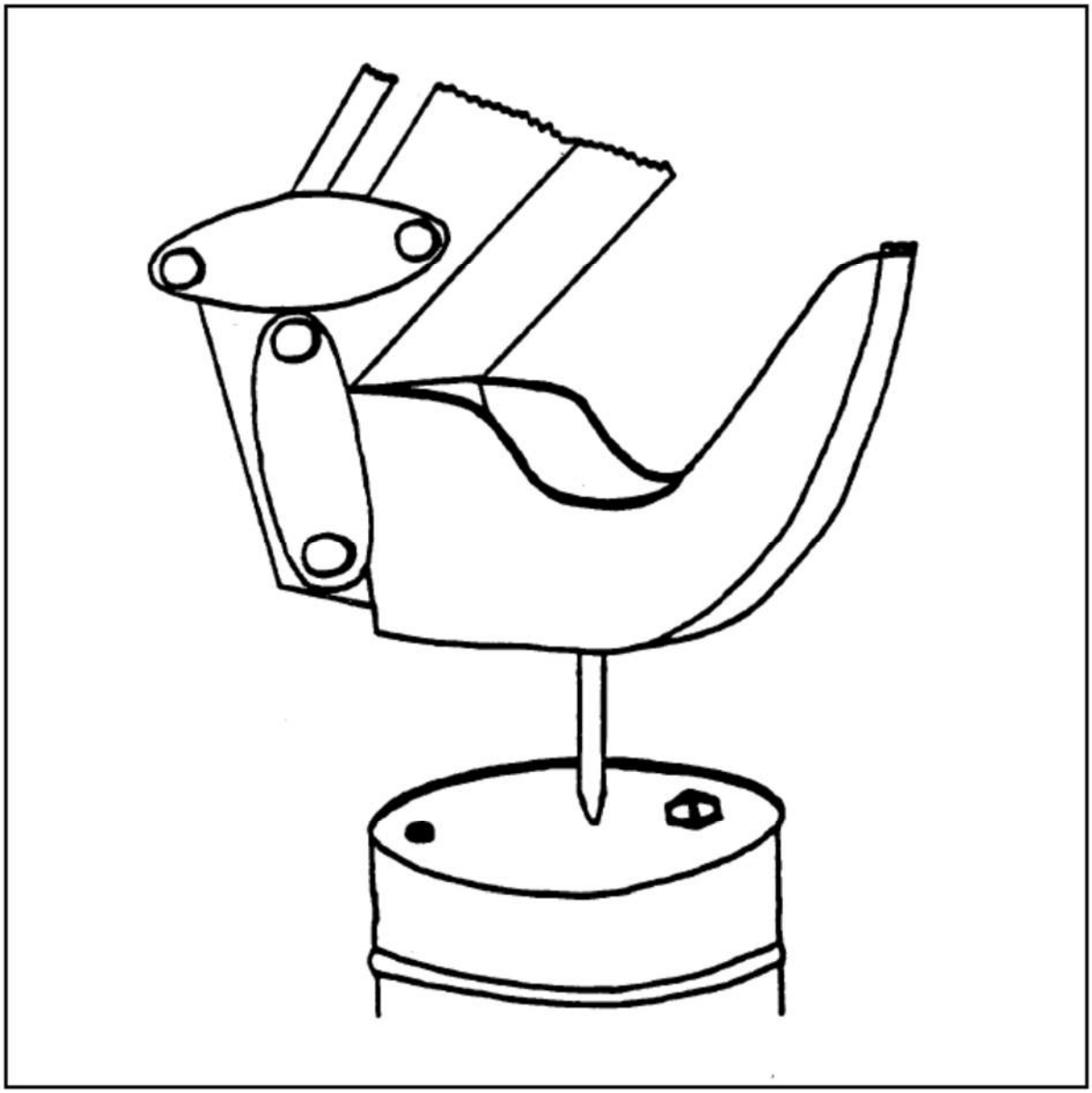


Figure 3. Backhoe Spike

Appendix A

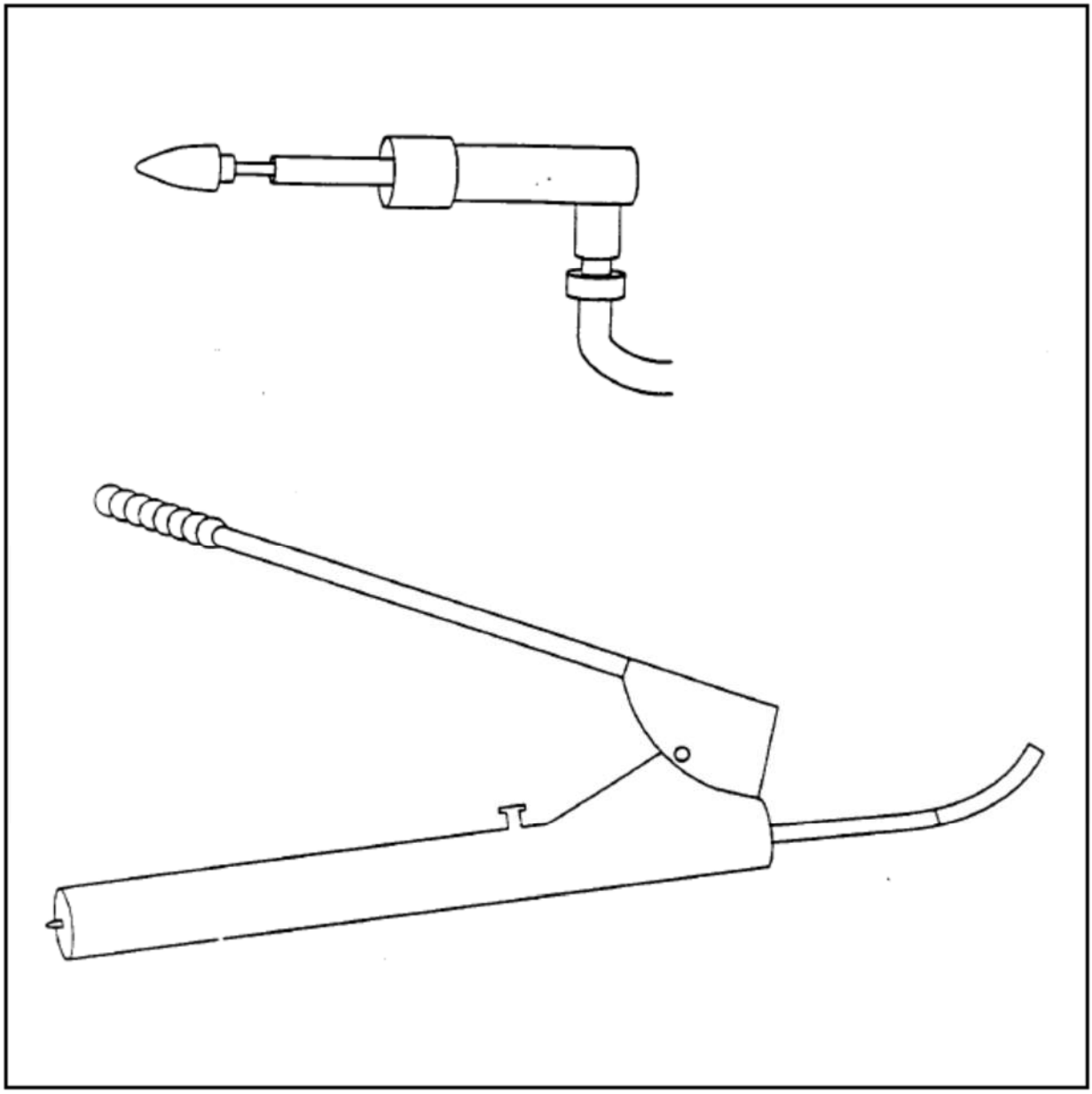


Figure 4. Hydraulic Drum Opener

Appendix A

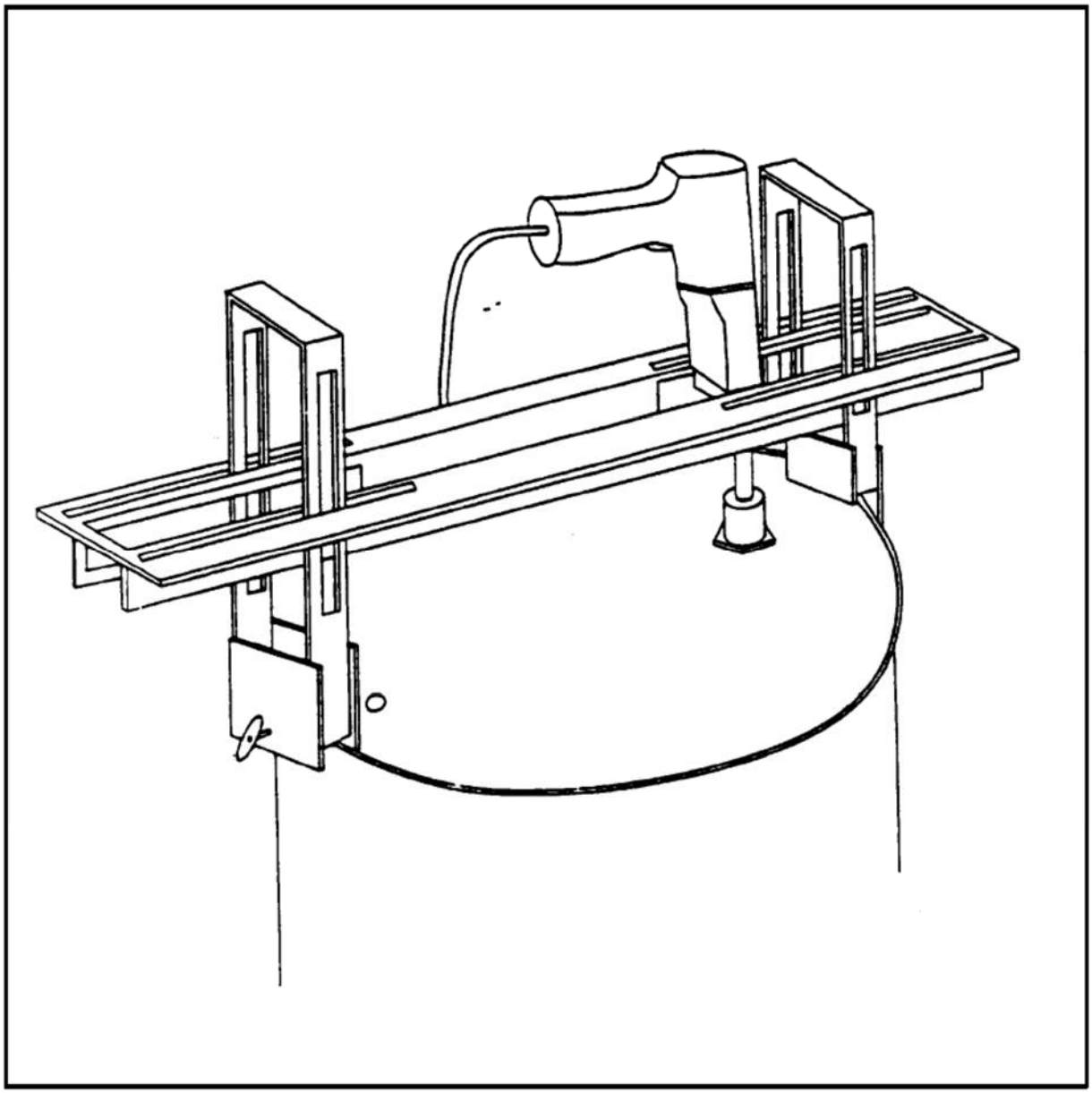


Figure 5. Pneumatic Bung Remover

Appendix A

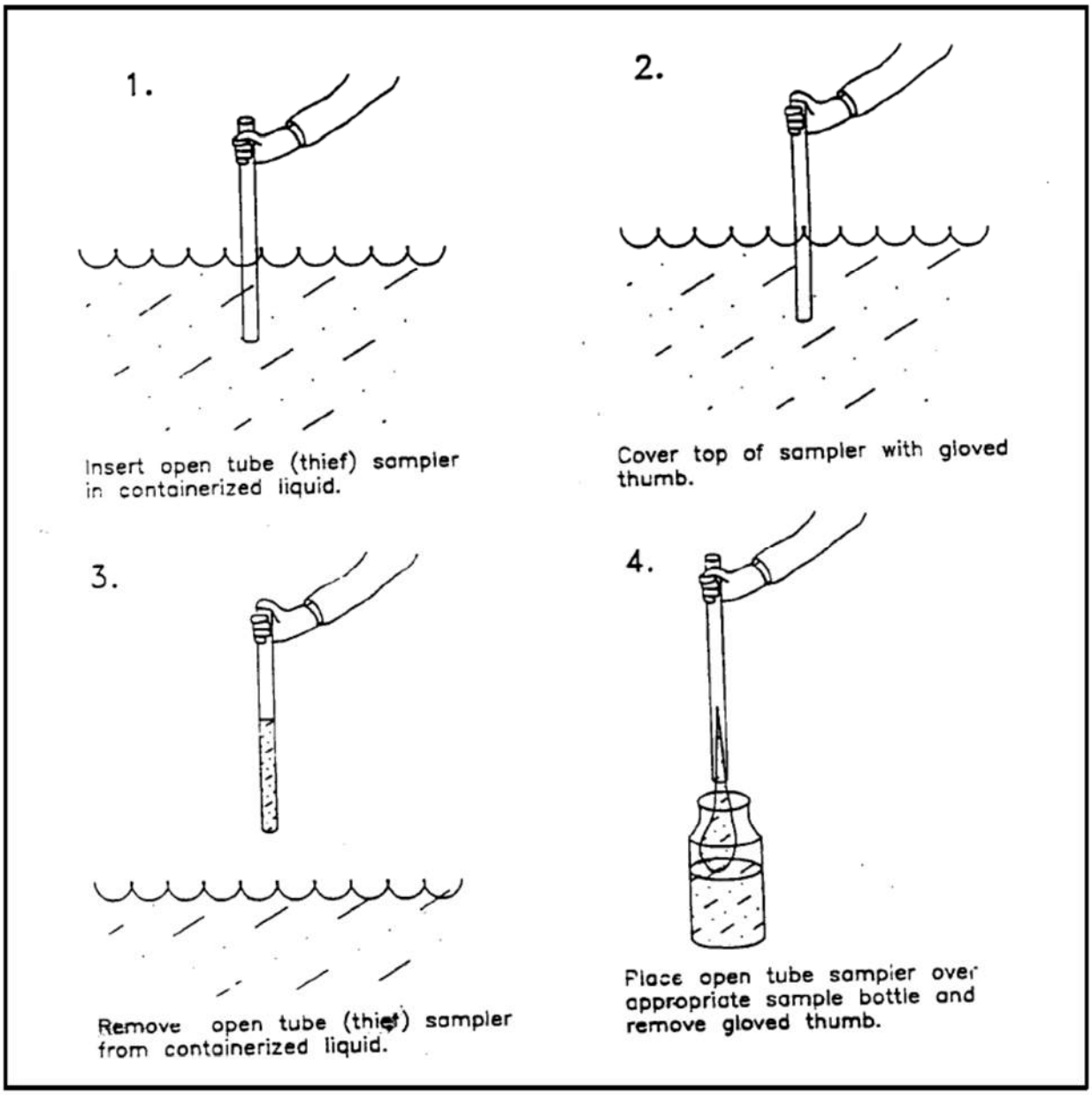


Figure 6. Glass Thief

Appendix A

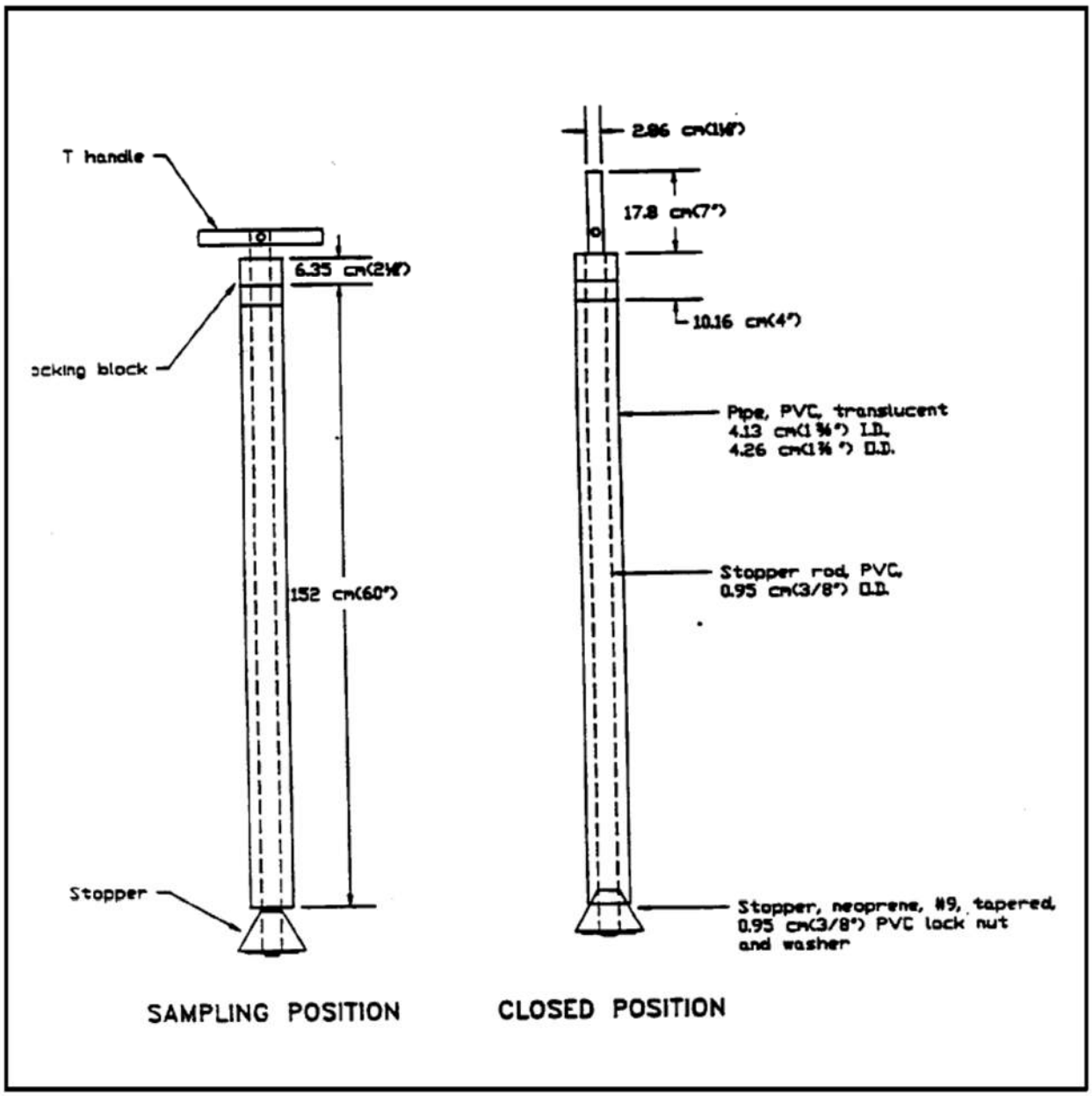


Figure 7. COLIWASA

Lagoon Sampling	SOP 1-14 Revision: 2 Date: February 2015

Approved: Signature **Technical Review:** Murray Wade

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define requirements for collection of surface water samples and containment of sediment/sludge samples in lagoons. This procedure is also applicable for sampling rivers, lakes, ponds, streams, embayments, mudflats, and surface impoundments.

2.0 Background

2.1 Definitions

Surface Water - Water that flows over or rests on the land and is open to the atmosphere. This includes lagoons, ditches, streams, rivers, lakes, pools, ponds, and basins.

Shallow Surface Water - Water within 1 to 2 feet of the surface of a body of water.

Deep Surface Water - Water deeper than 2 feet of the surface of a body of water.

Grab Sample - A discrete portion or aliquot taken from a specific location at a given point in time.

Simple Composite - Two or more subsamples taken from a specific media and site at a specific point in time. The subsamples are collected and mixed. Then a single average sample is taken from the mixture.

Temporal Composite - Two or more subsamples taken from a specific media and site over a period of time. The subsamples are collected and mixed. Then a single average sample is taken from the mixture.

Churn Splitter - Large vessel for compositing subsamples. Includes a mechanism to agitate the water to keep solids suspended.

Sediment - Geologic and/or organic material underlying a body of water. The material has been transported by a fluid and deposited within the boundaries of the body of water.

Sludge - Materials ranging in type from dewatered solids to high viscosity liquids. The material may exist suspended throughout the water or settled from the water as all or part of the sediment.

2.2 Associated Procedures

- SOP 1-2, *Sample Custody*
- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-2, *Photographic Documentation of Field Activities*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.3 Discussion

Surface water and sediment/sludge samples are collected to determine the type(s) and level(s) of contamination in a particular surface water body such as a lagoon and/or its biological disposition. Sediment/sludge samples will provide a more historical account of contamination than will water samples because of the nature of the matrix.

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that field personnel are trained in the use of this and related SOPs and the required equipment.

Field Team Leader - The field team leader (FTL) is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any other SOPs pertaining to specific media sampling. The FTL must also ensure that the quantity and location of sediment/sludge samples collected meet the requirements of the site-specific plans.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

All or part of the equipment listed under the "as needed" category may be required at any specific site, depending on the plan(s) for that site.

- Site-specific plans
- Field logbook
- Indelible black ink pens and markers
- Labels and appropriate forms/documentation for sample shipment
- Appropriate sample containers
- Insulated cooler and waterproof sealing tape
- Ice bags or "blue ice"
- Plastic zip-top bags
- Clear waterproof tape
- Personal protective clothing and equipment
- Latex or appropriate gloves
- Rubber boots and/or rubberized waders
- Life jacket
- Teflon or stainless steel mixing bowls or trays
- Aluminum foil
- Kimwipe or paper towels
- ½- to ¾-inch (12- to 19-mm) braided nylon line or Teflon-coated wire rope
- Clean plastic sheeting
- Tap and deionized water
- Water spray bottle
- Appropriate photographic equipment and supplies
- Appropriate decontamination equipment and supplies
- Eckman grab sampler for depositional area (primarily stream) sediment sampling
- Ponar sampler for lake sampling
- Stainless steel or Teflon® spoons, spatulas, or scoops

As needed:

- Global Positioning System (GPS) unit
- Hand or gravity corer with extensions or stainless steel hand auger
- Core liners of Teflon, stainless steel, brass, aluminum, or polybutyrate, as specified in the site-specific plan(s)
- Stainless steel push tubes
- Dredge with 15- to 20-foot (4.5- to 6.0-meter) sampling pole (hollow) and insert (e.g., Peterson, Eckman, Ponar)
- Pond sampler with 1-liter (L) beaker (preferably Teflon®), clamp, and heavy-duty telescoping pole
- Weighted bottle sampler, 1-L capacity (preferably Teflon) and handle; see USGS Open File Report 2005-1087 for selection of sampler; a Kemmerer or Van Dorn sampler may be used if Teflon is not required
- Motorized coring device
- Boat with depth finder for deep water or inaccessible shorelines
- Any personal protective equipment specified in the site-specific health and safety plan
- Spare parts for all equipment
- Tape measure
- Churn splitter
- Peristaltic pump or suitable replacement
- Temperature, pH, and conductivity meter(s), dissolved oxygen meter, redox potential meter (as required by project plan)

5.0 Procedures

5.1 Preparation for Sampling Surface Water

The following steps should be taken when preparing for sampling surface water:

1. Review site-specific health and safety plan and project plans before initiating sampling activity.
2. Don the appropriate personal protective clothing as dictated by the site-specific health and safety plan.

3. Select wadeable lagoon sampling locations that exhibit cross-sectional homogeneity and are well-mixed. Avoid areas where the channel is constricted or bends where scouring may have occurred.

Note: For lake samples, the investigator should consider the lake stratification caused by seasonal temperature differences. If possible, select a location that can be described precisely, such as xx feet upstream of xx bridge. Use caution when wading in lagoons more than 1 to 2 feet deep. Flowing water can be a safety hazard.

4. Prepare sampling site by laying out clean plastic sheeting on the ground or any flat, level surface near the sampling area. Then place equipment to be used on the plastic sheeting.

5. Make field measurements as required by the project plans in physical, chemical, and biological characteristics of the water (e.g., discharge, gage height, temperature, dissolved oxygen, conductivity, pH).

6. The samples should be collected from areas of least to greatest contamination (when known). When collecting several samples in 1 day, always collect from downstream to upstream.

7. The sampler should be facing upstream when sampling, both for proper sample collection and for safety (i.e. ability to observe floating objects).

8. Document the sampling events, recording all information in the designated field logbook and take photographs if required or if possible. Document any and all deviations from this SOP and include rationale for changes.

9. The collection points should be located on a site map and described in the field logbook. Use GPS to determine location if required or if possible.

10. Label each sample container with the appropriate information. Secure the label by covering it with a piece of waterproof clear tape.

11. Decontaminate reusable sampling equipment after sample collection according to SOP 4-5.

12. Processes for verifying depth of samples must be included in site-specific project plans.

13. Check that a trip blank/temperature blank, when necessary, is included in the chilled cooler. Quality assurance/quality control sample requirements vary from project to project. Consult the project-specific work plan for quality requirements.

5.2 Shallow Surface Water Sample Collection for Wadeable Lagoons

5.2.1 Method for Collecting Samples for Volatile Organic Compound Analysis

All volatile organic compound (VOC) samples should be discrete samples. The following steps must be taken when collecting shallow surface water VOC samples:

If the volatile organic analysis (VOA) vials do not require a preservative:

1. Approach the sample location from downstream; do not enter the sample area. Slowly submerge VOA vials completely into an area of gently flowing water and fill. Do not disturb bottom sediments. The open end of the vials should be pointed upstream.

Note: When collecting samples for VOC analysis, avoid collecting from a surface water point where water is cascading and aerating.

2. Cap the VOA vial while it is underwater. Be sure to dislodge all air bubbles from the cap before sealing the vial.

3. Turn the capped vial upside down and check for air bubbles. Tap the bottom of the vials to dislodge any bubbles that may have formed around the cap or sides. Discard and resample if bubbles are present.

4. Proceed to Step 5 below.

If the VOA vials require a preservative [i.e. hydrochloric acid (HCl)]:

1. Collect a sufficient sample in a clean glass jar as in Steps 1 and 2 above for unpreserved vials. Specific sampling devices to be used must be specified in site-specific plans.

2. Decant the sample immediately into pre-preserved VOA vials. It is recommended that the amount of preservative be predetermined on a separate aliquot of sample that is subsequently discarded. Tip vials slightly while filling to reduce turbulence until nearly filled. Then straighten vial to vertical for final filling. Ensure that a meniscus is raised above the lip of the vial before capping.

3. Cap each vial once the meniscus has formed.

4. Turn the capped vial upside down and check for air bubbles. Tap the bottom of the vials to dislodge any bubbles that may have formed around the cap or sides. Discard and resample if bubbles are present.

5. Wipe the outside of sample vials with a Kimwipe or clean paper towel. Affix a completed sample label.

6. Place sample vial(s) in a zip-top plastic bag and seal the bag.

7. Immediately pack all samples into a chilled cooler.

8. Check that a trip blank/temperature blank, when necessary, is included in the chilled cooler. Quality assurance/quality control requirements vary from project to project. Consult the project-specific work plan for quality requirements.

5.2.2 Method for Collecting Discrete Shallow Surface Water Samples for Nonvolatile Organic or Inorganic Compound Analysis

The following steps must be followed when collecting discrete shallow surface water samples for nonvolatile organic or inorganic compound analysis:

1. Directly dip the sample container, with the opening facing upstream, into the surface water and fill. If wading is necessary, approach the sample location from downstream; do not enter the actual sample area. Do not disturb underlying sediments.

2. Filter samples if required by the site-specific plan.

3. Add appropriate preservatives to the sample containers if required and check pH.

Note: Use a separate container when field testing pH, conductivity, temperature, etc. Do not insert pH paper or probe directly into sample container.

4. Cap the sample containers and wipe the outer surfaces of the sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.

5. Place sample container(s) in individual zip-top plastic bags, if possible, and seal the bags.

6. Immediately pack all samples into a chilled cooler.

5.2.3 Method for Collecting Simple Composite Shallow Surface Water Samples for Nonvolatile Organic or Inorganic Compound Analysis

If the QAPP requires the use of simple composite samples, then a sampler capable of collecting composite samples is required. For width and depth integrated (WDI) composite samples, a DH-48 or DH-81 sampler is recommended, but the QAPP may specify an alternative. The following steps must be followed when collecting simple composite shallow surface water samples for nonvolatile organic or inorganic compound analysis:

1. Record the gage height, if any, before and after sampling.

2. Select the number of width increments based on the requirements of the QAPP. Generally, small well mixed streams require few increments while large or poorly mixed streams require more increments.

3. For fewer than six width increments, subsample locations can be visually estimated. For more than five width increments, string a tape measure across the stream above the water surface to be able to accurately identify the subsample locations. Increments should be evenly spaced across the stream for equal width-integrated (EWI) sampling.

4. If depth-integrated sampling is required, collect a subsample at each width increment by submerging the sampler, orifice facing upstream, from the surface to near the bottom and back up to the surface again in an even steady motion. Do not disturb the sediment at the bottom. The sampler should be retrieved less than full. If the sampler is full, empty it and repeat the subsample collection.

5. If depth-integrated sampling is not required, submerge the sampler with the orifice facing upstream into the surface water and fill.

6. Empty the sampler into a churn splitter or temporary container for later splitting.

7. Repeat Steps 4 to 6 for each width increment.

8. If temporary containers were used, empty into churn splitter. Operate the churn splitter by moving the churn up and down in a steady motion fast enough to homogenize the sample without causing aeration. While the churn is in motion, fill the sample bottles from the tap on the churn.

9. Follow Steps 2 through 6 in Section 5.2.2.

5.2.4 Method for Collecting Temporal Composite Shallow Surface Water Samples for Nonvolatile Organic or Inorganic Compound Analysis

If the QAPP requires the use of temporal composite samples, this can be accomplished using a series of discrete samples collected by hand or an automated sampler, or using a series of simple composite samples. Refer to the preceding sections for collecting the subsamples. The compositing scheme can be time-based (e.g., once per hour for 4 hours) or time-discharge (or time gage height) based (e.g., once per hour until the gage height exceeds xx feet, then change to once per 15 minutes).

Because of the project-specific nature of temporal composite sampling, the specific requirements should be identified in the QAPP. The following are general steps to be followed to collect temporal composite samples:

1. Provide for a method of measuring discharge or gage height before, during, and after sample collection as required in the QAPP.

2. Select the number of time increments based on the requirements of the QAPP. If the time increments change based on a change in flow or water quality, specify the trigger, the new time increment, and any additional trigger to return to the previous increment.

3. Calculate the storage volume for the subsamples and provide a churn splitter of adequate size to contain the entire sample to be composited.
4. Collect the samples according to a method described in this SOP or alternate specified in the QAPP.
5. Provide for cold storage of subsamples, if possible. Do not process any subsamples by filtering or preserving unless specified in the QAPP.
6. Following collection of all subsamples, empty the containers into a churn splitter. If discrete data are required including laboratory or field analysis, retain a portion of the subsample.
7. Operate the churn splitter by moving the churn up and down in a steady motion fast enough to homogenize the sample without causing aeration. While the churn is in motion, fill the sample bottles from the tap on the churn.
8. Follow Steps 2 through 6 in Section 5.2.2.
9. Field parameters should be measured in the surface water at the time of collection. Some field parameters can be measured on the subsamples at the time of compositing, but the temperature and temperature-dependant parameters will not be representative.

5.3 Deep Surface Water Sample Collection

5.3.1 Method for Collecting Samples at Specified Depth Using a Weighted Bottle Sampler

The following steps must be followed when collecting surface water samples at specific depths using a weighted bottle sampler:

1. Lower the weighted bottle sampler to the depth specified in the site-specific plan.
2. Remove the stopper by pulling on the sampler line and allow the sampler to fill with water.
3. Release the sampler line to reseat the stopper and retrieve the sampler to the surface.
4. Wipe the weighted bottle sampler dry with a Kimwipe or clean paper towel.
5. Remove the stopper slowly. Fill the specified number of sample containers by slightly tipping the sampler against each sample bottle. Samples to be used for VOC analysis should be decanted directly from the sampler first into pre-preserved VOA vials. It is recommended that the amount of preservative be predetermined on a separate aliquot of sample that is subsequently discarded. Add appropriate preservatives to the other sample containers and check pH. Samples may be pooled in stainless steel, glass, or Teflon containers to obtain the necessary volumes. Filter samples if required. Collect sample in separate container for pH, conductivity, temperature, and other measurements if necessary.
6. Close each sample container with the Teflon-lined cap once it is filled. Check for air bubbles in the VOC sample containers. If bubbles are present, discard and resample.
7. Wipe the outside of the sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.
8. Place sample container(s), if possible, in individual zip-top plastic bags and seal the bags.
9. Immediately pack all samples into a chilled cooler.

5.3.2 Method for Deep Surface Water Sample Collection Using a Peristaltic Pump

The following steps must be followed when collecting deep surface water samples using a peristaltic pump:

1. Install clean medical-grade silicon or Teflon tubing on the pump head. Leave sufficient tubing on the discharge side for convenient dispensing of liquid directly into sample containers.

2. Select the appropriate length of Teflon intake tubing necessary to reach the specified sampling depth. Attach the intake sampling tube to the intake pump tube.

3. Lower the intake tube into the surface water at the specified sampling location to the specified depth; make sure the end of the intake tube does not touch underlying sediments.

4. Start the pump and allow at least three tubing volumes of liquid to flow through and rinse the system before collecting any samples. Do not immediately dispense the purged liquid back to the surface water body. Instead, collect the purged liquid and return it to the source after sample collection is complete.

5. Fill the specified number of sample containers directly from the discharge line. Filter samples if required by the site-specific plan. While filling, allow the liquid to flow gently down the inside of the sample bottle to minimize turbulence.

   For VOC samples, fill pre-preserved VOA vials and allow a meniscus to form above the top of the container before capping. It is recommended that the amount of preservative be predetermined on a separate aliquot of sample that is subsequently discarded. Check VOA vials to ensure that there are no air bubbles. Add appropriate preservatives to the other samples and check pH.

Note: Use a separate container when field-testing pH, conductivity, temperature, etc. Do not insert pH paper or probe directly into sample container.

6. Cap the sample container(s). Wipe the outside of sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.

7. Place sample container(s) in individual zip-top plastic bags and seal the bags.

8. Immediately pack all samples into a chilled cooler.

9. Drain the pump system, rinse it with deionized water, and wipe it dry. Replace all tubing with new tubing before sampling at another location. Place all used tubing in plastic bags to be discarded or decontaminated according to the site-specific plans.

5.4 Preparation for Sampling Sediment/Sludge

The following steps shall be taken when preparing for sampling sediment/sludge:

1. Review site-specific health and safety plan and project plans before initiating sampling activity.

2. Don the appropriate personal protective clothing as dictated by the site-specific health and safety plan.

3. Select lagoon sampling locations that exhibit cross-sectional homogeneity. Avoid areas where the channel is constricted or bends where scouring may have occurred. For lakes, collect sediment samples away from the shoreline.

4. Prepare sampling site by laying out clean plastic sheeting on the ground or any flat, level surface near the sampling area. Then place equipment to be used on the plastic sheeting.

5. If surface water is present at the sample location, make field measurements of physical, chemical, and biological characteristics of the water (e.g., temperature, dissolved oxygen, conductivity, pH), as dictated by the project-specific plans.

6. The samples shall be collected from areas of least to greatest contamination (when known). When collecting several samples in 1 day, always collect from downstream to upstream.

7. When sampling sediment and surface water from the same surface water body, collect surface water samples before sediment samples.

8. Document the sampling events, recording all information in the designated field logbook and take photographs (if required). Document any and all deviations from this SOP and include rationale for changes.

9. The collection points should be located on a site map and described in the field logbook. Use GPS if required or if possible.

10. Label each sample container with the appropriate information. Secure the label by covering it with a piece of waterproof clear tape.

11. Decontaminate reusable sampling equipment after sample collection according to SOP 4-5.

12. Processes for verifying depth of samples must be included in site-specific project plans.

13. Check that a trip blank/temperature blank, when necessary, is included in the chilled cooler. Quality assurance/quality control requirements vary from project to project. Consult the project-specific work plan for quality requirements.

5.5 Sediment/Sludge Sample Collection from Shallow Waters

5.5.1 Method for Collecting Samples for Volatile Organic Compound (VOC) Analysis

The following steps must be followed when collecting shallow water sediment/sludge VOC samples:

1. Use a decontaminated stainless steel or Teflon, long-handled scoop, corer, push tube, or dredge to collect the entire sample in one grab. If wading is necessary, approach the sample location from downstream. Do not enter the actual sample area.

2. Retrieve the sampling device and slowly decant off any liquid.

3. Immediately fill the specified sample container(s) with the solid. Use a clean stainless steel or Teflon spoon or spatula to completely fill the container(s), ensuring no headspace.

Note: Samples to be analyzed for VOCs or other compounds degraded by aeration should be taken as grab samples. Do not homogenize or composite these samples.

4. Once each container is filled, close the container with the Teflon-lined cap. Wipe the outside of the container clean with a Kimwipe or clean paper towel. Affix a completed sample label.

5. Place the sample container(s) in individual zip-top plastic bags and seal the bags.

6. Immediately pack all samples into a chilled cooler.

5.5.2 Method for Collecting Samples for Nonvolatile Organic and Inorganic Compound Analysis

The following steps must be taken when collecting shallow water sediment/sludge samples for analytes not degraded by aeration:

1. Collect sufficient volume to fill specified sample containers using decontaminated stainless steel or Teflon-lined equipment (e.g., scoops, corer, dredge sampler, etc.). If wading is necessary, approach the sample location from downstream. Do not enter the actual sample area.

2. Retrieve the sampling device with the sample and slowly decant off any liquid.

3. Pool and homogenize samples in a stainless steel, Teflon, or appropriate pan or mixing bowl, using stainless steel spatula or spoon.

4. Fill each sample container with the homogenized sample to approximately 75 to 90 percent capacity, filling sample containers for organic analyses first.

5. Once each container is filled, close the container with a Teflon-lined cap. Wipe the outside of sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.

6. Place the sample container(s) in individual zip-top plastic bags and seal the bags.

7. Immediately pack all samples into a chilled cooler.

5.6 Subsurface Sediment/Sludge Sample Collection Using a Corer or Auger from Shallow Waters

5.6.1 Method for Collecting Samples for Volatile Organic Compound Analysis Using an Unlined Corer

(also applies to augers)

The following steps must be taken when collecting subsurface sediment/sludge VOC samples that underlie shallow water:

1. At the specified sampling location, force or drive the corer to the specified depth.

2. Twist and withdraw the corer in a smooth motion.

3. Retrieve the sampling device, remove the corer nosepiece (if possible), and extrude the sample into the specified sampling container(s). Use a clean stainless steel or Teflon spoon or spatula to completely fill the container(s), ensuring no headspace.

4. Once each container is filled, close the container with the Teflon-lined cap. Wipe the outside of the sample container clean with a Kimwipe or clean paper towel. Affix a completed sample label.

5. Place the sample container(s) in individual zip-top plastic bags and seal the bags.

6. Immediately pack all samples into a chilled cooler.

5.6.2 Method for Collecting Samples for Volatile Organic Compound Analysis Using a Lined Corer

The following steps must be followed when collecting shallow water subsurface sediment/sludge VOC samples that underlie shallow water:

1. Install decontaminated liner(s) in the corer barrel.

2. At the specified sampling location, force or drive the corer to the specified depth.

3. Twist and withdraw the corer in a smooth motion.

4. Retrieve the sampling device, remove the corer nosepiece (if possible) and remove the liner(s), cap the liner(s), and seal the caps with Teflon tape.

5. Wipe the outside of the liner clean with a Kimwipe or clean paper towel. Label the top and bottom ends of the liner(s). Affix a completed sample label.

6. Place capped and sealed liners in individual zip-top plastic bags and seal the bags.

7. Immediately pack all samples into a chilled cooler.

5.6.3 Method for Collecting Samples for Nonvolatile Organic and Inorganic Compound Analysis Using a Corer (also applies to augers)

The following steps must be followed when collecting subsurface sediment/sludge samples that underlie shallow water for analytes not degraded by aeration:

1. At the specified sampling location, force or drive the corer to the specified depth.
2. Twist and withdraw the corer in a smooth motion.
3. Retrieve the sampling device. Remove the corer nosepiece (if possible) and extrude the sample into a stainless steel or Teflon-lined pan or bowl. Collect sufficient sample volume to fill all containers.
4. Use a stainless steel or Teflon spoon or spatula to homogenize and then divide the sample material into the appropriate number of sample containers.
5. Fill each container to approximately 75 to 90 percent capacity, filling containers for organic analyses first. Close the container with a Teflon-lined cap. Wipe the outside of sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.
6. Place the sample container(s) in individual zip-top plastic bags and seal the bags.
7. Immediately pack all samples into a chilled cooler.

5.7 Sediment/Sludge Sample Collection Using a Dredge from Deep Waters

5.7.1 Method for Collecting Samples for Volatile Organic Compound Analysis

The following steps must be followed when collecting deep-water sediment/sludge VOC samples:

1. Attach a clean piece of ½- to ¾-inch (12- to 19-mm) braided nylon line or Teflon-coated wire rope to the top of the sampler. The line must be of sufficient length to reach the sediment or sludge and have enough slack to release the mechanism. Mark the distance to the bottom on the line.
2. Attach the free end of the sampling line to a fixed support to prevent loss of the sampler.
3. At the specified sampling location, open the sampler jaws and slowly lower the sampler until contact with the bottom (sediment/sludge) is felt.
4. Release tension on the line and allow sufficient slack for the mechanism (latch) to release. Slowly raise the sampler.
5. Once the sampler is above the water surface, place the sampler in a decontaminated stainless steel or Teflon-lined tray or pan. Open the sampler. Immediately collect the sample for VOC analysis, using a stainless steel or Teflon spoon or spatula. Fill each container completely to minimize headspace.
6. Once each container is filled, close the container with the Teflon-lined cap. Wipe the outside of sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.
7. Place the sample container(s) in individual zip-top plastic bags and seal the bags.
8. Immediately pack all samples into a chilled cooler.

5.7.2 Method for Collecting Samples for Nonvolatile Organic and Inorganic Compounds

The following steps must be followed when collecting deep-water sediment/sludge samples for analytes not degraded by aeration:

1. Attach a clean piece of ½- to ¾-inch (12- to 19-mm) braided nylon line or Teflon-coated wire rope to the top of the sampler. The line must be of sufficient length to reach sediment or sludge and have enough slack to release the mechanism. Mark the distance to the bottom on the line.
2. Attach the free end of the sampling line to a fixed support to prevent loss of the sampler.
3. At the specified sampling location, open the sampler jaws and slowly lower the sampler until contact with the bottom (sediments/sludge) is felt.
4. Release tension on the line and allow sufficient slack for the mechanism (latch) to release. Slowly raise the sampler.
5. Once the sampler is above the water surface, place the sampler in a decontaminated stainless steel or Teflon-lined tray or pan. Open the sampler.
6. Collect sufficient volume of sample to fill the specified sampler containers. Pool the grab samples in a tray, pan, or bowl. Homogenize the pooled samples by mixing them together with a stainless steel or Teflon spoon or spatula.
7. Fill the specified sample containers to approximately 75 to 90 percent capacity with the homogenized sample using the stainless steel or Teflon spoon or spatula. Fill sample containers for organic analyses first.
8. Once each container is filled, close the container with the Teflon-lined cap. Wipe the outside of sample containers clean with a Kimwipe or clean paper towel. Affix a completed sample label.
9. Place sample container(s) in individual zip-top plastic bags and seal the bags.
10. Immediately pack all samples into a chilled cooler.

6.0 Restrictions/Limitations

Peristaltic pumps are generally not capable of lifting water distances greater than 20 to 25 feet (6 to 7.5 meters) above the normal hydrostatic level.

Core sampling devices may not be usable if cobbles exist in the sediment/sludge. Bumping of core sampling devices and Ponar dredge samplers may result in the loss of some of the sample.

Grab sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration is necessary to minimize sample disturbance and, hence, analyte loss. The representativeness of this sample, however, is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed.

7.0 References

U. S. Environmental Protection Agency, Region 2. *CERCLA Quality Assurance Manual*. March 1988 or current revision.

U. S. Geological Survey. *National Field Manual for the Collection of Water-Quality Data*, *Chapter A4*. September 1999. Updates are available at: http://water.usgs.gov/owq/FieldManual/chapter4/html/Ch4_contents.html

_____. A guide to the Proper Selection and Use of Federally Approved Sediment and Water Quality Samplers. Open-File Report 2005-1087. 2005.

2014. Region 4. The Field Branches Quality System and Technical Procedures, Sediment Sampling. SESDPROC-200-R3. August.

Packaging and Shipping Environmental Samples	SOP 2-1 Revision: 6 Date: February 2015

Approved: *Signature* **Technical Review:** T. Burgesser/C. Zakowski

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to outline the requirements for the packaging and shipment of environmental samples. Additionally, Sections 2.0 through 7.0 outline requirements for the packaging and shipping of regulated environmental samples under the Department of Transportation (DOT) Hazardous Materials Regulations, the International Air Transportation Association (IATA), and International Civil Aviation Organization (ICAO) Dangerous Goods Regulations for shipment by air and applies only to domestic shipments. This SOP does not cover the requirements for packaging and shipment of equipment (including data loggers and self-contained breathing apparatus [SCBAs] or bulk chemicals that are regulated under the DOT, IATA, and ICAO.

1.1 Packaging and Shipping of All Samples

This SOP applies to the packaging and shipping of all environmental samples. If the sample is preserved or radioactive, the following sections may also be applicable.

Section 2.0 - Packaging and Shipping Samples Preserved with Methanol
Section 3.0 - Packaging and Shipping Samples Preserved with Sodium Hydroxide
Section 4.0 - Packaging and Shipping Samples Preserved with Hydrochloric Acid
Section 5.0 - Packaging and Shipping Samples Preserved with Nitric Acid
Section 6.0 - Packaging and Shipping Samples Preserved with Sulfuric Acid
Section 7.0 - Packaging and Shipping Limited-Quantity Radioactive Samples

NOTE: This SOP does not address shipment of hazardous materials. Don't ship a hazardous material unless you have received training that meets the requirements of CDM Smith and the DOT. Check with CDM Smith University for training courses.

1.2 Background

1.2.1 Definitions

Environmental Sample - An aliquot of air, water, plant material, sediment, or soil that represents the contaminant levels on a site. Samples of potential contaminant sources, like tanks, lagoons, or non-aqueous phase liquids are normally not "environmental" for this purpose. This procedure applies only to environmental samples that contain less than reportable quantities for any foreseeable hazardous constituents according to DOT regulations promulgated in 49 CFR - Part 172.101 Appendix A.

Custody Seal - A custody seal is a narrow adhesive-backed seal that is applied to individual sample containers and/or the container (i.e., cooler) before offsite shipment. Custody seals are used to demonstrate that sample integrity has not been compromised during transportation from the field to the analytical laboratory.

Inside Container - The container, normally made of glass or plastic, that actually contacts the shipped material. Its purpose is to keep the sample from mixing with the ambient environment.

Outside Container - The container, normally made of metal or plastic, that the transporter contacts. Its purpose is to protect the inside container.

Secondary Containment - The outside container provides secondary containment if the inside container breaks (i.e., plastic overpackaging if liquid sample is collected in glass).

Excepted Quantity - Excepted quantities are limits to the mass or volume of a hazardous material in the inside and outside containers below which DOT, IATA, ICAO regulations do not apply. The excepted quantity limits are very low. Most regulated shipments will be made under limited quantity.

Limited Quantity - Limited quantity is the maximum amount of a hazardous material below which there are specific labeling or packaging exceptions.

Performance Testing - Performance testing is the required testing of outer packaging. These tests include drop and stacking tests.

Qualified Shipper - A qualified shipper is a person who has been adequately trained to perform the functions of shipping hazardous materials.

1.2.2 Associated Procedures

- SOP 1-2, *Sample Custody*

1.2.3 Discussion

Proper packaging and shipping is necessary to ensure the protection of the integrity of environmental samples shipped for analysis. These shipments are potentially subject to regulations published by DOT, IATA, or ICAO. Failure to abide by these rules places both CDM Smith and the individual employee at risk of serious fines. The analytical holding times for the samples must not be exceeded. The samples shall be packed in time to be shipped for overnight delivery. Make arrangements with the laboratory before sending samples for weekend delivery.

1.3 Required Equipment

- Coolers with return address of the appropriate CDM Smith office
- Heavy-duty plastic garbage bags
- Plastic zip-type bags, small and large
- Clear tape
- Nylon reinforced strapping tape
- Duct tape
- Kitty litter/pine bedding (or an equivalent nonflammable material that is inert and absorbent)*
- Bubble wrap (optional)
- Ice
- Custody seals
- Completed chain-of-custody record or contract laboratory program (CLP) custody records, if applicable
- Completed bill of lading
- This End Up and directional arrow labels

*Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

1.4 Packaging Environmental Samples

The following steps must be followed when packing sample bottles and jars for shipment:

1. Verify the samples undergoing shipment meet the definition of “environmental sample” and are not a hazardous material as defined by DOT. Professional judgment and/or consultation with qualified persons such as the appropriate health and safety coordinator or the health and safety manager shall be observed.

2. Select a sturdy cooler in good repair. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Ensure the handles used for carrying the cooler are in good repair. Also, tape the drain plug from the outside of the cooler. Line the cooler with a large heavy-duty plastic garbage bag.

3. Be sure the caps on all bottles are tight (will not leak); check to see that labels and chain-of-custody records are completed properly (SOP 1-2, Sample Custody).

4. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Up to three volatile organic analyte (VOA) vials may be packed in one bag. Binding the vials together with a rubber band on the outside of the bag, or separating them so that they do not contact each other, will reduce the risk of breakage. VOA vials may be packaged in foam containers designed for packaging them as well. Bottles may be wrapped in bubble wrap. Optionally,

   place three to six VOA vials in a quart metal can and then fill the can with kitty litter/pine bedding or equivalent. ***Note***: Trip blanks must be included in coolers containing VOA samples.

5. Place 2 to 4 inches of an absorbent material into a cooler that has been lined with a garbage bag, and then place the bottles and cans in the bag with sufficient space to allow for the addition of packing material between the bottles and cans. It is preferable to place glass sample bottles and jars into the cooler vertically. Glass containers are less likely to break when packed vertically rather than horizontally.

6. While placing sample containers into the cooler, conduct an inventory of the contents of the shipping cooler against the chain-of-custody record. The chain-of-custody with the cooler shall reflect only those samples within the cooler.

7. Put ice in large plastic zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place the ice bags on top of and/or between the samples. Several bags of ice are required (dependant on outdoor temperature, staging time, etc.) to maintain the cooler temperature at approximately 4° Celsius (C) if the analytical method requires cooling. Fill all remaining space between the bottles or cans with packing material. Securely fasten the top of the large garbage bag with fiber or duct tape.

8. Place the completed chain-of-custody record or the CLP traffic report form (if applicable) for the laboratory into a plastic zip-top bag, seal the bag, tape the bag to the inner side of the cooler lid and close the cooler.

9. The cooler lid shall be secured with nylon reinforced strapping tape by wrapping each end of the cooler a minimum of two times. Attach a completed chain-of-custody seal across the opening of the cooler on opposite sides. The custody seals shall be affixed to the cooler with half of the seal on the strapping tape so that the cooler cannot be opened without breaking the seal. Complete two more wraps around with fiber tape and place clear tape over the custody seals.

10. The shipping container lid must be marked **"THIS END UP"** and arrow labels that indicate the proper upward position of the container shall be affixed to the cooler. A label containing the name and address of the shipper (CDM Smith) shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Air Craft, Flammable Solids, etc.) are not permitted on the outside of containers used to transport environmental samples and shall not be used. The name and address of the laboratory shall be placed on the container, or when shipping by common courier, the bill of lading shall be completed and attached to the lid of the shipping container.

2.0 Packaging and Shipping Samples Preserved with Methanol

2.1 Containers

1. The maximum volume of methanol in a sample container is limited to 30 milliliters (ml).

2. The sample container must not be full of methanol.

2.2 Responsibility

It is the responsibility of the qualified shipper to:

1. Ensure that the samples undergoing shipment contain no other contaminant that meets the definition of "hazardous material" as defined by DOT.

2. Determine the amount of preservative in each sample so that accurate determination of quantities can be made.

   Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

2.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

1. Inner packing may consist of glass or plastic jars

2. Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test
3. Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
4. Class 3 flammable liquid labels
5. Orientation labels
6. Consignor/consignee labels

2.4 Packaging Samples Preserved with Methanol

The following steps are to be followed when packaging limited-quantity sample shipments:

1. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
2. All sample containers will be properly labeled and the label protected with waterproof tape before sampling.

   At a minimum the label must contain:

   - Project name
   - Project number
   - Date and time of sample collection
   - Sample location
   - Sample identification number
   - Collector's initials
   - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)

3. Wrap each container (40-ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.
4. Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
5. Place wrapped containers inside a polyethylene bottle filled with an absorbent; seal the bottle. (Maximum of 4 VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)
6. Total volume of methanol per shipping container must not exceed 500 ml.
7. Place sufficient amount of an absorbent in the bottom of the cooler to absorb any leakage that may occur.
8. Place a garbage bag in the cooler.
9. Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
10. Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
11. Seal the garbage bag by tieing or taping.
12. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
13. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
14. If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.

15. Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.

16. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.

17. Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

    Methanol Mixture
    UN1230
    LTD. QTY.

18. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.

19. Affix a Flammable Liquid label to the outside of the cooler.

20. Affix package orientation labels on two opposite sides of the cooler.

21. Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of cooler labeling/marking locations is shown in Figure 1.

    Note: No marking or labeling can be obscured by strapping or duct tape.

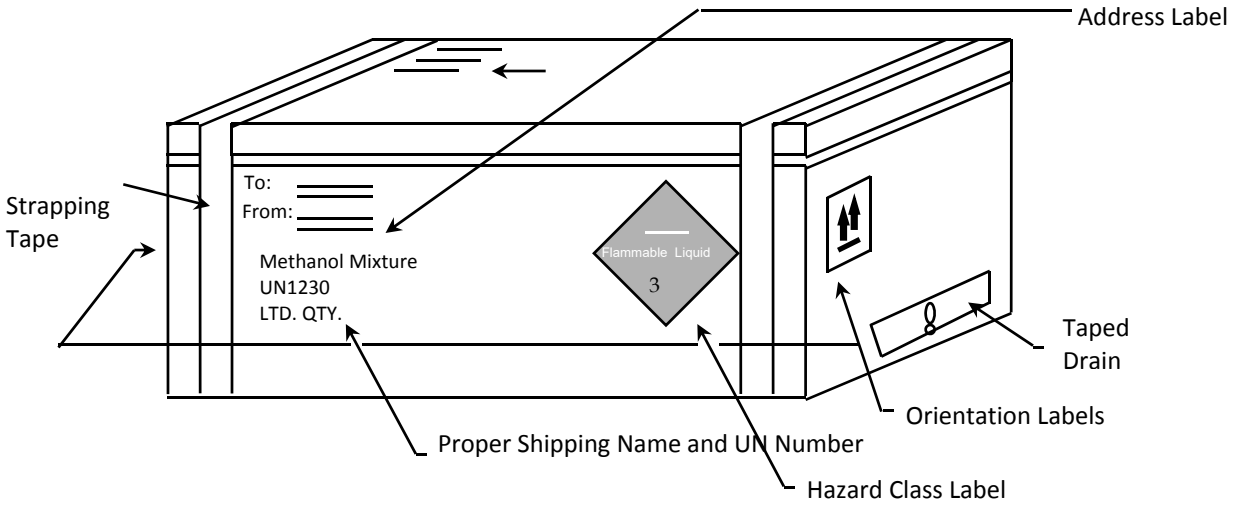
    Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

22. When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 millirem/hour (mrem/h) on all sides. This survey will be documented and the results reviewed by the qualified shipper.

23. Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity(Appendix A).

24. Complete a Dangerous Goods Airbill.

Figure 1
Example of Cooler Label/Marking Locations



3.0 Packaging and Shipping Samples Preserved with Sodium Hydroxide

3.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Sodium Hydroxide Preservatives

Preservative		*Desired in Final Sample*		*Quantity of Preservative (ml) for Specified Container*				
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**	**500 ml**	**1 L**
NaOH	30%	>12	0.08%		.25	0.5	1	2

5 drops = 1 ml

3.2 Responsibility

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QAPP.

3.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test
- Inner packings may consist of glass or plastic jars no larger than 1 pint
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

3.4 Packaging Samples Preserved with Sodium Hydroxide

Samples containing sodium hydroxide (NaOH) as a preservative that exceed the excepted concentration of 0.08 percent (2 ml of a 30 percent NaOH solution per liter) may be shipped as a limited quantity per packing instruction Y819 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited-quantity samples shipments:

1. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

2. All sample containers will be properly labeled and the label protected with waterproof tape before sampling.

   At a minimum the label must contain:

   - Project name
   - Project number
   - Date and time of sample collection
   - Sample location
   - Sample identification number
   - Collector's initials
   - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)

3. This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.

4. Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
5. Place glass containers inside a polyethylene bottle filled with an absorbent; seal the bottle.
6. The total volume of sample in each cooler must not exceed 1 liter.
7. Place sufficient amount of an absorbent in the bottom of the cooler to absorb any leakage that may occur.
8. Place a garbage bag in the cooler.
9. Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
10. Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
11. Seal the garbage bag by tieing or taping.
12. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
13. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
14. If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
15. Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
16. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
17. Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

    Sodium Hydroxide Solution
    UN1824
    LTD. QTY.

18. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
19. Affix a Corrosive label to the outside of the cooler.
20. Affix package orientation labels on two opposite sides of the cooler.
21. Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of cooler labeling/marking locations is shown in Figure 1.

    Note: Samples meeting the exception concentration of 0.08 percent NaOH by weight may be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.

    Note: No marking or labeling can be obscured by strapping or duct tape.

    Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

22. When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

23. Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).

24. Complete a Dangerous Goods Air Bill.

4.0 Packaging and Shipping Samples Preserved with Hydrochloric Acid

4.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Hydrochloric Acid Preservatives

Preservative		*Desired in Final Sample*		*Quantity of Preservative (ml) for Specified Container*		
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**
HCl	2N	<1.96	0.04%	.2	.5	1

5 drops = 1 ml

4.2 Responsibility

It is the responsibility of the qualified shipper to:

- Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT.

- Determine the amount of preservative in each sample so that accurate determination of quantities can be made.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QAPP.

4.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3.

- Inner packing may consist of glass or plastic jars no larger than 1 pint.
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

4.4 Packaging Samples Preserved with Hydrochloric Acid

The following steps are to be followed when packaging limited-quantity sample shipments:

1. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

2. All sample containers will be properly labeled and the label protected with waterproof tape before sampling.

At a minimum the label must contain:

- Project name
- Project number
- Date and time of sample collection
- Sample location
- Sample identification number
- Collector's initials
- Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)

- Wrap each container (40-ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place wrapped containers inside a polyethylene bottle filled with an absorbent; seal the bottle. (No more than 4 VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)
- Total volume of sample inside each cooler must not exceed 1 liter.
- Place sufficient amount of an absorbent in the bottom of the cooler to absorb any leakage that may occur.

3. Place a garbage bag in the cooler.
4. Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
5. Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
6. Seal the garbage bag by tieing or taping.
7. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
8. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
9. If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
10. Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
11. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
12. Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

    Hydrochloric Acid Solution
    UN1789
    LTD. QTY.

13. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
14. Affix a Corrosive label to the outside of the cooler.
15. Affix package orientation labels on two opposite sides of the cooler.

16. Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of cooler labeling/marking locations is shown in Figure 1.

    Note: Samples containing less than the exception concentration of 0.04 percent HCl by weight will be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.

    Note: No marking or labeling can be obscured by strapping or duct tape.

    Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

17. When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

18. Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).

19. Complete a Dangerous Goods Airbill.

5.0 Packaging and Shipping Samples Preserved with Nitric Acid

5.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Nitric Acid Preservatives

Preservative		*Desired in Final Sample*		*Quantity of Preservative (ml) for Specified Container*				
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**	**500 ml**	**1 L**
HNO_3	6N	<1.62	0.15%		2	4	5	8

5 drops = 1 mg/L

5.2 Responsibility

It is the responsibility of the qualified shipper to:

1. Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT.

2. Determine the amount of preservative in each sample so that accurate determination of quantities can be made.

    Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QAPP.

5.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Inner packings may consist of glass or plastic jars no larger than 100 ml.
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

5.4 Packaging Samples Preserved with Nitric Acid

Samples containing nitric acid (HNO_3)as a preservative that exceed the excepted concentration of 0.15 percent HNO_3 will be shipped as a limited quantity per packing instruction Y807 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited-quantity sample shipments:

1. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

2. All sample containers will be properly labeled and the label protected with waterproof tape before sampling.

   At a minimum the label must contain:
   - Project name
   - Project number
   - Date and time of sample collection
   - Sample location
   - Sample identification number
   - Collector's initials
   - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)

3. This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.

4. Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.

5. Place glass containers inside a polyethylene bottle filled with an absorbent; seal the bottle.

6. Place sufficient amount of an absorbent in the bottom of the cooler to absorb any leakage that may occur.

7. Place a garbage bag in the cooler.

8. Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.

9. Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.

10. Seal the garbage bag by tieing or taping.

11. The maximum volume of preserved solution in the cooler must not exceed 500 ml.

12. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.

13. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.

14. If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.

15. Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.

16. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.

17. Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

    Nitric Acid Solution (with less than 20 percent)
    UN2031
    Ltd. Qty.

18. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.

19. Affix a Corrosive label to the outside of the cooler.

20. Affix package orientation labels on two opposite sides of the cooler.

21. Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of cooler labeling/marking locations is shown in Figure 1.

    Note: Samples meeting the exception concentration of 0.15 percent HNO3 by weight will be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.

    Note: No marking or labeling can be obscured by strapping or duct tape.

    Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

22. When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

23. Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).

24. Complete a Dangerous Goods Airbill.

6.0 Packaging and Shipping Samples Preserved with Sulfuric Acid

6.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Sulfuric Acid Preservatives

Preservative		*Desired in Final Sample*		*Quantity of Preservative (ml) for Specified Container*				
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**	**500 ml**	**1 L**
H_2SO_4	37N	<1.15	0.35%	.1	.25	0.5	1	2

5 drops = 1 ml

6.2 Responsibility

It is the responsibility of the qualified shipper to:

1. Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT.

2. Determine the amount of preservative in each sample so that accurate determination of quantities can be made.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QAPP.

6.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Inner packings may consist of glass or plastic jars no larger than 100 ml.
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

6.4 Packaging of Samples Preserved with Sulfuric Acid

Samples containing sulfuric acid (H_2SO_4) as a preservative that exceed the excepted concentration of 0.35 percent will be shipped as a limited quantity per packing instruction Y809 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited-quantity samples shipments:

1. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

2. All sample containers will be properly labeled and the label protected with waterproof tape before sampling.

   At a minimum the label must contain:

   - Project name
   - Project number
   - Date and time of sample collection
   - Sample location
   - Sample identification number
   - Collector's initials
   - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)

3. Wrap each glass container in bubble wrap (secure with waterproof tape) to prevent breakage.

4. Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.

5. Place glass containers inside a polyethylene bottle filled with an absorbent; seal the bottle.

6. Place sufficient amount of an absorbent in the bottom of the cooler to absorb any leakage that may occur.

7. Place a garbage bag in the cooler.

8. Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.

9. Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.

10. Seal the garbage bag by tieing or taping.

11. The maximum volume of preserved solution in the cooler must not exceed 500 ml.

12. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.

13. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.

14. If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.

15. Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.

16. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.

17. Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

    Sulfuric Acid Solution
    UN2796
    LTD. QTY.

18. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.

19. Affix a Corrosive label to the outside of the cooler.

20. Affix package orientation labels on two opposite sides of the cooler.

21. Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of cooler labeling/marking locations is shown in Figure 1.

    Note: Samples containing less than the exception concentration of 0.35 percent H_2SO_4 by weight will be shipped as nonregulated or nonhazardous in accordance with the procedure described in Section 1.4.

    Note: No marking or labeling can be obscured by strapping or duct tape.

    Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

22. When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

23. Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).

24. Complete a Dangerous Goods Airbill.

7.0 Packaging and Shipping Limited-Quantity Radioactive Samples

7.1 Containers

The inner packaging containers that may be used for these shipments include:

1. Any size sample container

7.2 Description/Responsibilities

The qualified shipper will determine that the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT.

The qualified shipper will ship all samples that meet the Class 7 definition of radioactive materials and meet the activity requirements specified in Table 4 and 7 of 49 CFR 173.425, as Radioactive Materials in Limited Quantity. The qualified shipper will verify that all packages and their contents meet the requirements of 49 CFR 173.421, *Limited Quantities of Radioactive Materials*.

The packaging used for shipping will meet the general requirements for packaging and packages specified in 49 CFR 173.24 and the general design requirements provided in 173.410. These standards state that a package must be capable of withstanding the effects of any acceleration, vibration, or vibration resonance that may arise under normal condition of transport without any deterioration in the effectiveness of the closing devices on the various receptacles or in the integrity of the package as a whole and without loosening or unintentionally releasing the nuts, bolts, or other securing devices even after repeated use.

If the shipment is from a DOE facility, radiological screenings will be completed on all samples taken. The qualified shipper will review the results of each screening (alpha, beta, and gamma speciation). Samples will not be shipped offsite until the radiological screening has been performed.

The total activity for each package will not exceed the relevant limits listed in Table 4 and 7 of 49 CFR 173.425. The A_2 value of the material will be calculated based on all radionuclides found during previous investigations (if any) in the area from which the samples are derived. The A_2 values to be used will be the most restrictive of all potential radionuclides as listed in 49 CFR 173.435.

The radiation level at any point on the external surface of the package bearing the sample(s) will not exceed 0.005 millisievert per hour (mSv/h) (0.5 mrem/hour). These will be verified by dose and activity monitoring before shipment of the package.

The removable radioactive surface contamination on the external surface of the package will not exceed the limits specified in 49 CFR 173.443(a). CDM Smith will apply the DOE-established free release criteria for removable surface contamination of less than 20 dpm/100 cm^2 (alpha) and 1,000 dpm/100 cm^2 (beta/gamma). It shall be noted that these values are more conservative than the DOT requirements for removable surface contamination.

The qualified shipper will verify that the outside of the inner packaging is marked “Radioactive.”

The qualified shipper will verify that the excepted packages prepared for shipment under the provisions of 49 CFR 173.421 have a notice enclosed, or shown on the outside of the package, that reads, **“This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910.”**

> ***Note:*** Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QAPP.

7.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Survey documentation/radiation screening results (if shipping from DOE or radiological sites)
- Orientation labels
- Excepted quantities label
- Consignor/consignee labels

7.4 Packaging of Limited-Quantity Radioactive Samples

The following steps are to be followed when packaging limited-quantity sample shipments:

1. The cooler is to be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

2. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

3. All sample containers will be properly labeled and the label protected with waterproof tape before sampling. At a minimum the label must contain:

   - Project name
   - Project number
   - Date and time of sample collection
   - Sample location
   - Sample identification number
   - Collector's initials

4. This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.

5. Place sufficient amount of an absorbent, or approved packaging material, in the bottom of the cooler to absorb any leakage that may occur.

6. Place a garbage bag in the cooler.

7. Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.

8. If required, place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.

9. Seal the garbage bag by tieing or taping.

10. Place a label marked Radioactive on the outside of the sealed bag.

11. Enclose a notice that includes the name of the consignor or consignee and the following statement: ***"This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910."***

12. Note that both DOT and IATA apply different limits to the quantity in the inside packing and in the outside packing.

13. The maximum weight of the package shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.

14. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.

15. If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.

16. If a cooler is used, wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.

17. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.

18. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.

19. Affix package orientation labels on two opposite sides of the cooler/package.

20. Affix a completed Excepted Quantities label to the side of the cooler/package.

21. Secure any marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of the cooler labeling/marking is shown in Figure 2.

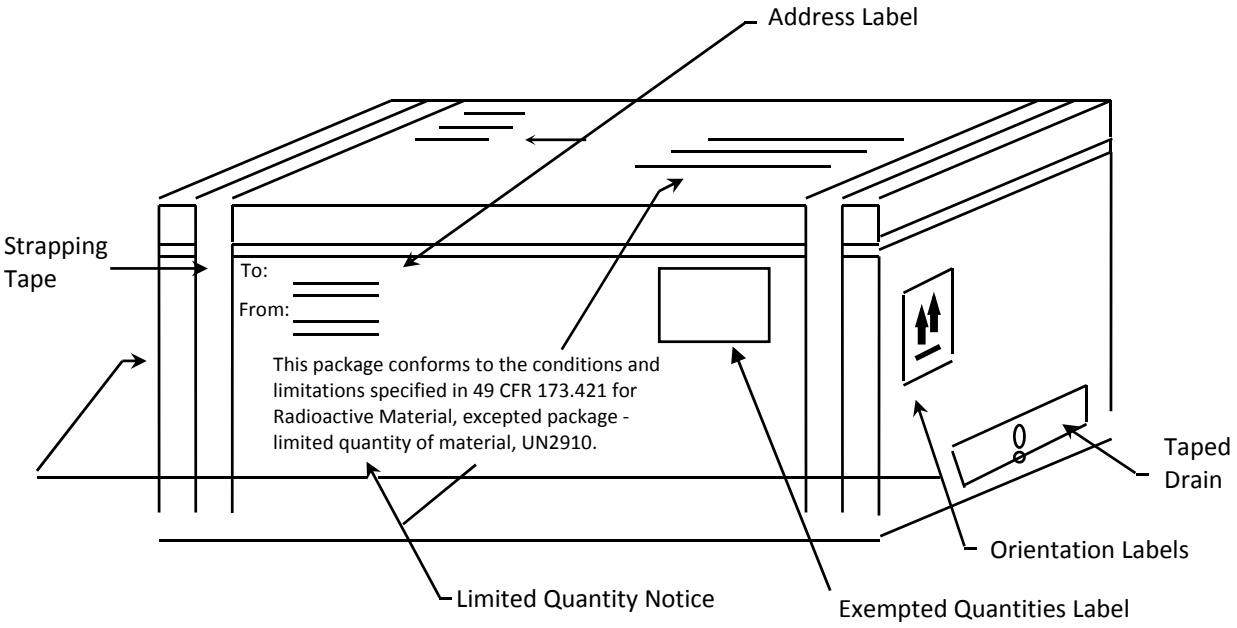
    Note: No marking or labeling can be obscured by strapping or duct tape.

22. Complete the Shipment Quality Assurance Checklist (Appendix B).

    Note: Except as provided in 49 CFR 173.426, the package will not contain more than 15 grams of ^{235}U.

    Note: A declaration of dangerous goods is not required.

Figure 2
Radioactive Material – Limited-Quantity Cooler Marking Example



8.0 References

U. S. Environmental Protection Agency. Region IV. May 2013, or current revision. *Field Branches Quality Management Plan*.

_____. August 2011 or current revision. Region IV. *Field Branches Quality Policy*.

_____. 2007 or current revision. *Sampler's Guide, Contract Laboratory Program, Guidance for Field Samplers*, EPA-540-R-07-06.

Title 49 Code of Federal Regulations, Department of Transportation. 2015 or current revision. *Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, and Training Requirements*, 49 CFR 172.

Title 49 Code of Federal Regulations, Department of Transportation. 2015 or current revision. *Shippers - General Requirements for Shipments and Packagings*, 49 CFR 173.

Appendix A

Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity

Sample Packaging

Yes	No	N/A	
.	☐	☐	The VOA vials are wrapped in bubble wrap and placed inside a zip-type bag.
☐	☐	☐	The VOA vials are placed into a polyethylene bottle, filled with an absorbent, and tightly sealed.
☐	☐	☐	The drain plug is taped inside and outside to ensure control of interior contents.
☐	☐	☐	The samples have been placed inside garbage bags with sufficient bags of ice to preserve samples at 4°C.
☐	☐	☐	The cooler weighs less than the 66-pound limit for limited-quantity shipment.
☐	☐	☐	The garbage bag has been sealed with tape (or tied) to prevent movement during shipment.
☐	☐	☐	The chain-of-custody has been secured to the interior of the cooler lid.
☐	☐	☐	The cooler lid and sides have been taped to ensure a seal.
☐	☐	☐	The custody seals have been placed on both the front and back hinges of the cooler, using waterproof tape.

Air Waybill Completion

Yes	No	N/A	
☐	☐	☐	Section 1 has the shipper's name, company, and address; the account number, date, internal billing reference number; and the telephone number where the shipper can be reached.
☐	☐	☐	Section 2 has the recipient's name and company along with a telephone number where they can be reached.
☐	☐	☐	Section 3 has the **Bill Sender** box checked.
☐	☐	☐	Section 4 has the **Standard Overnight** box checked.
☐	☐	☐	Section 5 has the **Deliver Weekday** box checked.
☐	☐	☐	Section 6 has the number of packages and their weights filled out. Was the total of all packages and their weights figured up and added at the bottom of Section 6?
☐	☐	☐	Under the **Transport Details** box, the **Cargo Aircraft Only** box is obliterated, leaving only the **Passenger and Cargo Aircraft** box.
☐	☐	☐	Under the **Shipment Type**, the **Radioactive** box is obliterated, leaving only the **Non-Radioactive** box.
☐	☐	☐	Under the **Nature and Quantity of Dangerous Goods** box, the **Proper Shipping Name, Class or Division, UN or ID No., Packing Group, Subsidiary Risk, Quantity and Type of Packing, Packing Instructions, and Authorization** have been filled out for the type of chemical being sent.
☐	☐	☐	The **Name, Place and Date, Signature, and Emergency Telephone Number** appears at the bottom of the FedEx Airbill.
☐	☐	☐	The statement "In accordance with IATA/ICAO" appears in the **Additional Handling Information** box.
☐	☐	☐	The **Emergency Contact Information** at the bottom of the FedEx Airbill is truly someone who can respond any time of the day or night.

Packaging and Shipping Environmental Samples

SOP 2-1
Revision: 6
Date: February 2015

Proper Shipping Name	*Class or Division*	*UN or ID No.*	*Packing Group*	*Sub Risk*	*Quantity*	*Packing Instruction*	*Authorization*
Hydrochloric Acid Solution	8	UN1789	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Nitric Acid Solution (with less than 20%)	8	UN2031	II		1 plastic box × 0.5 L	Y807	Ltd. Qty.
Sodium Hydroxide Solution	8	UN1824	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Sulfuric Acid Solution	8	UN2796	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Methanol	3	UN1230	II		1 plastic box × 1 L	Y305	Ltd. Qty.

Sample Cooler Labeling

Yes	No	N/A	
☐	☐	☐	The proper shipping name, UN number, and Ltd. Qty. appears on the shipping container.
☐	☐	☐	The corresponding hazard labels are affixed on the shipping container; the labels are not obscured by tape.
☐	☐	☐	The name and address of the shipper and receiver appear on the top and side of the shipping container.
☐	☐	☐	The air waybill is attached to the top of the shipping container.
☐	☐	☐	**Up Arrows** have been attached to opposite sides of the shipping container.
☐	☐	☐	Packaging tape does not obscure markings or labeling.

Packaging and Shipping Environmental Samples	SOP 2-1 Revision: 6 Date: February 2015

Appendix B

Shipment Quality Assurance Checklist

Date: _____ Shipper: _____ Destination: _____

Item(s) Description: _____

Radionuclide(s): _____

Radiological Survey Results: surface _____mrem/hr 1 meter _____

Instrument Used: Mfgr: _____ Model: _____

S/N: _____ Cal Date: _____

Limited-Quantity or Instrument and Article

Yes	**No**		
___	___	1.	Strong tight package (package that will not leak material during conditions normally incidental to transportation).
___	___	2.	Radiation levels at any point on the external surface of package less than or equal to 0.5 mrem/hr.
___	___	3.	Removable surface contamination less than 20 dpm/100 cm^2 (alpha) and 1,000 dpm/100 cm^2 (beta/gamma).
___	___	4.	Outside inner package bears the marking "Radioactive."
___	___	5.	Package contains less than 15 grams of ^{235}U (check yes if ^{235}U not present).
___	___	6.	Notice enclosed in or on the package that includes the consignor or consignee and the statement, **"This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910."**
___	___	7.	Activity less than that specified in 49 CFR 173.425. Permissible package limit: Package Quantity:
___	___	8.	On all air shipments, the statement **Radioactive Material, excepted package-limited quantity of material** shall be noted on the air waybill.

Qualified Shipper: _____ Signature: _____

Guide to Handling Investigation-Derived Waste	SOP 2-2 Revision: 8 Date: February 2015

Approved: *Signature* **Technical Review:** Dave Sembrot

1.0 Objective

This technical standard operating procedure (SOP) presents guidance for the management of investigation-derived waste (IDW). The primary objectives for managing IDW during field activities include:

- Leaving the site in no worse condition than existed before field activities
- Removing wastes that pose an immediate threat to human health or the environment
- Proper handling of onsite wastes that do not require offsite disposal or extended aboveground containerization
- Complying with federal, state, local, and facility applicable or relevant and appropriate requirements (ARARs)
- Careful planning and coordination of IDW management options
- Minimizing the quantity of IDW

2.0 Background

2.1 Definitions

Hazardous Waste - Discarded material that is regulated listed waste, or waste that exhibits ignitability, corrosivity, reactivity, or toxicity as defined in 40 CFR 261.3 or state regulations.

Investigation-Derived Wastes - Discarded materials resulting from field activities such as sampling, surveying, drilling, excavation, and decontamination processes that, in present form, possess no inherent value or additional usefulness without treatment. Wastes may be solid, sludge, liquid, gaseous, or multiphase materials that may be classified as hazardous or nonhazardous.

Mixed Waste - Any material that has been classified as both hazardous and radioactive.

Radioactive Wastes - Discarded materials that are contaminated with radioactive constituents with specific activities in concentrations greater than the latest regulatory criteria (i.e., 10 CFR 20).

Treatment, Storage, and Disposal Facility (TSDF) - Permitted facilities that accept hazardous waste shipments for further treatment, storage, and/or disposal. These facilities must be permitted by the U. S. Environmental Protection Agency (EPA) and appropriate state and local agencies.

Aqueous liquid – a water based polar solution with a specific gravity at or near 1. Light non-aqueous phase liquids, also known as (a.k.a) LNAPL (non-polar), such as oils, typically float on aqueous (polar) solutions (or pure water). Dense non-aqueous phase liquids (a.k.a. DNAPL), such as chlorinated organic solvents or PCB containing oils, sink in aqueous based liquids.

2.2 Discussion

Field investigation activities result in the generation of waste materials that may be characterized as hazardous or radioactive. IDWs may include drilling muds, cuttings, and purge water from test pit and well installation; purge water, soil, and other materials from collection of samples; residues from testing of treatment technologies and pump and treat systems; personal protective equipment (PPE); solutions (aqueous or otherwise) used to decontaminate nondisposable protective clothing and equipment; and other wastes or supplies used in sampling and testing potentially hazardous or radiologically contaminated material.

Note: The client's representatives may not be aware of all potential contaminants. The management of IDW must comply with applicable regulatory requirements.

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that all IDW procedures are conducted in accordance with this SOP. The site manager is also responsible for ensuring that handling of IDW is in accordance with site-specific requirements.

Project Manager - The project manager is responsible for identifying site-specific requirements for the disposal of IDW in accordance with federal, state, and/or facility requirements.

Field Crew Members - Field crew members are responsible for implementing this SOP and communicating any unusual or unplanned condition to the project manager's attention.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/project specific quality assurance plan.

4.0 Required Equipment

Equipment required for IDW containment will vary according to site-specific/client requirements. Management decisions concerning the necessary equipment required shall consider: containment method, sampling, labeling, maneuvering, and storage (if applicable). Equipment must be onsite and inspected before commencing work.

The selection of the container type and size for containerizing IDW must consider:

- waste/contaminant segregation (i.e. do not mix hazardous and non-hazardous wastes or incompatible materials),
- efficiency/ability to move the containerized waste (i.e. size of equipment needed vs. accessibility and bulk vs. individual containers),
- cost of storage, (i.e. rental vs. purchase)
- transportation and disposal of the material in the containers selected.

4.1 IDW Containment Devices

The appropriate containment device (drums, tanks, etc.) will depend on site- or client-specific requirements and the ultimate disposition of the IDW. Typical IDW containment devices can include:

- Plastic sheeting (polyethylene) with a minimum thickness of 20 micrometers
- Department of Transportation (DOT)-approved steel containers
- Polyethylene or steel bulk storage tanks

Containment of IDW shall be segregated by waste type (i.e., solid or liquid, corrosive or flammable, etc.) and source location. Volume of the appropriate containment device will depend on site-specific requirements.

4.2 IDW Container Labeling

A "Waste Container" or "IDW Container" label or indelible marking shall be applied to each container. Labeling or marking requirements for onsite IDW not expected to be transported offsite are as detailed below.

- Labels and markings must contain the following information: project name, generation date, location of waste origin, container identification number, sample number (if applicable), and contents (drill cuttings, purge water, PPE, etc.).
- Each label or marking will be applied to the upper one-third of the container at least twice, on opposite sides.
- Containers that are 5 gallons or less may only require one label or set of markings.
- Labels or markings will be positioned on a smooth part of the container. The label must not be affixed across container bungs, seams, ridges, or dents.
- Labels must be constructed of a weather-resistive material with markings made with a permanent marker or paint pen and capable of enduring the expected weather conditions. If markings are used, the color must be easily distinguishable from the container color.
- Labels will be secured in a manner to ensure that they remain affixed to the container.

Labeling or marking requirements for IDW expected to be transported offsite must be in accordance with the requirements of 49 CFR 172.

4.3 IDW Container Movement

Staging areas for IDW containers shall be predetermined and in accordance with site-specific and/or client requirements. Arrangements shall be made before field mobilization as to the methods and personnel required to safely transport IDW containers to the staging area. Transportation of IDW containers offsite via a public roadway is prohibited unless 49 CFR 172 requirements are met.

4.4 IDW Container Storage

Containerized IDW awaiting results of pending chemical analysis or further onsite treatment shall be staged on site. Staging areas and bulk storage procedures are to be determined according to site-specific requirements. Containers are to be stored in such a fashion that the labels can be easily read. A secondary/spill container must be provided for liquid IDW storage and as appropriate for solid IDW storage (e.g., steel drums shall not be stored in direct contact with the ground).

5.0 Procedures

The three general options for managing IDW are: (1) collection and onsite disposal, (2) collection for offsite disposal, and (3) collection and interim management. Attachment 1 summarizes media-specific information on generation processes and management options. The option selected shall take into account the following factors:

- Type (soil, sludge, liquid, debris), quantity, and source of IDW
- Risk posed by managing the IDW onsite
- Compliance with regulatory requirements
- IDW minimization and consistency with the IDW remedy and the site remedy

In all cases the client shall approve the plans for IDW. Formal plans for the management of IDW must be prepared as part of a work plan or separate document.

5.1 Collection and Onsite Disposal

5.1.1 Soil/Sludge/Sediment

The options for handling soil/sludge/sediment IDW are:

1. Return IDW to boring, pit, or source immediately after generation as long as returning the media to these areas will not increase site risks (e.g., so that “clean” areas are not contaminated, the IDW material will not be replaced at a greater depth, or in a different area than from where it was originally obtained.

2. Spread IDW around boring, pit, or source within the area of contamination (AOC) as long as returning the media to these areas will not increase site risks (e.g., direct contact with surficial contamination).

3. Consolidate IDW in a pit within the AOC as long as returning the media to these areas will not increase site risks (e.g., the contaminated soil will not be replaced at a greater depth than where it was originally so that it will not contaminate “clean” areas).

4. Send to onsite TSDF. This option may require results of laboratory analysis before treatment/disposal.

Note: These options may require client and/or regulatory approval.

5.1.2 Aqueous Liquids

The options for handling aqueous liquid IDW are:

1. Discharge to surface water, only when IDW is not contaminated, and with written client approval.

2. Discharge to ground surface close to the well from which it was extracted, only if soil contaminants will not be mobilized in the process and the action will not contaminate clean areas. If IDW from the sampling of background upgradient wells is not a community concern or associated with soil contamination, this presumably uncontaminated IDW may be released on the ground around the well with written client approval.

3. Discharge to sanitary sewer, only when IDW is not contaminated and with written client approval.

4. Send to onsite treatment/disposal facility, with facility acceptance and written client approval.

Note: These options may require results of laboratory analysis to obtain client and/or regulatory approval.

5. When small amounts (i.e., less than 5 gallons) of used decontamination fluids are generated during site characterization activities (e.g., during soil sampling using direct push technology methods), the fluids may be allowed to evaporate by spreading them on an asphalted surface, or allowing for evaporation from an open bucket.

5.1.3 Disposable PPE

The options for handling disposable PPE are:

1. Double-bag contents in nontransparent trash bags and place in onsite industrial dumpster, only if PPE is not contaminated.

2. Containerize, label, and send to onsite TSDF. This may require results of laboratory analysis before treatment/disposal.

5.2 Collection for Offsite Disposal

Before sending IDW to an offsite TSDF or to a publicly owned treatment works (POTW), laboratory analysis may be required. Manifests are required to accompany any IDW determined to be hazardous. In some instances, a bill of lading can be used for nonhazardous solid IDW (i.e., wooden pallets, large quantities of plastic sheeting). Arrangements must be made with the client responsible for the site to sign as generator on any waste profile and all manifests or bill of ladings; it is CDM Smith's policy not to sign any waste profile or manifest. The TSDF and transporter must be permitted for the respective wastes. Nonbulk containers (e.g., drums) must have a DOT-approved label adhered to the container and all required associated placard stickers before leaving for an offsite TSDF. These labels must include information as required in 49 CFR 172. Bulk containers (i.e., rolloffs, tanks) do not require container specific labels for transporting offsite, but must include appropriate placards as required in 49 CFR 172.

5.2.1 Soil/Sludge/Sediment

When the final site remedy requires offsite treatment and disposal, the IDW may be stored (e.g., drummed, covered in a waste pile) or returned to its source until final disposal. The management option selected shall take into account the potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

5.2.2 Aqueous Liquids

When the final site remedy requires offsite treatment and disposal, the IDW may be stored (e.g., mobile tanks or drums with appropriate secondary containment) until final disposal. The management option selected shall take into account the potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

5.2.3 Disposable PPE

When the final site remedy requires offsite treatment and disposal, the IDW may be containerized and stored. The management option selected shall take into account potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

5.3 Collection and Interim Management

All interim measures must be approved by the client and regulatory agencies.

1. Storing IDW onsite until the final action may be practical in the following situations:
   - Returning wastes (especially sludges and soils) to their onsite source area would require reexcavation for disposal as determined for the final site remedy.
   - Interim storage in containers may be necessary to provide adequate protection to human health and the environment.
   - Offsite disposal options may trigger land disposal regulations under the Resource Conservation and Recovery Act (RCRA). Storing IDW until the final disposal of all wastes from the site will eliminate the need to address this issue more than once.
   - Interim storage may be necessary to provide time for sampling and analysis.
2. Segregate and containerize all waste for future treatment and/or disposal.
   - Containment options for soil/sludge/sediment may include drums or covered waste piles in AOC.
   - Containment options for aqueous liquids may include mobile tanks or drums.
   - Containment options for PPE may include drums or roll-off boxes.

6.0 Restrictions/Limitations

Site managers shall determine the most appropriate disposal option for aqueous liquids on a site-specific basis. Parameters to consider, especially when determining the level of protection, include the volume of IDW, the contaminants present in the aqueous liquid, the nature of contaminants present in the site soil , and whether groundwater or surface water is a drinking water supply, and if obtained from contaminated groundwater, whether the plume is contained or migrating. Special disposal/handling may be needed for drilling fluids because they may contain significant solid components and therefore may need to be handled, treated, disposed as non-liquid wastes.

Disposable sampling materials, disposable PPE, decontamination fluids, etc. will always be managed on a site-specific basis. Under no circumstances shall these types of materials e stored in a site office or warehouse.

7.0 References

Environmental Resource Center. 1997. *Hazardous Waste Management Compliance Handbook 2nd Edition*. Karnofsky (Editor).

Academy of Certified Hazardous Materials Manager. May 1999. *Hazardous Materials Management Desk Reference*. Cox.

Title 49 Code of Federal Regulations, Department of Transportation. 2005 or current revision. *Hazardous Materials Table, Special Provisions, Hazardous, Materials Communications, Emergency Response Information, and Training Requirements*, 49 CFR 172.

U. S. Environmental Protection Agency. 1987. *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001.1.

_____. August 1990. *Low-Level Mixed Waste: A RCRA Perspective for NRC Licensees*, EPA/530-SW-90-057.

_____. May 1991. *Management of Investigation-Derived Wastes During Site Inspections*, EPA/540/G-91/009.

_____. January 1992. *Guide to Management of Investigation-Derived Wastes*, 9345.3-03FS.

_____. Region IV. November 2001. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*.

Attachment 1
IDW Management Options

Type of IDW	*Generation Processes*	*Management Options*
Soil	▪ Well/Test pit installations ▪ Borehole drilling ▪ Soil sampling	**Onsite Disposal** ▪ Return to boring, pit, or source immediately after generation ▪ Spread around boring, pit, or source within the AOC ▪ Consolidate in a pit (within the AOC) ▪ Send to onsite TSDF **Offsite Disposal** ▪ Client to send to offsite TSDF **Interim Management** ▪ Store for future treatment and/or disposal
Sludge/Sediment	▪ Sludge pit/sediment sampling	**Onsite Disposal** ▪ Return to pit or source immediately after generation ▪ Send to onsite TSDF **Offsite Disposal** ▪ Send to offsite TSDF* **Interim Management** ▪ Store for future treatment and/or disposal
Aqueous Liquids (groundwater, surface water, drilling fluids, wastewater)	▪ Well installation/development ▪ Well purging during sampling ▪ Groundwater discharge during pump tests ▪ Surface water sampling ▪ Wastewater sampling	**Onsite Disposal** ▪ Pour onto ground close to well (nonhazardous waste) ▪ Discharge to sewer ▪ Send to onsite TSDF **Offsite Disposal** ▪ Send to offsite TSDF* ▪ Client to send to publicly owned treatment works (POTW) **Interim Management** ▪ Store for future treatment and/or disposal
Decontamination Fluids	▪ Decontamination of PPE and equipment	**Onsite Disposal** ▪ Send to onsite TSDF ▪ Evaporate (for small amounts of low contamination organic fluids) ▪ Discharge to ground surface **Offsite Disposal** ▪ Send to offsite TSDF* ▪ Discharge to sewer **Interim Management** ▪ Store for future treatment and/or disposal
Disposable PPE and Sampling Equipment	▪ Sampling procedures or other onsite activities	**Onsite Disposal** ▪ Place in onsite industrial dumpster ▪ Send to onsite TSDF **Offsite Disposal** ▪ Send to offsite TSDF* **Interim Management** ▪ Store for future treatment and/or disposal

* Client must sign waste profile, manifest, etc. for any waste sent offsite.
Adapted from U. S. Environmental Protection Agency, *Guide to Management of Investigation-Derived Wastes*, 9345-03FS, January 1992.

Geoprobe® Sampling	SOP 3-1 Revision: 7 Date: February 2015
Approved: Signature	**Technical Review:** Stuart Barden

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to define the requirements for collecting soil, soil gas, groundwater, and pneumatic slug test data using the Geoprobe® sampling system. Geoprobe is a trade name proprietary to Geoprobe Systems of Salina, Kansas.

2.0 Background

2.1 Definitions

Geoprobe - A hydraulically-operated hammer device installed on the back of a van, pickup truck, or tracked vehicle used to advance a hollow-stem rod into the soil for the purpose of collecting soil, soil gas, or groundwater samples.

Probe-Drive Sampler - A sampling device, similar to a split-spoon sampler, used to collect soil samples with a Geoprobe rig. Two primary types of soil samplers are available: Dual Tube (DT), and Macro-Core® (MC) sampler (with a PVC liner). Sample core lengths range from 24 to 72-inches and diameters range from 1.125 to 4-inches. Search Geoprobe Systems website for probe rods and DT and MC samplers for additional specific sizes available.

Extension Rod - Stainless steel rod used to remove stop-pin and drive-point assembly.

Extension Rod Coupler - Stainless steel connector used to join sections of extension rods.

Drive Point - Solid steel retractable point used to advance sample collection device to the required sample depth.

Probe Rod - Hollow, flush-threaded, steel rod similar to a drill rod.

Stop-Pin - Steel plug that threads into the top of the drive cap to hold the drive point in place during advancement of the probe rods.

Drive Cap - Threaded, hardened-steel top cap that attaches to the top of the probe rod; used when advancing the probe rods with the hydraulic hammer.

Pull Cap - Threaded, hardened-steel top cap that attaches to the top of the probe rod; used when retracting the probe rods.

Extruder Rack and Piston - A device used in conjunction with the Geoprobe to force soil sample volume out of the sample tube.

Screen Point Groundwater Sampler - A groundwater sampling device designed for use with the Geoprobe consisting of a well screen encased in a perforated stainless steel sleeve.

Mill-Slotted Well Rod and Point - A groundwater sampling device designed for use with the Geoprobe consisting of a Geoprobe probe rod with 15-mil slots, each 5 cm long by 0.05 cm wide (2 inches long x 0.020 inches wide).

Post-Run Tubing System (PRT) - The Geoprobe soil vapor sampling system uses disposable polyethylene or Teflon tubing (inserted into the probe rods at the desired sampling depth) and a vacuum.

Expendable Drive Point - Solid steel point attached to the end of the screen point groundwater sampler and PRT expendable point holder.

Membrane Interface Probe (MIP) - A screening tool with semi-quantitative capabilities acting as an interface between the volatile contaminates in the subsurface and gas phase detectors at the surface. The membrane is placed in a heated block attached to the probe. Heating the block accelerates diffusion of the contaminant through the membrane into the carrier gas, which flows up hole to the detectors.

2.2 Associated Procedures

- SOP 1-2, *Sample Custody*
- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 1-5, *Groundwater Sampling Using a Bailer*
- SOP 1-6, *Water Level Measurements*
- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-3, *Well Development and Purging*
- SOP 4-5, *Field Equipment Decontamination*

2.3 Discussion

The Geoprobe unit consists of a hydraulically-operated hammer device that can be mounted on the back of a van, a pickup truck or track-mounted (Figure 1). The Geoprobe system hydraulically advances small-diameter, hollow rods to the desired sampling depth. The specific type of Geoprobe sampling equipment for soil, soil gas, and groundwater collection is then employed.

The use of Geoprobe technology may be a cost-effective alternative to using conventional drilling techniques for collecting subsurface soil, soil gas, and groundwater samples depending on the site-specific geologic and hydrogeologic conditions and sample requirements. The Geoprobe system is generally used to gather screening-level data. The site-specific sampling plans must consider such factors as soil types, presence of cobbles, depth to groundwater, quantity and depth of samples, site access and topography, data quality objectives (DQOs), analytical requirements, and waste handling and disposal requirements before selecting the use of the Geoprobe.

Advantages of using the Geoprobe Systems include:

- Areas usually considered inaccessible by drill rigs because of overhead wires, steep slopes, size constraints, etc., may be accessed with a van-, pickup truck-, or track-mounted Geoprobe.
- Investigation-derived wastes such as soil cuttings and purge water are minimized with the Geoprobe due to its small diameter rods and its displacement of soil horizontally, not vertically.
- Pneumatic slug testing is also an option when using the SP16 groundwater sampler. Analyses of these tests will yield hydraulic conductivity values for the surrounding aquifer materials.

A Geoprobe membrane interface probe (MIP) and integrated electrical conductivity (EC) dipole combination can be deployed with direct push methods to discriminate variation in grain size and volatile organic contaminants (VOCs). As a result, lithologic changes and distribution of contaminants (chlorinated and nonchlorinated) can be determined in the subsurface.

Cost savings over conventional drilling techniques may be realized. The Geoprobe is rented/leased on a daily, weekly, or monthly basis for a fixed price as opposed to drilling subcontractors who are generally compensated based on the footage drilled. For shallow probing, the Geoprobe may be hand- operated by field personnel rather than subcontractors. A cost evaluation based on project-specific requirements and site conditions shall be conducted to determine the most cost-effective method for a particular project.

Two people are required to operate the Geoprobe and conduct sampling and recordkeeping activities. Safety considerations shall be addressed when operating the Geoprobe. A safety hazard is present whenever the Geoprobe is operated. The hydraulic system operates with a fluid pressure of over 2,000 pounds per square inch (psi). A leaking hose may produce a stream of hydraulic fluid with sufficient pressure to penetrate skin. Therefore, periodic checks of the hydraulic lines and hoses shall be conducted to ensure they are in good condition and connections are tight. Do not attempt to repair or tighten hoses with the engine running and the system under pressure. Use paper or cardboard to check for leaks.

3.0 General Responsibilities

Field Team Leader (FTL) - The field team leader (FTL) is responsible for ensuring that sampling efforts are conducted in accordance with this procedure, associated SOPs, and the site-specific plans.

Sampling Personnel - Field team members are responsible for conducting Geoprobe sampling events in accordance with this procedure, all associated SOPs, and requirements as described in the site-specific plans.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

General

- Site-specific plans
- Field logbook, chain-of-custody forms, other forms for documenting sample shipment
- Indelible black or blue ink pens and markers
- Sample containers with labels and preservatives
- Insulated coolers
- Bagged ice or “blue ice”
- Plastic zip-top bags
- Waterproof sealing tape
- Temperature, conductivity, pH, dissolved oxygen, and turbidity meters (with clean beakers or other appropriate containers), as required by the site-specific plans
- Monitoring/screening instruments as required by the site-specific health and safety plan or sampling plan
- Decontamination supplies, as required by SOP 4-5
- Personal protective equipment (PPE), as required by the site-specific health and safety plan (at a minimum, hard hat, steel-toed shoes, safety glasses, and hearing protection are required)
- Latex or appropriate gloves
- Geoprobe rig (van, truck, or track-mounted) with the following:
  - Probe rods (1 to 5-foot [ft]) lengths
  - Extension rods (1 to 5-ft) lengths, couplers, and handle
  - Piston stop-pins (two each per rig, minimum)
  - Drive caps and pull caps (two each per rig, minimum)
  - Carbide-tipped drill bit for working in concrete- or asphalt-covered areas
  - O-rings

Geoprobe Soil Sampling Equipment

- Extruder rack and piston (i.e., if soil is to be extruded into a sample container)
- Assembled DT or MC soil samplers (The stainless steel sample tubes or PVC liners from these samplers may be individually sealed and shipped directly to the laboratory, as indicated in site-specific plans)
- Refer to the Geoprobe Systems website for specific parts and accessories for each sampler

Geoprobe Soil Gas Sampling Equipment

- Expendable drive points (one each per sample location, plus spares)
- Extension rod ram
- 10 millimeter (mm) (3/8-inch) polyethylene (Teflon-lined) tubing and PRT adapter
- Vacuum or sampling system
- Syringe
- PRT adapter
- PRT expendable point holder

Geoprobe Groundwater Sampling Equipment

- Expendable drive points (one each per sample location, plus spares)
- Mill-slotted well point or screen point groundwater sampler assemblies
- Extension rod ram
- 10-mm (3/8-inch) polyethylene or Teflon-lined tubing
- Check valves (if using Waterra system)
- Peristaltic pump (limited to shallow depths)
- Mini-bailer (with thin nylon line)

5.0 Procedures

Procedures common to all three sampling methods are discussed below.

Before sampling:

- Review site-specific health and safety plan and project plans before initiating sampling activity.
- Arrange utility clearance.
- Decontaminate all Geoprobe equipment according to SOP 4-5, *Field Equipment Decontamination*.
- Don the appropriate PPE as dictated by the site-specific health and safety plan.
- If the sampling site is in a concrete- or asphalt-covered area, drill a hole using the rotary function and a specially designed carbide-tipped drill bit of the diameter appropriate for the selected sampler diameter. Otherwise, the area needs to be cleared of heavy underbrush and immediate overhead obstructions.

After sampling is completed:

- Thread the pull cap onto the top probe rod and retract the probe rods.
- Seal the borehole with sand, neat cement, or bentonite grout, if necessary.
- Record all appropriate data in the field logbook and on the chain-of-custody forms as outlined in SOP 4-1, *Field Logbook Content and Control* and SOP 2-1, *Packaging and Shipping Environmental Samples*.
- Decontaminate the sampling equipment according to SOP 4-5, *Field Equipment Decontamination*.

5.1 Soil Sampling

Assembly

1. Assemble the sampling device as follows:

    - Screw the cutting shoe to the bottom end of the sample tube, unless using standard probe drive sampler which has a built-in cutting edge.
    - Screw the piston tip onto the piston rod.

- Screw the drive head onto the top end of the sample tube.
- If using Teflon liner, insert liner into sample tube.
- Slide the piston rod into the sample tube, leaving the piston tip sticking out of the bottom end of the sample tube.
- Screw the piston stop-pin onto the top end of the piston rod in a counter-clockwise direction.

2. Attach the assembled sampler onto the leading probe rod. A 30-cm (12-inch) probe rod is recommended to start the DT and MC samplers.

Probing

3. Thread the drive cap onto the top of the probe rod and advance the sampler. Replace the 30-cm (12-inch) rod with rod appropriate for the stroke of the rig as soon as the top of the sampler is driven to within 15 cm (6 inches) of the ground surface.

4. Advance the sampler to the interval to be sampled using the hydraulic hammer. Add additional probe rods as necessary to reach the specified sampling depth.

Stop-Pin Removal

5. Move the probe unit back from the top of the probe rods and remove the drive cap.

6. Lower the extension rods into the inside diameter of the probe rods using extension rod couplers to join the extension rods.

7. Attach the extension rod handle to the top extension rod and rotate the handle clockwise until the leading extension rod is screwed into the piston stop-pin. Continue to rotate the handle clockwise until the stop-pin disengages from the drive head.

8. Remove the extension rods and attached piston stop-pin from the probe rods.

Interval Sampling

9. Replace the drive cap, mark the top probe rod with a marker or tape at a distance above the ground equal to the length of the sample tube.

10. Advance the probe rods using the hydraulic hammer the length of the sample tube.

11. Replace the drive cap with the pull cap and retract the probe rod(s). Secure the rod(s) with a clamp or by hand during removal so they do not fall back down the resulting borehole.

12. Detach the sampler from the lead probe rod, verifying that sufficient sample volume was recovered (Note: The length of sample contained within the tube is approximately equal to the length of exposed piston rod).

13. Disassemble the sampler. If the sample is to be analyzed for VOCs, then the sample tube or liner shall be sealed immediately by placing a Teflon septa over the ends and covering them with plastic caps.

14. If samples do not require VOC analysis, they may be extruded from the sampler and transferred to the sample jars specified in the site-specific plans or SOP 2-1, Packaging and Shipping Environmental Samples. Samples can be extruded by one of two methods:

    - Using the Geoprobe rig and the extruder rack (Figure 2), position the extruder rack on the foot of the Geoprobe derrick; insert the sample tube into the extruder rack with cutting end up; and position the extruder piston, pushing the sample out of the sample tube using the “probe” function. Catch the sample as it exits beneath the extruder in a sample jar or stainless steel mixing bowl.

    Note: Samples to be collected for VOCs will be collected directly from the sample tube into the sample jars.

- Lightly tap the side of the sample tube with a hammer while also lightly pushing the Piston Rod.

15. Label the sample liner or sample jars as required, securing the label by covering it with a piece of clear, waterproof tape.

16. Homogenize the sample in a stainless steel bowl with a stainless steel spoon or spatula. Transfer the sample from the bowl to the sample container.

17. Clean the outside of the sample jars and place individual samples into sealable bags and seal the closure.

18. Place samples in a cooler containing ice according to SOP 2-1, Packaging and Shipping Environmental Samples.

Continuous Sampling
The DT21/22, DT325/35, DT45, DT60, MC5, and MC7 are direct push systems for collecting continuous core samples of unconsolidated materials from within sealed probe rods. Samples are collected and retrieved within a liner that is threaded onto the leading end of a string of Geoprobe rods inserted to the bottom of the outer casing.

5.2 Soil Gas Sampling

Assembly

1. Assemble the sampling device as follows (Figure 3):

   - Test fit the adapter with the PRT expendable point holder or retractable point holder to ensure that threads are compatible and fit together smoothly.
   - Attach the PRT adapter to flexible tubing equal in length to the depth of sampling, with some additional tubing for sampling activities.
   - Secure the PRT adapter with a length of electrical tape and check the condition of the O-ring attached to the end of the PRT adapter.
   - Screw the PRT expendable point holder into the bottom of the lead probe rod.
   - Attach an expendable drive point to the bottom of the PRT expendable point holder.

2. Attach the assembled sampler onto the leading probe rod. A 30-cm (12-inch) probe rod is recommended to start the standard and large bore samplers.

Probing

3. Thread the drive cap onto the top of the probe rod and advance the sampler. Replace the 30-cm (12-inch) rod with rod appropriate for the stroke of the rig as soon as the top of the sampler is driven to within 15 cm (6 inches) of the ground surface.

4. Advance the sampler to 30 cm (1 ft) past the interval to be sampled using the hydraulic hammer. Add additional probe rods as necessary to reach the specified sampling depth.

5. Connect the out-of-hole tubing to a vacuum or sampling system. A short section of inert silicon tubing may be connected to the end of the out-of-hole tubing so that a sample can be collected with a glass gas chromatograph (GC) syringe.

6. Start the vacuum or sampling system and allow the system to operate for 2 to 3 minutes to ensure that a sufficient volume of air has been run through the tubing. Document the depth, vacuum pressure, and purge duration in logbook.

   Note: Make sure the vacuum evacuation pump is able to pull vapors from the formation. Excessive vacuum may occur in clay/clayey units resulting in insufficient sample volume.

7. Collect sample using the method specified in the site-specific plan.

8. Label all sample containers as required, securing the label by covering it with a piece of clear, waterproof tape.

9. Remove the tubing from the probe rods. Dispose of the tubing or set it aside for decontamination.

10. Remove probe rod(s) from hole. Leave tubing in place for longer term monitoring.

Other Vapor Sampling Options

1. The Vacuum/Volume System is a combined pump and tank system. In typical operation this tank is pumped down with the system vacuum pump. The vacuum pump is then shut off and the tank is opened to the soil gas sampling train. By measuring the change in pressure in the tank, the soil gas sampling can be performed at the same pressure at each sampling point, improving the comparability of samples.

2. Vapor Implants are permanent sampling points inserted into the subsurface for ongoing monitoring. Implants are stainless steel screens that can be inserted down the bore of a probe rod and anchored at depth. As probe rods are removed from the hole, the implant and associated tubing remain firmly anchored at the bottom and available for ongoing sampling.

5.3 Groundwater Sampling

Assembly

1. Assemble the screen point groundwater sampler as shown on Figure 4 and described below (see Geoprobe Systems website, Groundwater Assessment Tools):

   - Push the screen insert and plug into the screen sleeve from the bottom. The bottom end has one drain hole.
   - Push the screen connector over the top end of the screen sleeve and push the screen connector pin into place. The pin must be held in place as it has a loose fit.
   - Insert the screen sleeve, screen connector first, into one end of the sampler sheath.
   - Slide the drive point seat over the end of the screen assembly that protrudes from the sampler sheath. Thread it in until tight using a 22-mm (7/8-inch) wrench.
   - Push the screen assembly just far enough into the sampler sheath that an expendable drive point can be pushed into place in the drive seat.
   - Screw the groundwater drive head with the O-ring end first into the open end of the sampler sheath.
   - O-rings are installed at various critical places in the sampler assembly. Ensure that all O-rings have not been worn and that the connections made at O-ring locations are tight.
   - The mill-slotted well point does not need any assembly.

2. Attach the mill-slotted well point, or screen point groundwater sampler, onto the leading probe rod. A 30-cm (12-inch) probe rod is recommended to start either groundwater sampler.

Probing

3. Thread the drive cap onto the top of the probe rod and advance the sampler using either the hydraulic hammer or hydraulic probe mechanism on the Geoprobe rig. Replace the 30-cm (12-inch) rod with rod appropriate for the stroke of the rig as soon as the top of the sampler is driven to within 15 cm (6 inches) of the ground surface.

4. Advance the sampler to the interval to be sampled using the hydraulic hammer. Add additional probe rods as necessary to reach the specified sampling depth.

Developing and Sampling

6. Move the probe unit back from the top of the probe rods and remove the drive cap.

7. The next step varies depending on the type of sampler being used:

   - Mill-slotted well point - measure and record the water level, allowing time for the water level to reach equilibrium.

   - Screen point groundwater sampler - attach the pull cap to the top probe rod, retract the probe rods approximately 60 cm (2 ft), push the screen into the formation using extension rods fitted with a ram, remove extension rods from the probe rods, and measure and record the water level, allowing time for the water level to reach equilibrium.

8. Surging and purging shall be conducted throughout the length of the exposed screen to properly develop the well point before sampling.

9. Label all sample containers as required, securing the label by covering it with a piece of clear, waterproof tape.

10. Collect groundwater samples using one of three methods (as outlined in site-specific plans) described below:

    - Collect sample from the inside diameter of the probe rods using a decontaminated mini-bailer. Follow SOP 1-5, Groundwater Sampling Using a Bailer.
    - Collect sample using a peristaltic pump and flexible tubing system.
    - Collect sample using a check valve (Waterra-type valve) attached to the bottom of 10-mm (3/8-inch) diameter tubing. The tubing is lowered into the probe rods below the top of the water table, check valve-end first. Water sample is collected through the tubing by rapidly oscillating the tubing up and down creating an inertial pump.

11. Clean the outside of the sample containers and place individual samples into sealable bags and seal closure.

12. Place samples in a cooler containing ice according to SOP 2-1, Packaging and Shipping Environmental Samples.

5.4 Pneumatic Slug Testing

Assembly

1. Assemble the screen point groundwater sampler and the pneumatic manifold assembly as shown on Figure 5 (see Geoprobe Systems Instructional Bulletin No. MK3181).

2. Be sure to accurately document all well construction parameters and site geologic information:

   - Effective screen length (includes sand or filter pack)
   - Height of water column in well
   - Radius of filter pack
   - Radius of transducer and cable (for wells 1-inch diameter or less)
   - Depth of transducer below static water level
   - Saturated thickness of the aquifer
   - True screen length
   - Screen radius
   - Casing radius
   - Static water level from a fixed reference point
   - Total depth of well from a fixed reference point
   - Initial head change

3. Once the pneumatic head is in place, a vented pressure transducer assembly is installed. The transducer itself is inserted through the port on top of the pneumatic head and lowered into the well about 2-feet below the static water level and off the bottom of the well.

4. Let the transducer equilibrate to ambient groundwater temperature and then zero out the transducer.

Testing

5. Set up slug test data acquisition software and select preferred options (refer to Instructional Bulletin No. MK3087).

6. Close inlet and release valves and close the pressure regulator on the manifold assembly.

7. Adjust zero setting on pressure gauge, if needed.

8. Operate foot pump to pressurize supply hose to approximately 30 to 40 psi.

9. Open inlet valve on pneumatic head.

10. Slowly open the pressure regulator. From the fully closed position it takes about five revolutions to begin opening the regulator. Observe the pressure gauge on the pneumatic head (scaled as inches of water). Let the pressure in the well head rise slowly to a few inches above the level desired for testing (e.g. if you want to initiate the slug test with H_0 of 10 inches let the gauge rise to about 12 inches).

11. Quickly close the inlet valve and allow the pressure observed from the transducer in the well head to return to equilibrium and stabilize. Record the stabilized gauge pressure. The readings shall return to the levels noted before pressurization was started.

12. Leak test the fittings on the pneumatic head and connection to the rods with a soapy fluid. Tighten fittings if necessary and retest. It is preferable to locate and correct any slow leaks before continuing with the slug test.

13. Once the transducer readout is back to equilibrium and stable, the slug test is ready to initiate. The slug test is initiated by opening the release valve as quickly as possible.

14. A very rapid initial drop in the transducer readout (head) shall be observed as the air pressure is released. Then the rise or recovery of the water level to the pre-test equilibrium level (baseline) will occur. Once the water level has returned to the pre-test level and is stable, the slug test is complete.

15. It is strongly recommended that at least three slug tests are run using different initial head values (H_0) to verify appropriate well performance and development. If there is significant deviation between the repeat tests, additional development of the well or sampler may be necessary.

Geoprobe Systems has designed a simple user-friendly software package and data logger that allows acquisition and filing of pneumatic transducer data on a laptop computer. The data files are stored in ASCII format for easy export to spreadsheet and data analysis programs. The selection and application of the appropriate data analysis methods is beyond the scope of this SOP for field techniques.

6.0 Restrictions/Limitations

Smaller diameter Geoprobe sampling systems are not designed for collecting large sample volumes, thereby limiting the number of analytical parameters. Soil sample recovery will be poor in soils. Production rates will vary substantially depending on sampling depths/intervals, subsurface conditions, and the platform used. However, a minimum of between 10 and 15 samples per day can be expected in most situations.

The most efficient sampling depth is limited by the geologic and hydrogeologic conditions. Practical, efficient sampling depths shall be limited to approximately 6 meters (20 feet) under most conditions. However, sampling depths in excess of 30 meters (100 feet) have been achieved in unconsolidated, homogeneous sandy soils using heavy duty platforms and MC5 tools. Attainable depths will be greatly reduced in more consolidated and indurated formations and in soils with gravel and cobbles.

The presence of gravel and cobbles in soils will likely damage soil sampling tubes and possibly probe rods, couplers, stop-pins, and other probing equipment. A sufficient supply of replaceable equipment shall be kept on site in the event of damage or breakdowns. Replacement may be at the project's - not the subcontractor's - expense. The Geoprobe Systems website shall be accessible onsite; Geoprobe Systems provides overnight deliveries. MIP tooling is expensive and more fragile than standard soil sampling tooling and may not be appropriate in dense formations or formations with substantial amounts of gravel and/or cobbles.

Before conducting the Geoprobe sampling event, underground utilities and structures must be demarcated on the ground surface. The local utility companies must be notified at least 72 hours before the scheduled sampling event to allow sufficient time to locate and mark the utility lines. The selected sampling location shall be a safe distance from the demarcated utility. In some cases, records regarding utility locations may not exist. In any event, a good practice is to slowly push the probe rods the first few feet (rather than hammering) to ensure that no utilities, underground storage tanks, or other subsurface structures are present.

7.0 References

Department of Defense. *Environmental Field Sampling Handbook, Revision 1*. April 2013 or current revision.

Geoprobe® Systems. 2015. *Standard Operating Procedure.* < http://geoprobe.com/literature/...>.

U.S. Environmental Protection Agency, Region IV. *Science and Ecosystem Support Division, Operating Procedure SESDPROC-300-R3*. August 2014 or current revision.

Figure 1
Geoprobe® Unit

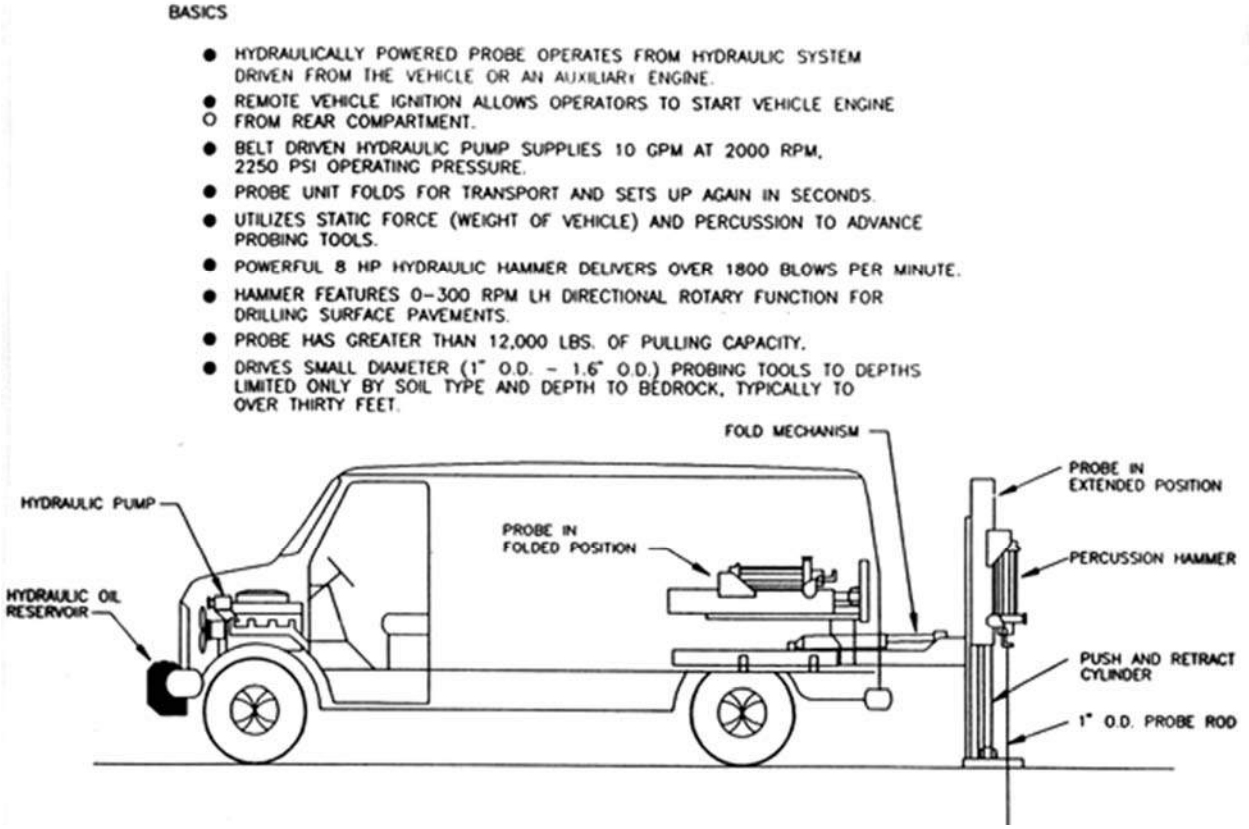


Figure 2
Sample Extruder Rack

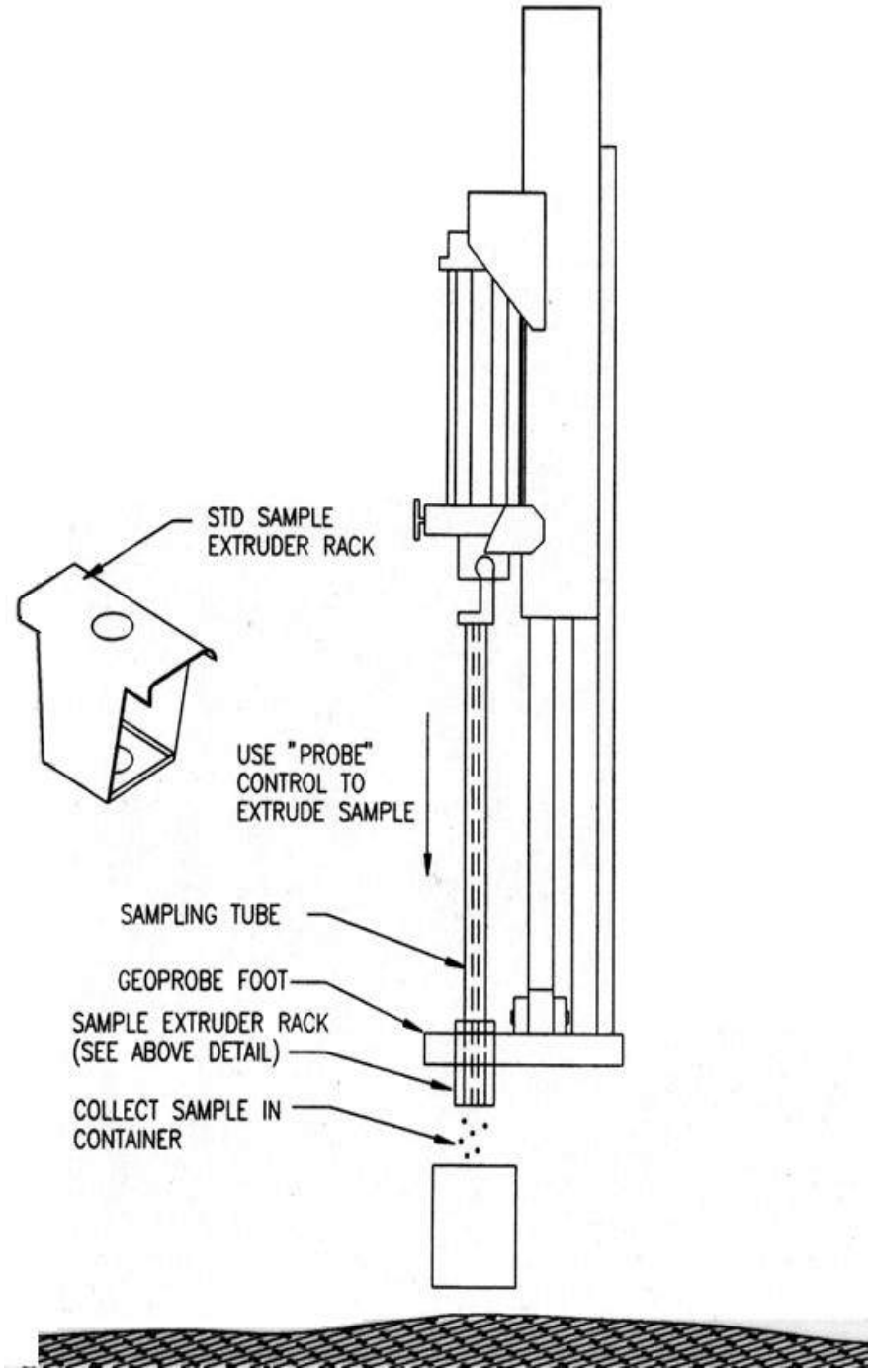


Figure 3
PRT Soil Gas Sampling System

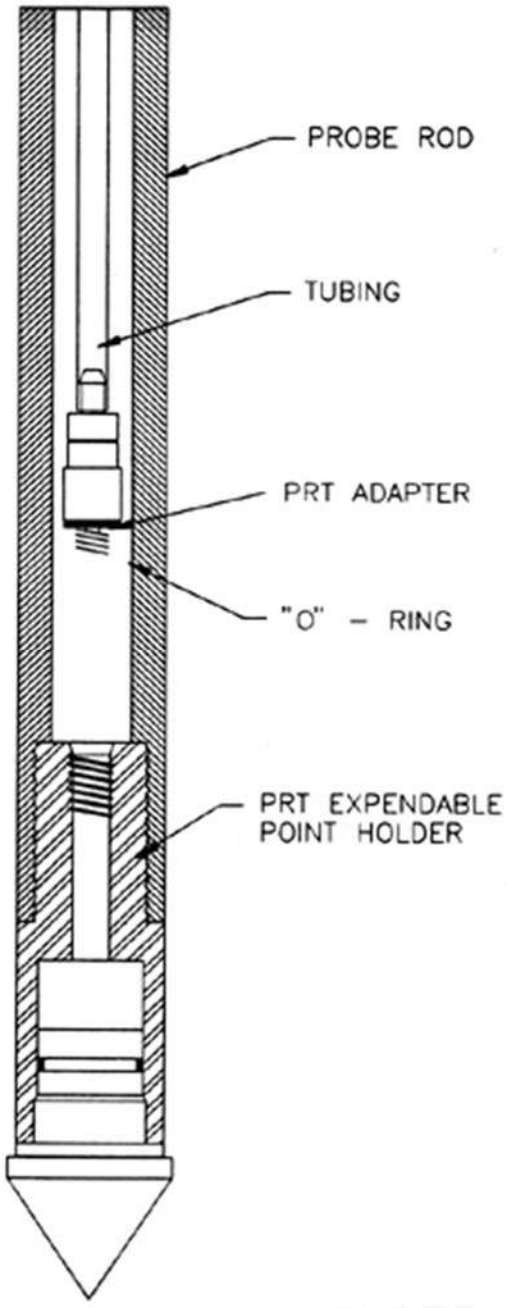


Figure 4
Groundwater Sampling

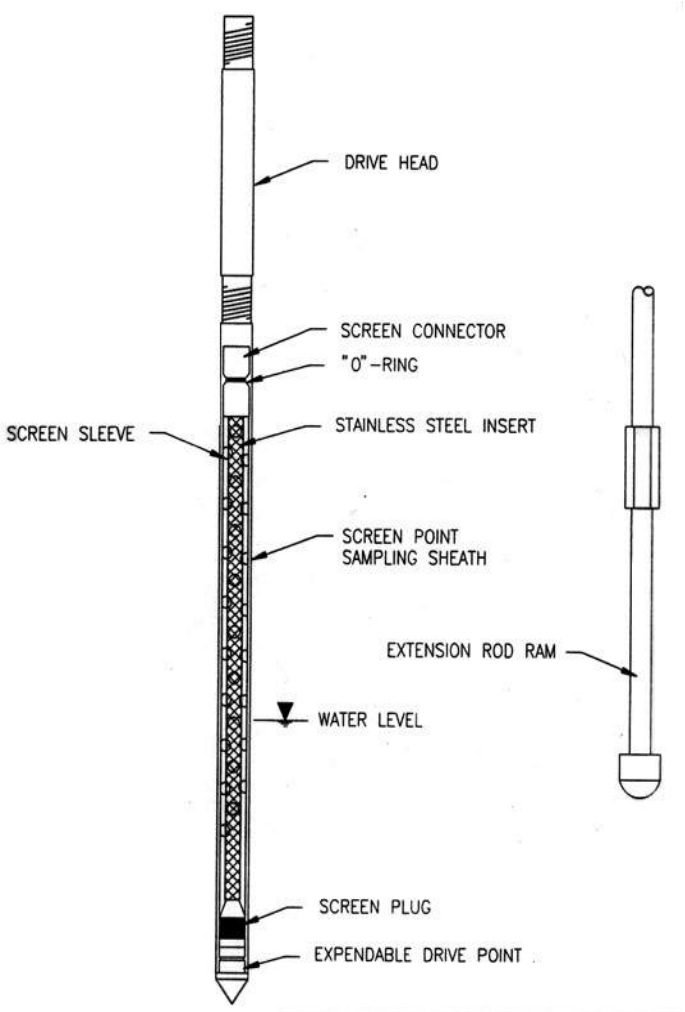
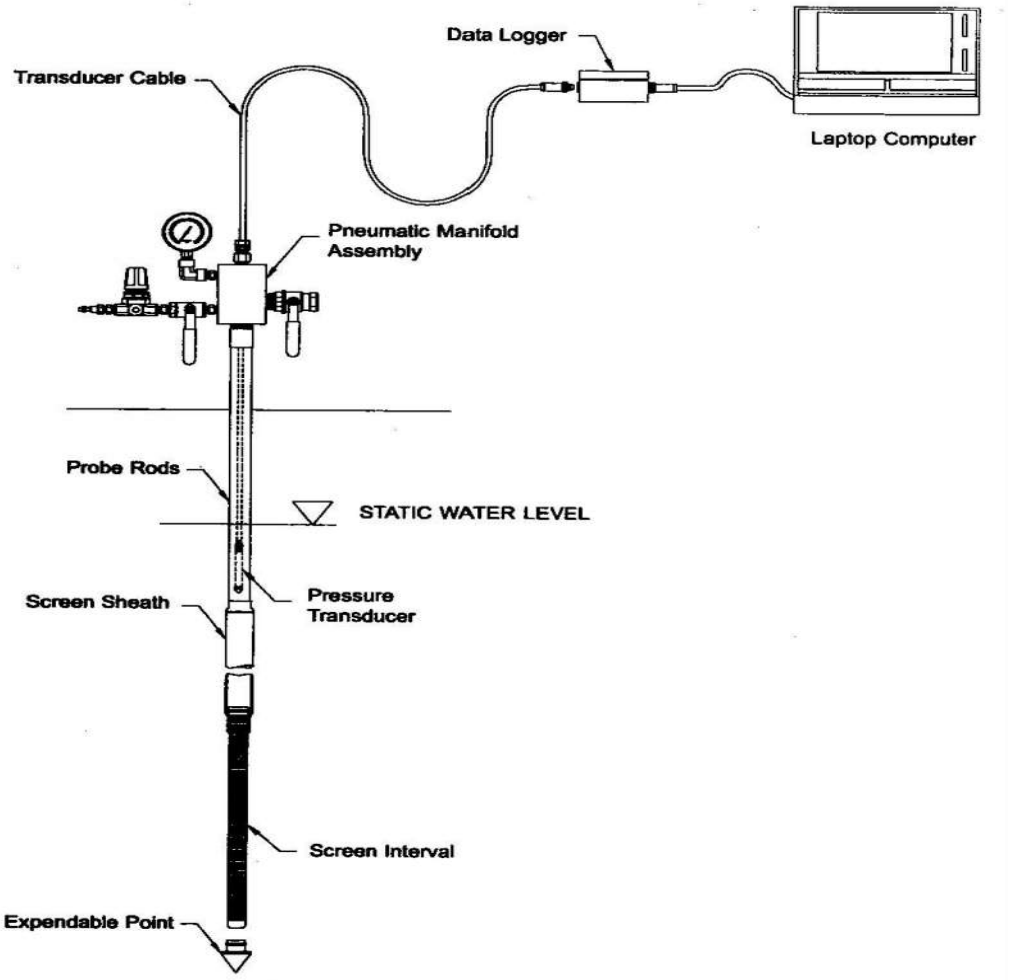


Figure 5
Pneumatic Slug Testing with an SP15/16 Groundwater Sampler



The

Topographic Survey	SOP 3-2 Revision: 8 Date: February 2015

Prepared: Jonell Mullins *Signature* **Technical Review:** Todd Bragdon

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to provide guidance for a site topographic survey. The survey will produce a base map of the area under study, showing topographic and site-specific features. Also, the base map will incorporate site-specific grid system coordinates, if appropriate, to show sample and exploration location, monitoring wells, test pits, and any other features required by the scope of work.

2.0 Background

A site-specific grid system may be established at the area under study to coordinate the collection of samples. The topographic survey will establish the coordinates for the grid and facilitate the transposition of the grid and sample locations from the field to the topographic base map. At areas where a grid system is not used, sample and exploration locations will be marked by the field team using appropriate markers such as stakes, nails, flagging, or paint. The base map will also locate site-specific planimetric details such as significant manmade and geographic features via the survey. In developing the topographic survey scope of work (SOW), considerations for the initial base map development should also include future project needs such as engineering design.

The scale for the base maps will vary based on the size of the area under study, but a suitable scale will be selected that clearly shows map features and sample locations. The base maps will be at a scale appropriate for the intended use. Areas with significant detail requirements will be shown in scale that ranges from 1 inch equals 10 feet to 1 inch equals 40 feet. Areas with less detail requirements will be shown in smaller scale such as 1 inch equals 100 feet or 200 feet. Topography will be shown with 1- or 2-foot contour intervals. However, the contour interval shall clearly identify the variation in topography to the degree necessary for the work to be performed. For example, gently sloping areas may require a smaller contour interval (i.e., 1 foot between contour lines) to reveal more subtle topographic variations. Similarly, steeply sloping areas may require larger contour intervals to legibly depict the topography. Index contours shall be indicated at elevations that are multiples of five times the contour interval.

If appropriate, aerial photographs (i.e., orthorectified imagery) may be used to assist in the development of the topographic base maps. Existing or new photographs can be used for this purpose. In areas with deciduous trees, new photographs shall be taken during late fall or winter when the leaves are off the trees and better ground surface image can be achieved. The scale of the aerial photographs shall provide sufficient detail for developing the topographic base map.

3.0 General Responsibilities

Project Manager - The project manager is responsible for ensuring that the topographic survey is completed in accordance with the project requirements.

Field Team Leader - The field team leader is responsible for developing the survey scope of work and ensuring that the topographic survey is coordinated properly with the grid system (if used) and the sampling points, so that the base map produced is a true representation of the field locations.

***Note*:** Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-specific/project-specific quality assurance plan.

4.0 Required Equipment

The required equipment for a topographic survey shall be provided by the selected surveyor. All equipment proposed by the surveyor shall be submitted to CDM Smith for approval before initiating the topographic survey work.

The selected surveyor must be licensed professional surveyor (PLS) in the state in which the survey is conducted.

For topographic surveys conducted at hazardous waste sites, all surveyor personnel who work onsite will be 40-hour health and safety trained per OSHA requirements for hazardous waste sites (29 CFR 1910.120), unless approved differently by the corporate health and safety manager.

All final drawings and maps must be signed and sealed by the licensed professional land surveyor.

5.0 Procedures

1. A site visit may be conducted before submitting the bid proposal. A kickoff meeting shall be held between the selected surveyor and CDM Smith's project manager to discuss the specific requirements of the scope of work.

2. The surveyor shall be responsible for executing the work, including deed search if required to support development of a legal description of the site or to establish property ownership boundaries.

3. The surveyor shall develop and implement a site-specific health and safety plan according to the requirements specified in the subcontract between CDM Smith and the surveyor.

4. To the extent practical, the work shall be performed in the presence of an authorized representative(s) of CDM Smith. CDM Smith will interpret and clarify the specifications and will answer all questions in connection therewith.

5. The CDM Smith field team leader will be responsible for ensuring that appropriate calibration procedures are performed and documented by the surveyor. Calibration procedures shall be consistent with the data quality objectives for the survey and with the equipment manufacturers' requirements.

6. The surveyor shall establish at least one primary horizontal control monument and one vertical benchmark, as established by the United States Coastal and Geodetic Survey (USC&GS) or equivalent authority. Additional monuments may be established by the surveyor.

7. Local benchmarks will be established at least every 500 feet or closer, if warranted by site conditions, to tie the basic control points together. Where required, established horizontal and vertical data, such as state planar coordinate systems and the national geodetic vertical datum of NAVD 88 or subsequent corrections and/or revisions, shall be used to tie the survey data to the national network.

8. Temporary monuments will be set as necessary to perform the surveying. They may be wood, metal, or otherwise marked on facilities such as sidewalks, paved streets, curbs, etc. All monuments shall be described in the field notes and marked on site maps for future reference.

9. If appropriate, the surveyor shall be encouraged to use technologies such as Global Positioning System (GPS) that will meet the accuracy requirements but that may be more flexible and efficient than traditional techniques. All geodetic control work shall conform to either the Standards and Specifications for Geodetic Control networks, Federal Geodetic Control Subcommittee or NAVSTAR Global Positioning System Surveying, U. S. Army Corps of Engineers, for third order Class II control surveys. Short traverses, less than 1 mile, may use generally accepted fourth order techniques (including vertical angles for elevations) that will provide the spatial accuracy required. Angles shall be doubled and redoubled if the mean of the doubled angle differs from the first angle by more than 10 seconds. Length measurements shall be made with a calibrated tape corrected for temperature and tension or with Electronic Distance Measuring (EDM) equipment corrected for variation of the index of refraction.

10. The CDM Smith field team leader will review the draft map to ensure that all sampling and exploration locations, grid coordinates, and other appropriate features are located by the surveyor. The surveyor will record all field survey information in a field logbook; a copy of the logbook shall be provided to CDM Smith with the submittal of the topographic map.

11. A working drawing of the base map will be field checked and corrected by the surveyor as necessary. The completed topographic base map shall be plotted on Mylar® or other suitable drafting film, as directed by the CDM Smith project manager. All survey and topographical data will be in digital format, compatible with the latest version of AutoCAD, ArcView/ArcInfo, DXF, or geographic information system (GIS) export format may also be acceptable. The specific format of the data to be provided to CDM Smith will be specified in the SOW. It is recommended that a review of CDM Smith client requirements be completed to determine the appropriate data format. Sufficient documentation of the digital information shall be provided to explain the data. For clarity, the surveyor will prepare the base map with groups of features on separate layers in the AutoCAD files. The CDM Smith project manager shall designate which features will be placed on the separate layers. Tick marks indicating the latitude and longitude in the state that the work is performed shall be provided on the base map. The project manager will be responsible for ensuring that the topographic base map and digital information is completed according to CDM Smith's drafting standards for the project; it is recommended that CDM Smith's drafting standards be included in the SOW to ensure clear communication of project requirements.

12. In the event that aerial photographs are used, the surveyor shall field edit and statistically test the aerial topographic mapping of the site base map for conformance with the horizontal and vertical components of the National Map Accuracy Standards. The surveyor shall run random baselines throughout the site (minimum of four) to verify that less than 10 percent of horizontal and/or vertical locations exceed the values determined in the National Map Accuracy Standards. If more than 10 percent of the locations exceed the values in the National Map Accuracy Standards, then the surveyor will notify CDM Smith. The project manager will be responsible for ensuring that the orthorectified aerial photographs files are transferred electronically to CDM Smith along with other required deliverables in the SOW.

13. Stereo map compilation by stereo photogrammetric methods will be accomplished through the use of approved stereophotogrammetric instruments using professionally recognized plotting ratios for each type of instrument. Fully trained and experienced photogrammetrists will be employed to complete stereomap compilation.

14. For broad area high precision topographic mapping, digital elevation/terrain model (DTM) compilation using light detection and ranging (LiDAR) technologies is becoming more common. This method can be an efficient and effective tool for increasing engineering production at all levels. However, the error budget for a given LiDAR mapping system is dependent on the accuracy of its core subsystems (i.e., the laser rangefinder, the GPS position solution, and the inertial measurement unit [IMU]). System engineers need to balance each subsystem contribution against desired system performance (Shrestha et al. 2000). The project manager will be responsible for ensuring that the DTM files are transferred electronically to CDM Smith along with other required deliverables in the SOW. DTM files can be utilized by AutoCAD Civil 3D for future modeling of the site.

15. The surveyor shall establish and maintain a quality control program to ensure that the survey is performed within acceptable limits. At a minimum, the surveyor will:

    - Check all equipment, including compasses, transits, and levels, for accuracy and maintain records of such checks. The surveyor will make records of these checks available to CDM Smith on request.
    - Maintain and submit copies of all survey field notes.
    - Field notes for each surveying activity will be kept in bound books dedicated exclusively to this project. Each book will have a table of contents. Each page of field notes shall be numbered, dated, and show the initials of all crewmembers. Black waterproof ballpoint pens will be used. Erasing is not acceptable. All errors will be crossed out with a single line and the correct data entered adjacent to the error. The crossed out and corrected data will be initialed by the party marking field notes.

16. Permits:
    - The surveyor shall be responsible for obtaining any federal, state, and local permits that may be required and to perform and complete the ground surveys at the site.
    - The surveyor shall not perform any work until permits (if required) are obtained.
    - The surveyor shall provide separate copies of all permits to CDM Smith before performing any onsite activities.

6.0 Restrictions/Limitations

The horizontal positions are to be surveyed within 1/10 of a foot, relative to the datum coordinate system. The vertical elevations of monitoring wells, piezometers, and staff gauges are to be surveyed within 1/100 of a foot (0.01 foot), relative to the local benchmarks. The vertical elevations of all other sampling points are to be surveyed within 1/10 of a foot, relative to the local benchmarks.

7.0 References

U. S. Department of Commerce, National Geodetic Survey (see http://www.ngs.noaa.gov).

Moffitt, F.H. and Bouchard, H. 1982. *SURVEYING* (10th ed.), Harper and Row, Publishers, New York.

Shrestha, R. L. et al. 2007_Version 1.2. *Airborne Laser Swath Mapping: Accuracy Assessment for Surveying and Mapping Applications*. University of Florida (see http://ncalm.berkeley.edu/reports/NCALM_WhitePaper_v1.2.pdf).

Geophysical Logging, Calibration, and Quality Control
(Includes Potential Radioactive Sites)

SOP 3-4
Revision: 7
Date: February 2015

Approved: Signature **Technical Review:** Michael Valentino

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to provide guidelines and define requirements for the generation of quantifiable geophysical logs of selected boreholes. The procedure defines use of a logging systems check, calibration and maintenance check, and well maintenance data documentation.

2.0 Background

Geophysical logging can be used to interpret the physical characteristics surrounding a borehole. These characteristics include the lithology, geometry, resistivity, formation resistivity factor, bulk density, porosity, permeability, structural integrity, and moisture content. Logging can also be used to evaluate well integrity and characterize vertical groundwater flow and groundwater quality within the water column. Interpretations from tool response can be used in a more quantitative fashion when the instruments used during a specific logging process are accurately and properly calibrated and operated by trained personnel. To provide consistently reliable data, a logging operator needs to ensure the following: proper tool calibration or standardization maintenance checks, a logging systems check, complete and well-maintained data documentation, and identification and protection against potential hazards.

2.1 Associated Procedures

- SOP 4-1, *Field Logbook Content and Control*

3.0 General Responsibilities

Site Manager - The site manager translates client requirements into technical direction of the project. The site manager plans and directs the overall project; sets technical criteria; reviews and approves technical progress; defines or approves what logs and tools are to be used after consultation with the field team leader; and considers the objectives of the project, the lithology surrounding the borehole, and the borehole conditions.

Field Team Leader - The field team leader (FTL) provides onsite supervision of the borehole logging program, administers the logging subcontractor operation, and ensures that this SOP is properly followed at all times. The FTL confers with the site manager and the logging subcontractor on what logs and tools are to be used and maintains the field logbook in accordance with SOP 4-1, Field Logbook Content and Control. The field team leader provides the logging subcontractor with a unique, site-specific document control or ID number for use in identifying each individual borehole or well, provides copies of checklists pertinent to the operation, and provides copies of borehole or well construction details.

Logging Subcontractor - The logging subcontractor provides equipment appropriate to the task as described in the project statement of work, provides appropriately trained and qualified personnel, and responds to administration of the FTL. The logging subcontractor ensures proper tool calibration or standardization maintenance checks, a logging systems check, complete and well-maintained data documentation, and identification and protection against potential hazards. The logging subcontractor ensures that logging subcontractor personnel read and observe requirements defined in this SOP and provides copies of their company standard and emergency operating procedures for approval by FTL before implementing any logging activities.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

4.0 Required Documentation

- Field logbook
- Appropriate log sheets (boring logs, well completion data sheets, or equivalent)

- File containing:
  a) One copy of the logging contract stating the technical requirements
  b) One approved copy of logging subcontractor SOPs and emergency operating procedures
  c) One copy of the current Nuclear Regulatory Commission (NRC) license listing certifications and approval of the type and activity level of the radioactive sources to be used onsite, if appropriate
  d) One copy of the current leak test (swipe) results of all radioactive sources containers on board the logging vehicle
  e) Physical survey forms indicating activity level at monitoring points adjacent to the radioactive source storage area of the logging vehicle
  f) Documentation of current radiation monitoring instrument calibration
  g) Documentation that all geophysical tools have been inspected and are properly operating
- Site-Specific Checklists (see attached examples)
  a) Health and Safety Checklist (Attachment 1)
  b) QA Geophysical Checklist for Borehole Logging (Attachment 2)
  c) Geophysical Logging Tool Checklist (Attachment 3)
  d) Example Log Heading (Attachment 4)

5.0 Procedures

5.1 Preparation

The site-specific health and safety plan shall be reviewed along with project plans before initiating logging activities. The logging subcontractor and the FTL or designee will confer before field activities regarding the suite of geophysical tools to be used in the operation. The FTL shall define what logs are to be used.

Considering the objectives of the logging program, the choice of tools may be further determined by the lithology surrounding the borehole and the borehole conditions. Prior logs will be used, if available, as aids to determine the appropriateness of the logging suite. These logs will be studied and serve as a baseline for the current logging activity.

Generally, fluid logs are run first within a logging suite with nuclear logs last. In uncased holes, a caliper log shall be run shortly after the fluid log, as it can yield measurements of the borehole rugosity and diameter variations useful in the onsite evaluation of uncompensated logs that may follow. Both are necessary in determining the borehole integrity and appropriateness and suitability of future logs.

The logging subcontractor shall have an approved emergency operating procedure covering all emergencies relating to tool operation, including the retrieval of a logging tool that has been lost down a hole. The logging subcontractor and FTL or designee will cooperate in determining the most suitable approach for retrieval of any tool.

Note: Some sites (such as the U. S. Department of Energy) require advance notification and approval before bringing any radiation source onto the site. This includes radioactive calibration check sources. The FTL will confirm that all site-specific requirements are met before mobilization of the logging equipment.

The following documents, certificates, and inspections will be completed by the logging subcontractor before well logging and shall be in an open, active file during logging activities.

- One copy of the current NRC license listing certifications and approval of the type and activity level of the radioactive sources to be used in the logging program.
- One copy of the current leak test (swipe) results of all radioactive sources containers on board the logging vehicle.
- Physical survey forms indicating activity level at monitoring points adjacent to the radioactive source storage area of the logging vehicle.
- Documentation of current calibration of radiation monitoring instrument.

- Copy of the logging SOP and emergency operating procedures to be followed during logging activities. Emergency procedures shall include provisions for retrieval of lost probes, with physical descriptions and drawings of the probes and head connections available at the well site.
- Documentation that all geophysical tools have been inspected and are properly operating.

Before proceeding to the field site, each geophysical logging tool will be "shop calibrated." The shop calibration (or standardization) is a method of subjecting the energized probe to a known signal level to ascertain the integrity of the logging equipment. These offsite standardization trials will be available for comparison to onsite calibration or standardization, which will be performed before and after each logging tool run. American Petroleum Institute (API) calibration scales are not usually applicable to geophysical logs made in cased holes, and log headings, in such cases, shall not exhibit environmental units or any other nonapplicable scales. Radiation logs, made through steel casing, shall display nonenvironmental "shop calibration" scale units, such as counts per second, counts per minute, or API units.

5.2 Operation

After all required equipment and material have been mobilized onsite and necessary consultations and preparations are completed, the field operations may begin. During field activities, to ensure the safety and quality of operations, a Health and Safety Checklist (Attachment 1) along with a QA Geophysical Checklist for Borehole Logging (Attachment 2) shall be completed.

Once at the site, each piece of logging equipment will go through routine standardization before and after the logging of every borehole using portable standards. All tool responses to each standard will be recorded on each borehole log trace. Personnel responsible for running the geophysical log shall perform the standardization of the logging equipment.

Calibrations and standardization will be recorded digitally.
Mechanical and electrical zero response and full scale settings will be recorded and labeled as such at the beginning of each logging trace.

Before logging any borehole, the FTL or designee will review the most up-to-date information on borehole conditions such as: depth, variations in diameter, lost circulation zones, casing, debris in hole, fluid type, and known contaminants (if any) in the borehole. Each logging procedure will be determined based on assessment of borehole conditions using the most recent and previously run logs (if available).

All probe O-ring seals will be thoroughly inspected to prevent potential water leaks from developing within the instrument. Seals shall be clean and dirt free.

All logging cables need to be kink free. Logging subcontractor personnel shall be prepared to wipe or clean cables before retraction onto the storage drum.

The cable measuring sheave between the winch and well must be pre-calibrated to the currently used cable size. The sheave shall be free of any debris (e.g., dirt, ice, or dry drilling mud).

Cable heads shall be checked for electrical leaks or shorts using a volt- or ohm-meter.

Before the completion of each logging suite, a geophysical checklist will be studied and completed, where appropriate, by the FTL or designee. All checklists will be completed in black indelible ink. The Geophysical Logging Tool Checklist (Attachment 3), to be completed by the logging subcontractor equipment operator, shall indicate the specific logging tools used, order of use, and the combination of tools used concurrently. A preview of the checklist by the logging subcontractor equipment operator will serve, in part, as a reminder of potential problems that may arise during the logging run.

An example log heading, as shown in Attachment 4, shall be used. The analog trace for each log will have all pertinent information (listed

below) written on the trace by the logging subcontractor equipment operator as it occurs. This may include:

- Depths per interval or division of log tracing paper
- Horizontal scale values (written at the start of each log)
- Scale changes (if done during a logging run)
- Pen positions
- Borehole/well identification
- Probe type
- Logging speed
- Tool calibration tails
- Module adjustments
- Digitized record information number

This information will be copied to a formal well log heading at the end of the logging run. Each log heading will give information relating to well and geophysical tool parameters. To interpret the well log as quantitatively as possible, it is imperative that all criteria and data entries within a log heading be filled out by the logging subcontractor equipment operator before the logging tool is removed from the cable. The completed heading will be attached to the well analog record at the well site. If required for the project, the FTL or designee may supply a unique site-specific document control or ID number written on both digital record and the log heading.

Borehole geophysical data stored on the laptop computer should be backed up to a USB drive in the field before leaving each borehole.

It is imperative that the logging operator continually monitor the data output on the laptop monitor to determine any spurious and anomalous conditions that may arise. A review of the geophysical checklist before logging will be done to prompt the logging subcontractor equipment operator of potential problems that might arise. The following briefly summarizes some of the potential problems associated with each logging technique. It shall not be a substitute for thorough training in the nuances and idiosyncrasies of specific instruments, as each tool used will have a unique circumstance that may result in a spurious log.

A. Extraneous Conditions Resulting in Anomalous Logs

The following briefly describes conditions that may give rise to anomalous log traces and shall be watched for by the logging subcontractor equipment operator.

1. Fluid Logging

In newly drilled holes, it is unusual to have chemical or thermal equilibrium between the borehole fluid and surrounding borehole matrix. Consequently, fluid logs generally measure the conditions of the borehole fluid rather than borehole matrix that sometimes requires months to reach equilibrium (this would be especially true if the hole was drilled using drilling mud and development was not properly performed).

Temperature Logs

The introduction of fluid or air to remove drill cuttings during the drilling operation causes false temperature anomalies often requiring months to reach equilibrium. Foreign material introduced into the annulus, especially curing cement, causes anomalies not related to formation properties. For these reasons, temperature logs in recently drilled holes shall not be interpreted as formation temperature logs. Temperature logs are not easily repeated due to disturbance of the fluid during initial entry. Therefore, formation or fluid temperature logs shall be done first and recorded as the probe descends down the borehole, generally long after the well has been developed.

Typical thermal gradients shall range between 0.47°C and 0.6°C per 30 meters (m) (100 feet) of well depth. If a log results in temperatures outside this range, extraneous effects affecting the log shall be considered. In rare instances, a log may be erroneous due to problems that arise within the probe such as thermal lag, electronic drift, and self-heating of the probe thermistor. Since these conditions are the exception and not the rule, other conditions shall be examined first as a result of questionable temperature logs.

Log traces that result in small temperature fluctuations near the fluid surface may arise from large diameter probes descending too

fast within the borehole and may not be representative of borehole conditions. Small temperature fluctuations with respect to depth may be a result of borehole conditions rather than improper tool operation. Inter-bore flow due to large head differential between aquifers can cause very small or even reversal of the thermal gradient. Fluid tracers or flow measurements may help to determine these effects.

Convection cells within the borehole can result in temperature readings that are unrelated to the flow of water within the borehole. This is especially true for boreholes that have large diameters, fracture zones, and washouts. Consequently, the more accurate temperature logs are taken in small boreholes with small diameter probes.

Conductivity Logs

Conductivity logs measure the fluid conductivity within the borehole and are not always a measurement of interstitial water conductivity. Temperature drifts and movement of the probe in and out of steel casing material may give rise to sharp log deflections.

Flow Logs

Impeller flow-meters (commonly called spinners) are generally less sensitive than heat pulse or tracer release methods. In many aquifers, water movement is predominantly in the horizontal direction, with substantial seepage velocity variations as a function of vertical position. In these cases, knowledge of hydraulic conductivity with vertical position is essential. These conditions can be investigated best by installing the impeller flow-meter below an operating pump or in flowing artesian wells. Inter-borehole flow can often be determined by the heat pulse or tracer release methods.

2. Caliper Logs

Caliper log errors shall be questioned when one of the arm traces shows no deflections. Several conditions are suspect. Heavy drilling muds, most prevalent at the bottom of the borehole, can cause failure of caliper arms to open up. Nonextended arms may be jarred open by bouncing the probe up and down or simply moving the probe to a zone with thinner muds.

Boreholes with a large degree of rugosity may give rise to peculiar log traces, especially in arm-averaging traces. For these types of holes, it is recommended that nonaveraging or a single arm caliper be used. When a single arm caliper is used, repeat logs may not be exact duplicates of one another due to potential probe rotation.

Electrical leakage and grounding problems may cause spurious trace spikes.

3. Electrical Logging

Spontaneous Potential (SP) Logs

Stray electrical currents caused by underground cables, lightening strikes, corroding underground pipes, and nearby electrical motors can affect SP logs, inducing spikes or a uniform cyclical response. A repeat log will need to be done shall such interferences occur.

If the logging cable has become magnetized, the log trace will consist of a cyclical sinusoidal wave that corresponds to each revolution of the cable winch. The cable shall be demagnetized and the log rerun.

Single Point Resistance Logs

Single point resistance logs are affected by electrical fields generated from underground cables. Alternating current from the underground cable becomes superimposed over the alternating current applied to the probe. This can be detected when the trace pen fluctuates when the probe is stationary. The problem can be alleviated by changing the current frequency applied to the probe electrode and double-checking while the pen is stationary.

Normal Resistivity Logs

Nearby underground cables with currents of 60 cycles per second will produce an oscillating periodic logging trace. This problem can be alleviated by changing the current frequency applied to the probe from the generator or other power source.

A logging trace that appears to be cupped, or reversed, may be the result of an electrode spacing that is greater than the formation

thickness.

Induction Logs
Problems will arise if the resistivity of the formation water is fives times greater than the resistivity of the borehole fluid. As a double check, the inverse of induction measurements shall be in close agreement with formation resistivity measurements taken with other instruments.

4. Acoustical Logging

Acoustical televiewer (ATV) logging relies on a rotating trigger switch that is activated when a sensor passes magnetic north. Lithologic formations that are composed of magnetized material, lost drilling equipment in the borehole, and metal casing will cause the switch to malfunction. Therefore, the logging operator will need to change the trigger switch to a mechanical mechanism if they suspect the presence of magnetically susceptible materials.

Boreholes that deviate from the vertical will require careful corrections on the ATV log; these corrections shall be noted on the logging trace. If the probe is not properly centralized, dark splotches will appear on the log. This may be exacerbated when coupled with low gains.

Acoustical logs are questionable when the log trace is composed of rapid fluctuations labeled as cycle skipping. This may be a result of several conditions, such as gains that are too small or too large. For example, an amplitude that is too small will result in the first compression wave being masked by pre-arrival "noise." Skipping may also be a result of borehole fractures, solution openings, attenuating rocks, or gas in the borehole fluid.

5. Nuclear Logging

All nuclear logs will be run near the end of the logging suite. Borehole conditions will be carefully scrutinized for integrity and their suitability for nuclear logging during previously run logs within the logging suite. Confirmation that the borehole is suitable for nuclear logging will be between the FTL or designee and the logging operator. This will be noted on the nuclear logging checklist.

Any suspicions of potential problems arising during a nuclear log operation will be addressed immediately and resolved before further nuclear logging is carried out.

The following paragraphs briefly describe conditions that may give rise to anomalous nuclear log traces and shall be watched for by the logging operator.

Natural Gamma (Gamma Ray) Logs
The natural gamma log trace shall have maximum deflection with minimal statistical variation. Pulses of natural gamma radiation are collected and averaged over a predetermined time constant. If the time constant is too short, relative to the amplifier gain, the trace will be masked by spikes of statistical variation. If the time constant is too long, relative to the probe travel and amplifier gain, the log trace is rounded off and lacks resolution. Logging speed and time constant setting shall produce a trace with sharp anomalous peaks, with a minimum of clutter and maximum horizontal span.

Natural gamma logs are sensitive to borehole configurations and packing materials. Sudden trace deflections or changes in amplitude may be due to borehole diameter irregularities and drilling or well construction fluids; consequently, all gamma traces must be checked with a caliper trace.

Migration of water containing radioactive colloidal particles (i.e., clays or sylvite) through the borehole cracks and fissures will give traces that are not representative of the borehole matrix.

Gamma-Gamma (Gamma Density) Logs
Gamma-gamma logs are affected by borehole configuration, casing, cement, mud, probe "stand off," and background radiation. Therefore, a high resolution caliper log and background radiation check shall always be compared with the gamma-gamma log trace to differentiate whether the trace response is due to matrix porosity and density parameters or extraneous effects.

A 4-pi gamma-gamma density log, because of its capability to propagate energy in four directions, is very effective in evaluating the

integrity of material placed between the well casing and borehole walls. A thorough background check is especially critical to the success of this logging technique.

Attention shall be given to sharp trace deflections. These shall be compared to other logs as they may be due to the presence of cement boundary zones, the threaded interface between casing lengths, casing nested strings, and gravel packs. In the case of the 4-pi density log, these anomalous features are the target.

Neutron Logs
Neutron logs are affected by the same borehole parameters as gamma-gamma logs (except borehole casing materials) but less dramatically. As with gamma-gamma logs, cross checks between high-resolution caliper logs, background radiation, and the neutron log shall be performed to adequately interpret the trace.

Large trace deflections will occur at the interface between fluids of different densities, such as a saline/fresh water or air/water interface.

The presence of clays and shales surrounding the borehole will give anomalously low trace responses, indicating high total porosity.

5.3 Post Operation

All checklists shown in this SOP's attachments shall be filled out before leaving the site. These checklists are designed for health and safety, quality assurance, and tool operation.

At the end of each logging run (within a suite), the logging operator will complete the appropriate portions of the tool checklist. The checklist will then be verified by the FTL or designee. Any correction in data entry will be crossed out with a single line, dated, and initialed. At the end of the logging suite, each category in the checklist will have a handwritten entry. If a specific parameter does not apply, the logging operator will enter "N/A" by that category.

The original of all records will be given to the FTL or designee along with copies as may be specified. Copies of all completed checklists will also be provided.

To check for leaks, a radiation survey will be done on all nuclear probes. Shields will be checked for proper installation and defective components before probe storage. If shields of neutron sources have been found to be defective, storage before repair will be away from sodium-iodide crystals (a common component in some probe detectors) as they become radioactive when impinged upon by a neutron source.

All radioactive sources and radioactive tracers will be kept in a designated and carefully monitored storage area that has been specifically designed for storing or transporting nuclear sources. Sources stored within the facility will be sealed and placed in source holders specifically designed for their use. Each storage container will be clearly labeled with the following information: source material, concentration in Curies, and dose rate at 1 meter from the holder. Labels, warning signs, and alarms for source containers and the storage facility will follow that prescribed by the NRC. The storage facility and source containers will not be altered in any way, and both will be locked when unattended.

Each probe will be securely fixed to a stand or other similar structure where it will be cooled, cleaned, and inspected for damage. Repairs will be made when possible. If the probe cannot be repaired onsite, a note will be made of this on the tool checklist and the tool will be labeled "out of service" until repairs can be made.

The site will be checked for any debris, spills, or litter resulting from logging activities. All such matter will be removed and disposed of in accordance with regulations applicable to the site.

5.4 Cleaning and Decontamination

All equipment brought onto the site shall be clean and free of leaking oil, grease, or hydraulic fluid. Equipment that may contact the interior of the borehole shall be cleaned and decontaminated in the designated decontamination area. Downhole equipment will be: (1) steam cleaned, (2) scrubbed with a brush and laboratory-grade detergent and water solution, and (3) rinsed with potable water before its use downhole and between holes. Some downhole geophysical probes may have restrictions pertaining to decontamination and will be decontaminated according to the manufacturer's specifications.

After cleaning and decontamination, all tools and equipment that may be used downhole must be kept clean and free of contaminants. Decontaminated equipment shall be kept off the ground by storing on clean racks and/or wrapping in plastic. All tools and equipment shall be cleaned and decontaminated as required to maintain an uncontaminated condition.

6.0 Restrictions/Limitations

Several hazards are associated with activities surrounding geophysical logging and these are outlined below:

6.1 Mechanical Hazards

The use of machinery to lift and lower the probe in and out of the borehole may require the use of hoists, cables, winches, rigs, etc., that are under tension or compression. Breakage or malfunction of any one of these components could cause the release of uncontrollable forces that may in turn impart tremendous physical damage to personnel and other equipment.

Site personnel shall be aware of swinging probes (weighing up to 136 kilograms [kg] [300 pounds]), or other associated equipment, suspended in midair before entry or after exiting the borehole. Site personnel shall also guard against falling components from probes that have had joints loosened.

6.2 Electrical Hazards

Hazards may arise from the use of several electrical power sources. Potential shocks may arise from improper handling of nongrounded power supplies or from probes using induced current electrodes or high voltage generators (120 to 600 V) used to power the tools. Lines from these power sources will be connected to ground fault circuit breakers.

All generators will be grounded. All extension cords from any power source will be composed of three conductor insulated wires and will be inspected for any frays, cuts, or other damage.

6.3 Nuclear Hazards

Many geophysical logging programs rely on a suite of techniques that are collectively classified as nuclear logging. Some nuclear logging tools contain radioactive materials that serve as sources of radiation that are uniquely attenuated by the surrounding borehole matrix. Other nuclear logging tools measure ambient gamma radiation and do not use radioactive sources but require frequent calibration with radioactive materials. All permits will be kept in an open file in the logging vehicle. The use of nuclear tools needs to be preplanned and carefully thought out, monitored, and well supervised by highly trained individuals familiar with the particular logging technique and equipment. Active nuclear logging tools will be stored in shields specifically designed for each tool (passive tools do not need shielding). Monitoring equipment must be used at specified times in nuclear tool storage facilities and will be calibrated at specified intervals. Tags indicating the dates of monitor equipment calibration will be attached. Wipe test results and calibration records will be stored onsite in open files.

For the health and safety of the staff engaged in geophysical logging, all personnel working with the geophysical team will wear a Thermoluminescent Dosimeter (TLD) badge or a Radiation Exposure Film Badge as required by project, client, and/or site requirements. This badge must be worn on the top front outer garment. Exception to this rule is given to visitors and temporary assistants, who must at all times maintain a safe distance (determined by the site safety officer) between themselves and the radioactive source(s).

A radiation survey meter will be available, checked, and calibrated for operation before site transport. An up-to-date copy of the radiation survey and meter calibration will be kept on file in the logging vehicle.

The following briefly summarizes the radioactive materials used for each logging technique:

- Natural gamma logging measures the ambient gamma radiation of in situ, naturally occurring elements within the geological strata such as potassium, thorium, and uranium. Tools used to detect natural gamma radiation do not emit radiation but are calibrated with radioactive material. Calibration standards requiring a license for purchase, handling, and transport will be used only by licensed and trained individuals.

- Neutron logging requires the use of radioactive sources that emit high-energy neutrons within the logging probe, some of which also emit gamma radiation. Neutron concentrations for these probes range between a low of 10 millicuries (mCi) to a high of 5 Curies (Ci). Radioactive sources commonly used in a neutron probe are americium/beryllium mixtures. Older probes may have mixtures of beryllium and radium or beryllium and plutonium. Radium/beryllium probes have an added danger over those made of americium/beryllium in that they emit high doses of gamma radiation. Because neutron radiation is most effectively attenuated by hydrogen atoms, shields for neutron sources are composed of hydrogenous materials. Storage facilities will be monitored to assure all shields are effectively blocking neutron radiation.

- Gamma-gamma and density logs contain radioactive sources that emit gamma radiation. The radioactive source most commonly used is cesium-137, with older probes using cobalt-60 as a gamma radiation source.

7.0 References

American Society for Testing and Materials. 2010. *Standard Guide for Planning and Conducting Borehole Geophysical Logging (D5753-05 (Reapproved 2010): Annual Book of ASTM Standards*, 9

Keys, W. Scott. 1989. National Water Well Association. *Borehole Geophysics Applied to Ground-Water Investigations*.

_____.
U. S. Geological Survey. 2013. Quality-Assurance Plan for Groundwater Activities,U.S. Geological Survey, Washington Water Science Center. Open File Report 2013-1151

8.0 Attachments

Attachment 1 - Example Health and Safety Checklist
Attachment 2 - Example QA Geophysical Checklist for Borehole Logging
Attachment 3 - Example Geophysical Logging Tool Checklist
Attachment 4 - Example Log Heading

Attachment 1
Example Health And Safety Checklist

Project Name/Contract Number _____

Well Name and Number _____ Location _____

Logging Company _____

Suite of Logs Run _____

Name of Recorder _____ Initials _____

Date and Time _____

General Health and Safety Checklist

Yes	No	
		All personnel working in the restricted area wearing TLD Badges or Radiation Exposure Film Badges as required?
		Restricted area properly controlled, thus preventing any unauthorized entry?
		All radioactive sources are stored in a secured, labeled, and properly shielded location?
		A copy of the operating and emergency procedures has been reviewed by logging supervisor and is on file in the field office?
		Were there any incidents that required implementation of emergency procedures? If yes, were the emergency procedures followed?
		Were storage facilities of radioactive sources posted with the proper labels?

Attachment 2
Example QA Geophysical Checklist For Borehole Logging

Project Name/Contract Number _____

Well Name and Number _____ Location _____

Logging Subcontractor _____ Date _____

Total Borehole Depth _____ (feet) Well Depth _____ (feet) Logger _____ (feet)

Ground Elevation _____ (feet) Permanent Datum _____

Permanent Datum Elevation _____ (feet)

Suite of logs to be run: Sp _____ Temp _____ "16-64" Ris _____ SPRis _____ Ind _____

Gam Ray _____ Gam Gam _____ Caliper _____ Other _____

Name of Operator: _____ Date: _____ Initials: _____

Name of Recorder: _____ Date: _____ Initials: _____

Monitor Well Construction Materials: Casing _____ Screen _____

Drilling/Construction Fluids: _____

General Quality Assurance Checks

Yes	No	
		Base map is located onsite.
		Before running the nuclear logging suite, borehole conditions were evaluated and considered suitable for such logs using information from caliper and previously run logs (e.g., depth, variations in diameter, lost circulation zones, casing, debris in hole, fluid type, and known contaminants)
		Appropriate scales have been chosen using nearby, onsite well logs. These are available for a comparison to present log readings.
		Onsite tool calibrations have been performed using calibration checks implemented before and after each log run.
		Logging scales chosen for all logging tools are appropriate for borehole conditions and required sensitivity. Off-scale logs have been rerun using scale adjustments when the offending off-scale run cannot generate an on-scale plot.
		Were there scale changes while running a log? If yes, which logs? _____
		Scale changes, exceptional conditions, and other anomalies have been appropriately annotated on the affected logs.
		Depth control during log mergings have been checked.
		After Survey Depth Errors (ASDE) have been checked.
		Hard copies have been examined for several log runs and there is good correlation of well depth between them.
		All well log hard copies have been examined upon completion of the logging suite and good correlation exists between them.
		Borehole mud samples have been taken just before logging and mud resistivity measured.

General Quality Assurance Checks (Cont.)

Yes	No	
		Optimal data acquisition has been achieved by choosing the most appropriate logging speed and time constant from test runs (when necessary) of varying speeds.
		Logging speeds for boreholes less than 2,000 feet have not exceeded 25 feet per minute. Some tools may require slower logging speeds.
		Field log headings are filled out as completely as possible. Final print log headings are completely filled out.
		The repeat length for a uniform section has not been less than 50 feet, a variable section not less than 200 feet. When possible, boreholes less than 1,000 feet have had the entire hole length repeated.
		If log quality appears questionable, attempts have been made to ascertain cause and make adjustments. If a satisfactory log cannot be obtained, note reason.
		The specific hardware brand and model number used for data acquisition has been recorded in the field logbook. All software used for data acquisition and retrieval has been recorded in the field logbook.
		Tool manufacturer, model, and serial numbers have been noted. Tool configuration, diagrams, and manuals are available and located in the field (or logging) operation office.
		Logging subcontractor has been questioned to determine whether any tools have been modified or deviate from factory specifications. Modifications have been noted on field log checklist.
		CD ROMs have been made of the unedited, raw digital logging data and are stored in the field operation office.
		Processed, raw digital data that is in final form accompanies the final processing report.
		The final processing report has the necessary audit and audit process documentation.

Attachment 3
Example Geophysical Logging Tool Checklist

Temperature Log (This log shall be run first.)		
Make	Model	Serial No.
Modification (Yes/No)? (Describe fully on separate sheet and attach.)		
Tool schematic and operating manual on file (Yes/No)		Location of schematic
Order in which tool was run.	Run in combination with (other tools)	
Logging Speed (m/min) (ft/min)	Repeat Section Interval Location	
__ Tool logged in fluid-filled hole? (Yes/No) __ Calibrated onsite (optional)? (Yes/No) If no, where calibrated and date of calibration? __ Readings appear to be of good quality, no noise. (Yes/No) If noise exists, what is this attributed to?		

Spontaneous Potential (SP) Log		
Make	Model	Serial No.
Modification (Yes/No)? (Describe fully on separate sheet and attach.)		
Tool schematic and operating manual on file (Yes/No)		Location of schematic
Order in which tool was run.	Run in combination with (other tools)	
Logging Speed (m/min) (ft/min)	Repeat Section Interval Location	
__ Tool logged in fluid-filled hole? (Yes/No) __ Calibration performed before logging? (Yes/No) __ Calibration performed after logging? (Yes/No) __ Calibration data printed and attached to log trace. __ Sensitivity for conditions appropriate? Line has "character." (Sensitivity sediment and fluid conditions may cause lack of character.) __ No excessive SP baseline drift. __ Baseline shifts (if necessary) annotated on log. __ Resistivity of borehole fluid taken and recorded on log. __ Resistivity of interstitial water taken and recorded on log.		

Project Name/Contract Number _____ Date _____

Logging Subcontractor _____ Geophysical Logging Engineer _____ Well _____ Location _____

(Initial to certify this page)

Attachment 3
Example Geophysical Logging Tool Checklist (Cont.)

Caliper (i.e., 1 arm, 3 arm?) **Log**		
Make	Model	Serial No.
Modification (Yes/No)? (Describe fully on separate sheet and attach.)		
Tool schematic and operating manual on file (Yes/No)		Location of schematic
Order in which tool was run.	Run in combination with (other tools)	
Logging Speed (m/min) (ft/min)	Repeat Section Interval Location	
__ Calibration check performed __ Caliper reading checked in casing, if casing is present __ Repeat section logged or second run performed		

Gamma-Gamma (Density) Log		
Make	Model	Serial No.
Modification (Yes/No)? (Describe fully on separate sheet and attach.)		
Tool schematic and operating manual on file (Yes/No)		Location of schematic
Order in which tool was run.	Run in combination with (other tools)	
Logging Speed (m/min) (ft/min)	Repeat Section Interval Location	
__ Hole condition good, checked by previous log runs (do not run nuclear tools under questionable hole conditions) __ Tool logged in fluid-filled or air-filled (circle one), uncased or cased (circle one) hole __ Radiation survey conducted in immediate vicinity of drill site before unshielding source __ Radiation survey conducted in immediate vicinity of drill site after last nuclear source is shielded and logging job is finished __ Calibration performed before logging __ Calibration performed after logging __ Calibration data printed and attached to log trace __ Log checked for anomalous spikes, drift, and cyclic readings __ Compare density log to caliper log; density shall show a variant reading in area of washouts __ With probe sitting still, statistical variation shall be on the order of the square root of the count rate		

Project Name/Contract Number _____ Date _____

Logging Subcontractor _____ Geophysical Logging Engineer _____ Well _____ Location _____

(Initial to certify this page)

Attachment 3
Example Geophysical Logging Tool Checklist (Cont.)

Normal Resistivity	Spacing between electrodes (inches)	
Make	Model	Serial No.
Modification (Yes/No)? (Describe fully on separate sheet and attach.)		
Tool schematic and operating manual on file (Yes/No)		Location of schematic
Order in which tool was run.	Run in combination with (other tools)	
Logging Speed (m/min) (ft/min)	Repeat Section Interval Location	
__ Tool logged in fluid-filled hole, uncased hole __ Calibration performed before logging __ Calibration performed after logging __ Calibration data printed and attached to log trace __ Curve is stable, with no abnormal looking excursions		

Single Point Resistance		
Make	Model	Serial No.
Modification (Yes/No)? (Describe fully on separate sheet and attach.)		
Tool schematic and operating manual on file (Yes/No)		Location of schematic
Order in which tool was run.	Run in combination with (other tools)	
Logging Speed (m/min) (ft/min)	Repeat Section Interval Location	
__ Tool logged in fluid-filled, uncased hole __ Calibration performed before logging __ Calibration performed after logging __ Calibration data printed and attached to log trace __ Resistance greater than cable line resistance __ Scale reads in ohms __ Check for sharp deflections of consistent amplitude and frequency caused by mechanical problems __ Compare to caliper log. Is log greatly affected by change in borehole diameter?		

Project Name/Contract Number _____ Date _____

Logging Subcontractor _____ Geophysical Logging Engineer _____ Well _____ Location _____

(Initial to certify this page)

Attachment 3
Example Geophysical Logging Tool Checklist (Cont.)

Induction	Spacing between electrodes (inches)	
Make	Model	Serial No.
Modification (Yes/No)? (Describe fully on separate sheet and attach.)		
Tool schematic and operating manual on file (Yes/No)		Location of schematic
Order in which tool was run.	Run in combination with (other tools)	
Logging Speed (m/min) (ft/min)	Repeat Section Interval Location	

__ Tool logged in fluid-filled or air-filled (circle one), uncased hole
__ Calibration performed before logging
__ Calibration performed after logging
__ Calibration data printed and attached to log trace
__ Check for negative resistivity readings for indication of improper calibration
__ Check for similarity to 16" normal resistivity curve, for single curve, shallow investigation induction log
__ Repeat section logged

Natural Gamma (Gamma Ray) Log		
Make	Model	Serial No.
Modification (Yes/No)? (Describe fully on separate sheet and attach.)		
Tool schematic and operating manual on file (Yes/No)		Location of schematic
Order in which tool was run.	Run in combination with (other tools)	
Logging Speed (m/min) (ft/min)	Repeat Section Interval Location	

__ Tool logged in fluid-filled or air-filled (circle one), uncased or cased (circle one)
__ Radiation survey conducted in immediate vicinity of drill site before unshielding source
__ Radiation survey conducted in immediate vicinity of drill site after last nuclear source is shielded after job is finished
__ Hole condition is shielded after job is finished
__ Hole condition good, checked by previous log runs; do not run nuclear tools under questionable hole conditions
__ Tool run in fluid-filled or air-filled (circle one), uncased or cased (circle one)
__ Calibration performed before logging
__ Calibration performed after logging
__ Calibration data printed and attached to log trace
__ Check for cyclic noise
__ Repeat section logging

Project Name/Contract Number _____ Date _____

Logging Subcontractor _____ Geophysical Logging Engineer _____ Well _____ Location _____

(Initial to certify this page)

Attachment 3
Example Geophysical Logging Tool Checklist (Cont.)

Neutron	Spacing between electrodes (inches)	
Make	Model	Serial No.
Modification (Yes/No)? (Describe fully on separate sheet and attach.)		
Tool schematic and operating manual on file (Yes/No)		Location of schematic
Order in which tool was run.	Run in combination with (other tools)	
Logging Speed (m/min) (ft/min)	Repeat Section Interval Location	
__ Hole condition good, checked by previous log runs (do not run nuclear tools under questionable hole conditions) __ Tool logged in fluid-filled or air-filled (circle one), uncased or cased (circle one) hole __ Radiation survey conducted in immediate vicinity of drill site before unshielding source __ Radiation survey conducted in immediate vicinity of drill site after last nuclear source is shielded after logging job is finished __ Calibration performed before logging __ Calibration performed after logging __ Calibration data printed and attached to log trace __ Check log for adequate sensitivity __ Check log for anomalous spikes, drifts, and cyclic readings __ Repeat section logged		

Acoustic Logs		
Make	Model	Serial No.
Modification (Yes/No)? (Describe fully on separate sheet and attach.)		
Tool schematic and operating manual on file (Yes/No)		Location of schematic
Order in which tool was run.	Run in combination with (other tools)	
Logging Speed (m/min) (ft/min)	Repeat Section Interval Location	
__ Tool logged in fluid-filled, uncased hole (yes/no). If no, explain. __ Calibration not required; check tool for function by listening for transmitter clicking; may calibrate tool in fluid-filled surface casing if present __ Tool centralized __ Log checked for noise spikes, cycle skipping, or other anomalies __ Repeat section logged		

Project Name/Contract Number _____ Date _____

Logging Subcontractor _____ Geophysical Logging Engineer _____ Well _____ Location _____

(Initial to certify this page)

Attachment 4
Example Log Heading

Logging Subcontractor:			Log Technique:		
Address:			Date:		
Logging Operator:					
Drilling Subcontractor:					
Client:					
Well:					
Location:					
State:			Ground Elevation (m) (ft): Log Depth Ref:		
Borehole Data					
Logging Depth:					
Customer Measurement (m) (ft):			Logging Subcontractor Measurement (m) (ft):		
Bit Record			Casing Record		
Size	From	To	Size/Wgt/TLK	From	To
Hole Medium:				Drilling Method:	
Borehole Mud:				Circulation Time:	
Weight:		Viscosity:		Restrictions: mud formation at Temperature:	

Project Name/Contract Number _____ Date _____

Logging Subcontractor _____ Geophysical Logging Engineer _____ Well _____ Location _____

(Initial to certify this page)

Attachment 4
Example Log Heading (Cont.)

Logging Data						
		Equipment			Logging	
Log Function	Run No.	Model	Probe No.	Uphole S.N.	Dig Int (m/ft)	Speed (m/min)(ft/min)

Logging Data							
Detect OR	Spacing		Source		Logged Interval		
Type	Tx-Rx (m/ft)	Rx-Rx	Type	Curie Amount	From	To	Int. (m/ft)

Project Name/Contract Number _____ Date _____

Logging Subcontractor _____ Geophysical Logging Engineer _____ Well _____ Location _____

(Initial to certify this page)

Geophysical Logging, Calibration, and Quality Control (Includes Potential Radioactive Sites)	SOP 3-4 Revision: 7 Date: February 2015

Lithologic Logging	SOP 3-5 Revision: 9 Date: February 2015

Approved: Signature **Technical Review:** David Schroeder

1.0 Objective

This technical standard operating procedure (SOP) governs lithologic logging of core, cuttings, split-spoon samples, and subsurface samples collected during field operations at sites where environmental investigations are performed. The purpose of this SOP is to present a set of descriptive protocols and standardized reporting formats to be used in making lithologic observations. It prescribes protocols for recording basic lithologic data including, but not limited to, lithologic names, texture, composition, color, sedimentary structures, bedding, lateral and vertical contacts, and secondary features such as fractures and bioturbation.

The goal of this SOP is to provide a set of instructions to produce uniform lithologic descriptions and to present a list of references to help in this task.

2.0 Background

2.1 Definitions

The following list of definitions corresponds to the description sequences outlined in Section 5.2.1. They are provided to aid the geologist in what to look for when following the sequences. Example lithologic logs are given in Attachment A.

Name of Sediment or Rock - In naming unconsolidated sediments, the logger shall use field equipment and reference charts to help identify the grain-size distribution and shall name the material according to the procedure in Section 5.2.1. In naming sedimentary, igneous, and metamorphic rocks, the logger shall examine the specimen for mineralogy and use the appropriate classification chart in the attachments.

Texture - In examining unconsolidated sediments, the texture shall refer to the grain-size distribution, particle angularity, sorting, and packing. The logger shall provide estimates of the grain sizes present using Attachment B and C. When larger particles such as cobbles are present, determine the size of the particles and give a percentage estimate. The sediment particles shall be examined for angularity by comparing with Attachment B and the sorting shall be determined by percentage estimation. The logger shall note that the Unified Soil Classification System (USCS) uses the term grading to describe how the materials are sorted. (A poorly sorted unconsolidated material is well graded.) In examining igneous rocks, texture refers to whether the specimen is aphanitic, phaneritic, glassy, fragmental, porphyritic, or pegmatitic. Attachment D has more specific definitions of these terms. For metamorphic rocks, texture refers to whether the specimen has a foliated structure (slaty, phyllitic, schistose, or gneissic) or nonfoliated structure (granular).

Color - Color may be determined using the appropriate Munsell color chart (soil or rock) and listing the Munsell number that corresponds to the color. If an unconsolidated material is mottled in color, the ranges in color shall be described. When describing core samples with several individual colors such as in phaneritic textures, individual color names shall be listed, and an overall best color name shall be given.

Sedimentary Structures - This term refers primarily to unconsolidated sediments and sedimentary rocks. There are several different sedimentary structures, and the logger is referred to Compton's *Manual of Field Geology* (1962) book for more details. Among the more common structures are bedding, cross-bedding, laminations, and burrows. These structures shall only be included in the description if found in the samples.

Degree of Consolidation - The degree of consolidation is applicable to sedimentary rocks and unconsolidated sediments and refers to how well the material has been indurated. Unconsolidated sediments may be compacted somewhat and shall be described as loose, moderately compacted, or strongly compacted. In some cases they may be slightly cemented by caliche and shall be described as slightly cemented, moderately cemented, or strongly cemented. Sedimentary rocks are typically indurated but may vary in the degree of cementation. These materials shall be described as friable, moderately friable, or well indurated. When describing the cementing material, a test for reaction to hydrochloric acid (HCl) shall be done and results recorded under the description. If the logger believes he/she can identify the cementing material, then it shall be included in the description.

Moisture Content - Moisture content refers to the amount of water within the sediment or the matrix. Typically sedimentary rocks and unconsolidated sediments may have water within and shall be described as dry, moist, wet (not flowing), or saturated (flowing water). Igneous and metamorphic rocks may have water within fractures and cavities. The presence of water and pertinent observations that may help in site evaluation in these rocks shall be noted.

Presence of Fractures, Cavities, and Secondary Mineralization - The rock that may be encountered during drilling may have fractures or joints present within them. Should fractures be observed, they shall be noted and a description as to the density of fractures shall be given. Cavities or vugs may be present, and the density of voids, as well as size estimation, shall be given. If fractures or cavities contain evidence of secondary minerals such as zeolites, clays, or iron oxides, then a description of the mineral fill shall be added.

Evidence of Contamination - The logger shall examine the core and note any obvious signs of contamination such as streaking, free product, odor, or discoloration. These observations shall be noted in the field book as shall any readings from the photoionization or flame ionization detector (PID/FID). PID/FID hits shall be recorded on the Lithologic Log Form also.

Description of Contacts - The logger shall note any significant change in lithology. These changes may be gradational contacts within sediments or may be sharp contacts such as sediments over rocks. The contacts shall be noted as to whether they are erosional, gradational, or sharp, and the depth below the surface shall be noted.

Composition - The composition of the rock refers to the mineralogy of the material encountered. For sedimentary rocks, it is important to note the matrix composition and use Attachment E in naming. In igneous and metamorphic rocks, the minerals that make up the rock shall be stated and an estimation of their percentage shall be noted. The classification charts listed in Attachments D and F provide a description of common compositions.

2.2 Associated Procedures

- SOP 4-1, *Field Logbook Content and Control*

2.3 Discussion

The installation of monitoring wells, piezometers, and boreholes is a standard practice at many sites requiring environmental investigations. The installation of these devices requires that a trained geologist, or other earth scientist under a geologist's supervision, provide lithologic descriptions as they encounter subsurface material during auguring or drilling. In evaluating these lithologic descriptions from different boreholes, monitoring wells, or piezometers, it is sometimes possible to correlate similar units. To help in this task, it is important to provide uniform and consistent descriptions.

In describing lithologies, it is helpful to have a set of references covering items such as the classification of igneous, metamorphic, and sedimentary rocks; grain-size percentage estimation; particle shape; grain-size charts; and lithologic symbols. To make lithologic descriptions produced by CDM Smith staff as uniform and consistent as possible, this SOP provides a list of references to be used in the field. This SOP also provides a sequence for recording information on a standardized log form to make descriptions as uniform and consistent as possible.

3.0 General Responsibilities

Geologist - The field person performing lithologic logging is responsible for making a consistent and uniform log and for turning in field forms and logbooks to the field team leader (FTL).

Field Team Leader (FTL) - The FTL is responsible for maintaining logbooks and forms and for approving techniques of lithologic logging not specifically described in this SOP.

***Note*:** Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/project specific quality assurance plan.

4.0 Required Equipment

The description of subsurface lithologies requires a minor amount of field equipment for the geologist. This section provides a list of equipment to be used by the lithologic logger but does not include equipment such as drill rigs, PID/FID, sampling equipment, and personal protection equipment. The following is a general list of equipment that may be used:

- Field logbook and Lithologic Log Form
- Clipboard
- Dilute (10 percent) HCl
- Plastic sheeting
- PVC sampling trays
- Waterproof pens
- No. 2 sieve
- 10x magnifying hand lens
- Reference field charts
- Engineers tape measure or folding stick

5.0 Procedures

5.1 Office

- Obtain field logbook and Lithologic Log Forms
- Coordinate schedules/actions with FTL
- Obtain necessary field equipment (i.e., hand lens, 10 percent HCl)
- Obtain CDM Smith reference field charts
- Review field support documents (i.e., sampling plan, health and safety plan)
- Review applicable geologic references such as U.S. Department of Agriculture (USDA) Soil Conservation Survey Soil Surveys and/or geologic maps

5.1.1 Documentation

Individuals performing lithologic logging will record their observations in a commercially available, bound field logbook (e.g., Lietz books) and/or on individual Lithologic Log Forms. Lithologic loggers will follow the general procedures for keeping a field logbook (SOP 4-1). When using a bound field logbook, record the same data required on the Lithologic Log Form. Data from the field logbook must be transcribed to the Lithologic Log Form if filling in the form in the field is not feasible. However, the data must be the same as that recorded in the field logbook. Editing of field logbook data is not allowed. In addition, if data are transcribed to the Lithologic Log Form, it shall be done within 1 day of the original data recording. All blanks in the Lithologic Log Form must be filled out. If an item is not applicable, an "NA" shall be entered. Note that the Lithologic Log may be modified based on the type of drilling (i.e., changing the blow count column to rock quality designation (RQD) for rock coring).

The Lithologic Log Form shall be filled out according to the following instructions:

The front page of the form contains general information:

- The project name, location, and description
- Borehole number
- Date that the drilling activity was started and completed
- Name of the person logging the well shall be recorded along with the total depth drilled
- Borehole diameter(s) and drilling methods shall be recorded
- Name and company of the driller and the type of drill rig and bits used

A map showing the drilling location may be attached.

The continuation page(s) shall be completed according to the instructions provided within this section and according to the sequence provided in Section 5.2.1. The depth column refers to the depth below ground surface and shall be provided in feet. The tick marks can be arbitrarily set to any depth interval depending on the scale needed except where client requirements dictate the spacing. The lithology column shall contain a schematic representation of the subsurface according to the symbols found in Attachment G. Use a single X to mark the area where no core was recovered, and notes shall be recorded as to why the section was not recovered. The X shall

be marked from the top to the bottom of the section so that the entire interval is marked. If the geologist can interpret the probable lithology of the missing section with reasonable confidence, they may fill in the symbols behind the X. Sharp or abrupt contacts between lithologies will be indicated by a solid horizontal line. Gradational changes in lithologic composition will be shown by a gradual change of lithologic symbol in the appropriate zone. PID/FID hits shall be recorded within the PID/FID column at the appropriate depth, if applicable. Blow counts specifically refer to the number of hammer blows it takes to drive a split-spoon into the ground. Usually this is recorded as the number of blows per 6 inches but may vary. The recording of blow counts provides a relative feel for the cohesiveness of the formation. The individual recording lithologic logs shall ask the FTL whether it is required information. The description column is the most important part of the Lithologic Log Form and is where the lithology is described. In completing this section, use the applicable reference charts and complete according to the sequence in Section 5.2.1. The sample interval column is reserved for noting any samples taken and processed for the laboratory. The sample number shall be filled in at the appropriate depth. The last column refers to the percent core recovery. The individual performing lithologic logging shall determine the amount recovered and write the percentage at the appropriate depth.

In addition to the information on the lithologic form, the logger shall record the appropriate information into the logbook when there is a rig shutdown, rig problems, failures to recover cores, or other issues.

5.2 General Guidelines for Using and Supplementing Lithologic Descriptive Protocols

This SOP is intended to serve as a guide for recording basic lithologic information with emphasis on those sediment or rock properties that affect groundwater flow and contaminant transport. The fields of specialization of geologists using this SOP will vary. If the user has expertise in a particular field of petrology or soil science that allows for descriptions of certain geologic sections beyond the basic level required by this SOP, they may expand their descriptions. This shall be done only with approval of the FTL. The descriptive protocol presented here must be followed in making basic observations. Any further descriptions must follow a protocol that is published and generally recognized by the geologic community as a standard reference.

General lithologic description will not include collecting detailed information such as can be obtained from sieve analysis or petrographic analysis. This SOP is a guide for recording visual observations of samples in the field aided by a 10x hand lens and the other simple tools. Field descriptions shall be supplemented by petrographic analysis and sieve analysis when the FTL needs data on numerical grain-size distributions, secondary porosity development, or other data that can be collected by these methods.

Description detail will also be dependent on the drilling and sampling methods used. Descriptions of drill cuttings will generally be very basic verses detailed descriptions of soil split-spoon or rock core samples.

This SOP includes protocols for describing igneous, metamorphic, bedrock, sedimentary rocks, and unconsolidated materials. Common abbreviations used for lithologic logging purposes are given in Attachment H. This SOP includes charts to be used for classification and naming of rocks, sediments, and soils and descriptions of texture, sedimentary structures, and percentage composition of grains. There is also a chart of lithologic symbols to be used and a list of abbreviations. For charts covering other observations or field procedures not specified by this SOP, the user is referred to the following for more information:

- *Compton's Manual of Field Geology and American Geological Society (AGI) Data Sheets for Geology in the Field, Laboratory, and Office* contain other reference charts applicable to descriptions. The source of the chart used must be recorded on the Lithologic Log Form or in the field logbook.
- The Munsell soil color chart may be used for descriptions of color.
- The *Dictionary of Geological Terms* (AGI) is to be used for definitions of geological terms.

Some observations will be common to all rock and soil descriptions. All descriptions shall include as appropriate: name of sediment or rock, color, sedimentary structures, texture, moisture content, composition, fabric, significant inclusions, and degree of consolidation or induration. The description of each category shall be separated by a semicolon. Each section that discusses descriptions of a particular lithology provides a sequence for recording observations. Follow these sequences for all descriptions. All interpretive comments shall be segregated from lithologic descriptions by recording them in the remarks column.

Secondary features affecting porosity and permeability such as fractures (joints or faults), cavities, and/or bioturbation shall be described if observed. Exact measurement of apparent bed thicknesses shall be made when logging core and shall supplement terminology such as "thin" or "thick." Particular attention is to be given to recording exact locations of water tables, perched saturated zones, and description of contaminants that may be visible.

In some cases individuals logging may wish to describe materials such as unconsolidated sediments and soils according to different systems such as the USCS or USDA Soil Taxonomy System. These descriptions can provide additional information from what is required by this SOP. If an individual is competent in using other description methods, then they shall do so with permission from the FTL.

It is often more practical to use abbreviations for often repeated terminology when recording lithologic descriptions. For the terms given in this SOP, its attachments, or the associated charts to be used for description in the field, use only the designated abbreviations. Other abbreviations are allowed; however, the abbreviation and its meaning shall be recorded on the lithologic log the first time it is used and shall be recorded at least once for every well or boring log. Loggers are cautioned to limit the use of abbreviations to avoid producing a lithologic log that is excessively cryptic.

5.2.1 Protocols for Lithologic Description of Discrete Soil or Rock Cores

This section describes the protocols for completing a lithologic description based on discrete soil or rock core samples. The logger shall use the appropriate portion of this section when describing cores. In recording descriptions of sedimentary sections from a whole core, it is possible to reduce the amount of description being written by at least two strategies. One is to look at as long of a section of core as possible, looking for the "big" picture. For instance, in a 20-foot-thick zone, the dominant lithology may be siltstone that is interrupted by several thin beds of another lithology such as gravel. This section description can be simplified by writing: 35-55 below ground surface (bgs) = siltstone (with other descriptors) except as noted; 37.5-38.5 gravel zone (with descriptors); 40-42 pebble zone (with descriptors); etc. This also aids in "seeing" the thickest unit designations possible for use in modeling. Another acceptable way to describe the same interval would be: 35-37.5 siltstone; 37.5-38.5 gravel zone (with descriptors); 38-40 same as 35-37.5; 40-42 pebble zone (with descriptors); etc.

Description of Unconsolidated Material

Unconsolidated material comprises a significant portion of the sections of interest at CDM Smith sites. The shallow subsurface is very important to the hydrologic investigation, as this is the portion of the geologic section where infiltration first occurs. Much of the contamination at sites being investigated is surface contamination and therefore lies on, or within, the upper portion of the surficial material.

For the purpose of this SOP, soil refers to the upper biochemically weathered portion of the regolith and not the entire regolith itself. Soils are to be described as unconsolidated material and shall use the same description format. The scientist shall use the USCS classification if consistent with project objectives (Attachment K). More detailed soil descriptions shall only be made in addition to descriptions outlined below.

Descriptions of unconsolidated sediments shall follow the following sequence:

- Name of sediment (sand, silt, clay, etc.)
- Texture
- Composition of larger-grained sediments
- Color
- Structure
- Degree of consolidation and cementation
- Moisture content
- Evidence of bioturbation
- Description of contacts

In naming unconsolidated material (refer to Attachment I - Naming of Unconsolidated Materials), the particle size with the highest percentage is the root name. When additional grains are present in excess of 15 percent, the root name is modified by adding a term in front of the root name. For instance, if a material is 80 percent sand and 20 percent gravel, then it is gravelly sand. If the subordinate grains comprise less than 15 percent but greater than 5 percent, the name is written:

_____(dominant grain) with _____(subordinate grain). For example, a sediment with 90 percent sand and 10 percent silt would be named a sand with silt. If a sediment contains greater than 15 percent of four particle sizes, then the name is comprised of the dominant grain size as the root name and modifiers as added before. For example, if a material is 60 percent sand, 20 percent silt, and 20 percent clay the name would be a silty clayey sand. If a material is 70 percent sand, 20 percent silt, and 10 percent clay, it would be a silty sand with clay. When large cobbles or boulders are present, their percentage shall be estimated and their mineralogy recorded. Use AGI Data Sheet 29.1 (Attachment B) for grain terms. Refer to Attachment J for an example sorting chart.

Description of Bedrock Material

Descriptions of rock core can vary in detail depending on the experience of the geologist and the scope of the project. However, features that shall be noted while logging rock core include depth of major fractures, mineralization in fractures and cavities, degree of weathering, hardness, and RQD. The RQD is a ratio of the total length of intact rock 4 inches in length or longer to the length of the core run. The RQD provides a numeric indication of the degree of fracturing and weathering, and thereby, and indication of conductive zones and preferential contaminant migration pathways.

Description of Sedimentary Rocks

Sedimentary rocks consist of lithified detrital sediments such as sand and clay, chemically precipitated sediments such as limestone and gypsum, and biogenic material such as coal and coquina. The classification scheme for naming these rocks is found in Attachment E - Classification of Sedimentary Rocks.

Descriptions for sedimentary rocks shall be given in the lithologic log in the following sequence:

- Name of rock
- Texture
- Color
- Bedding
- Sedimentary structures
- Degree of composition
- Presence of fractures or vugs
- Bioturbation
- Description of contacts

Description of Igneous and Metamorphic Rocks

Igneous and metamorphic rocks are not as commonly observed at work sites, but they may be found interspersed in the sedimentary section as ash layers and as bedrock. Where they form bedrock, the development of fractures and vugs is important to their hydrologic properties. If the logger is unsure of the name of the rock because of difficulty in determining mineralogy, the name shall be accompanied by a question mark. Attachments D and F provide a classification system for these materials.

Igneous and metamorphic rock descriptions shall follow the general format:

- Name of rock
- Texture
- Color
- Degree of induration for volcaniclastics
- Composition
- Presence of fractures or vugs
- Presence of secondary mineralization
- Foliation

5.2.2 Protocols for Lithologic Description from Drill Cuttings

The majority of boreholes drilled in bedrock are drilled without sampling or coring. This section describes the protocols that may be used for completing lithologic logs when discrete soil samples or rock cores are not collected. Lithologic logging of boreholes drilled without sampling generally requires a higher level of experience from the geologist as interpretations need to be made based on a number of factors that are usually not taken into account when logging from discrete samples. Certain details recorded on lithologic logs based on discrete sampling will not be seen (such as sedimentary structures) and therefore cannot be recorded from drill cuttings. Below are general guidelines that shall be used while filling out boring logs based on drill cuttings:

Auger Drilling
The following are general guidelines that can be used to describe cuttings from auger drilling:

- Collect cuttings for descriptions at least every 5 feet or if a change in the cuttings is noticed.
- Keep in mind travel time for cuttings to reach the surface when estimating the depth from which the cuttings originated.
- Pay attention to the reaction of the drill rig as different lithologies are encountered such as chattering versus smooth drilling, rapid easy auger advancement versus slow hard drilling, and auger refusal.
- Watch for the occurrence of water.

Bedrock Rotary Drilling (including air hammer, air rotary, and mud rotary)
The following are general guidelines that shall be used during rotary drilling:

- Use a strainer to collect cuttings at intervals of at least 10 feet or changes in lithology.
- Wash the cuttings in the strainer with potable water and examine for lithology.
- Note size of rock chips.
- Note changes in drill rig responses such as increasing or slowing drilling rate, sudden drop of the drill stem, increase in chatter and record in the remarks column of the lithologic log. These are usually good indicators of changes in lithology and/or fractures.
- If drilling with air, look for changes in color and reduction or disappearance of dust as an indicator of a lithology change and/or presence of water.
- If drilling with mud/fluid rotary, watch for gain or loss of water as an indicator of conductive zones.
- Record drilling rates as feet/minute, or as start and end times of each drill rod, in the remarks column of the boring log.

6.0 Restrictions/Limitations

Only geologists, or similarly qualified persons trained in lithologic description, are qualified to perform the duties described in this SOP. The FTL for a project will have the authority to decide whether or not an individual is qualified.

7.0 References

American Geological Society. 1989. *American Geological Society Data Sheets for Geology in the Field*, Laboratory, and Office, 3rd Ed.

Compton, R.R. 1962. *Manual of Field Geology*, John Wiley & Sons Inc., New York, New York.

Neuendorf, K.K.E, et. Al. 2005. *Glossary of Geology, Fifth Edition,* American Geological Institute.

Soil Test Inc. 1975. *Munsell Color Chart*. Evanston, Illinois.

U. S. Army Corp of Engineers. 1994. *Rock Foundations, EM 1110-1-2908,* Chapter 4. November 30.

_____. 1998. *Monitoring Well Design, Installation, and Documentation at Hazardous Toxic, and Radioactive Waste Sites, EM 1110-1-4000,* Chapter 4. November 1.

U. S. Department of Agriculture Soil Conservation Service. 1999. *Soil Taxonomy*, A Basic System of Soil Classification for Making and Interpreting Soil Surveys. Second Addition.

Woodward, L.A. 1988. *Laboratory Manual Physical Geology*, University of New Mexico Printing. Albuquerque, New Mexico.

8.0 Attachments

Note: These Attachments are for informational purposes. Other equivalent charts such as USCS or logs may be used.

Attachment A - Lithologic Logs
Attachment B - Grain-Size Scale; Graph determining size of sedimentary particles, particle degree of roundness charts
Attachment C - Comparison Chart for Estimating Percentage Composition
Attachment D - Classification of Igneous Rocks
Attachment E - Classification of Sedimentary Rocks
Attachment F - Classification of Metamorphic Rocks
Attachment G - Lithologic Symbol Chart
Attachment H - Common Abbreviations for Lithologic Logging
Attachment I - Naming of Unconsolidated Materials
Attachment J - Sorting Chart
Attachment K - Example of Unified Soil Classification System (USCS)

Attachment A

Lithologic Logs

CORING LOG		Client		Boring Number
Company Name CDM Smith		Drilling Subcontractor		Sheet 1 of Sheets
Project		Location		
Name of Driller		Drill Rig(s)		
Sizes and Types of Drilling and Sampling Equipment		Northing	Easting	ORNL Grid
		Surface Elevation		
		Date Started	Date Completed	
Overburden Thickness		Depth Groundwater Encountered		
Depth Drilled into Rock		Depth to Water and Elapsed Time After Drilling Completed		
Total Depth of Hole		Other Water Level Measurements (Specify)		
Drilling Method	Borehole Diameter(s)	Depth of Surface Casing		
Total Core Recovery	Total Number of Core Boxes	Signature of Geologist		
Location Map				
Project		Boring Number		

Attachment A

Lithologic Logs (cont.)

CORING LOG
(continuation sheet)

Boring Number:

Project:

Geologist:

Sheet of Sheets

Elev (ft)	Depth (ft) bgs	Description of Materials	USCS/ Litho	Field Screening Results (ppm)	Core Run No.	RQD	Recovery (ft)	Remarks

Project:

Boring Number:

Attachment A

Lithologic Logs (cont.)

DRILLING LOG		Client		Boring Number
Company Name: CDM Smith		Drilling Subcontractor		Sheet 1 of Sheets
Project		Location		
Name of Driller		Drill Rig(s)		
Sizes and Types of Drilling and Sampling Equipment		Northing	Easting	ORNL Grid
		Surface Elevation		
		Date Started	Date Completed	
Overburden Thickness		Depth Groundwater Encountered		
Depth Drilled into Rock		Depth to Water and Elapsed Time After Drilling Completed		
Total Depth of Hole		Other Water Level Measurements (Specify)		
Drilling Method	Borehole Diameter(s)	Depth of Surface Casing	Signature of Geologist	
Location Map				
Project			Boring Number	

Attachment A

Lithologic Logs (cont.)

DRILLING LOG
(continuation sheet)

Boring Number:

Project: | Geologist: | Sheet of Sheets

Elev (ft)	Depth (ft) bgs	Description of Materials	USCS/ Litho	Field Screening Results (ppm)	Sample No.	Blow Counts	Recovery (ft)	Remarks

Project: Boring Number:

Lithologic Logging

SOP 3-5
Revision: 9
Date: February 2015

Attachment B

Grain-Size Scale; Graph determining size of particles, particle degree of roundness charts

AGI DATA SHEET 29.1

Grain-size Scales

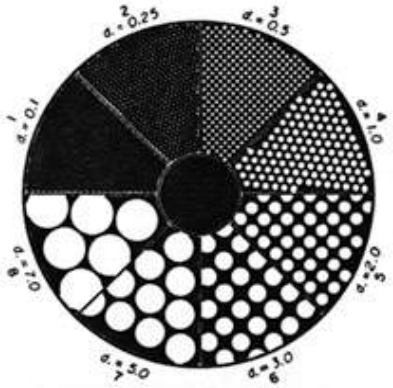
By Roy L. Ingram, University of North Carolina

GRAIN-SIZE SCALE USED BY AMERICAN GEOLOGISTS
Modified Wentworth Scale — after Lane, et al., 1947, Trans. American Geophysical Union, v. 28, p. 936-938

GRADE LIMITS phi	mm	mm	inches	U.S. Standard Sieve Series	GRADE NAME		
−12	4096		161.3		very large		
−11	2048		80.6		large		
−10	1024		40.3		medium	Boulders	
−9	512		20.2		small		
−8	256		10.1		large		
−7	128		5.0		small	Cobbles	GRAVEL
−6	64		2.52	63 mm	very coarse		
−5	32		1.26	31.5 mm	coarse		
−4	16		0.63	16 mm	medium	Pebbles	
−3	8		0.32	8 mm	fine		
−2	4		0.16	No. 5	very fine		
−1	2		0.08	No. 10	very coarse		
0	1		0.04	No. 18	coarse		
+1	1/2	0.500		No. 35	medium	Sand	SAND
+2	1/4	0.250		No. 60	fine		
+3	1/8	0.125		No. 120	very fine		
+4	1/16	0.062		No. 230	coarse		
+5	1/32	0.031			medium		
+6	1/64	0.016			fine	Silt	
+7	1/128	0.008			very fine		
+8	1/256	0.004			coarse		MUD
+9	1/512	0.002			medium		
+10	1/1024	0.001			fine	Clay size	
+11	1/2048	0.0005			very fine		
+12	1/4096	0.00025					

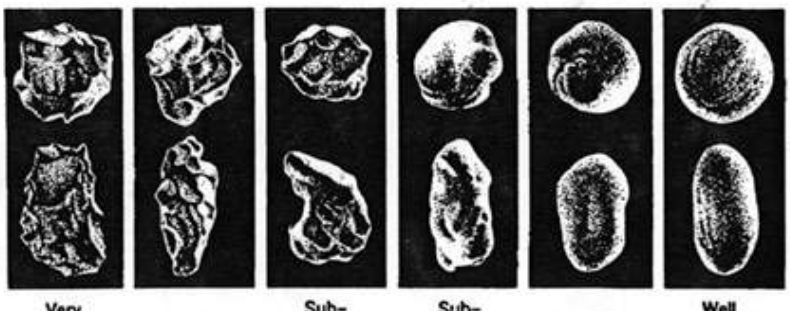
AGI DATA SHEET 28.2

LIGHT PARTICLES



References: (1) George V. Chilingar, 1958, Soviet classification of sedimentary particles and Vasil'evskiy graph: AAPG Bull., v. 40, no. 7, p. 1714. (2) M.S. Shvetsov, 1948, Petrography of sedimentary rocks, 2nd ed., 387 p. Gosgeoltizdat, Moscow-Leningrad

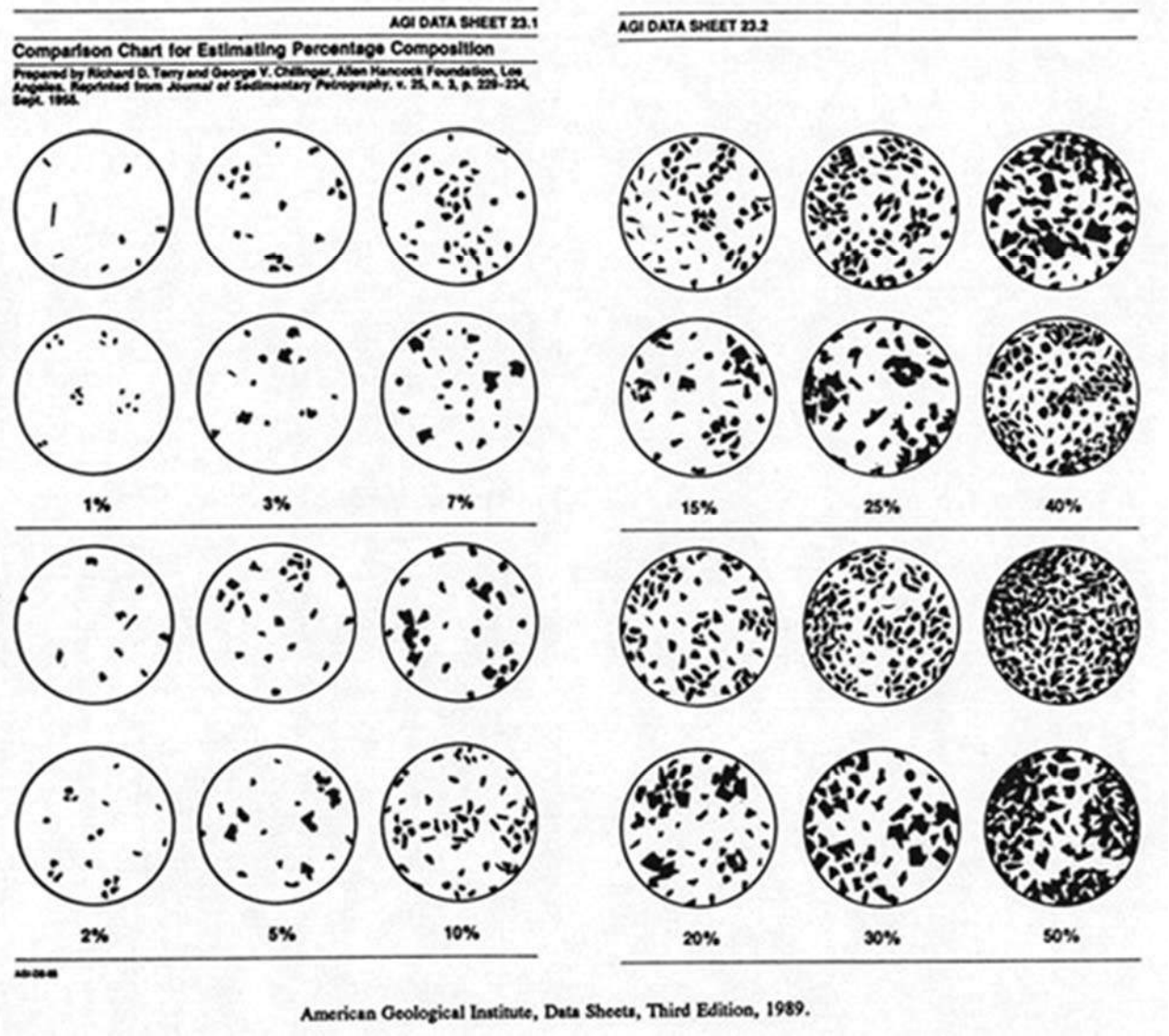
American Geological Institute, Data Sheets, Third Edition, 1989.



Compton, R.R., Manual of Field Geology, 1962.

Attachment C

Comparison Chart for Estimating Percentage Composition



American Geological Institute, Data Sheets, Third Edition, 1989.

Attachment D

Classification of Igneous Rocks

Classification of Igneous Rocks					
Mineral Composition					
		Quartz >10% Abundant feldspar Mafic minerals minor	Quartz <10% Abundant feldspar Mafic minerals moderate	Feldspar abundant Mafic Minerals 40-70%; Quartz minor or absent	Mafic minerals >70%
Color Index		Light Color	Intermediate color	Dark	Dark
Chemistry		SiO_2 70%	SiO_2 60%	SiO_2 50%	SiO_2 40%
TEXTURE	Phaneritic (visible with naked eye)	Granite (Gr)	Diorite (Dr)	Gabbro (Gb)	Peridotite (Pr) (mostly olivine)
	Aphanitic (microscopic)	Rhyolite (Ry) (quartz phenocrysts)	Andesite (An) (feldspar or mafic phenocrysts; no quartz)	Basalt (Ba)	Komatiite (Km) (very rare)
		Felsite (Fl) (no phenocrysts)			
	Glassy	Obsidian (ob) Pumice (Pu)		Rare	
	Glassy-Fragmental (Pyroclastic)	Tuff <4mm (Tf) Breccia >4mm (Br)		Rare	

Attachment E

Classification of Sedimentary Rocks

Classification of Sedimentary Rocks				
Detrital	***Detrital Classification***	***Principal Composition***	***Additional Identifying Characteristics***	***Name of Rock***
	Rudaceous (clast diameter > 2 mm)	Gravel	Rounded Clasts	Conglomerate (Cg)
			Angular Clasts	Breccia (Br)
	Arenaceous (clast diameter between 0.0625 mm [1/16 mm] and 2 mm)	Sand	Mineral composition and detrital matrix content varies. Additional detrital matrix qualifiers (arenite or wacke) and mineral composition qualifiers (quartz, arkose, feldspathic, etc.) may be necessary.	Sandstone (Sa)
	Argillaceous (clast diameter <0.0625 mm)	Mud	Non-fissile along bedding planes, silt predominant over clay	Siltstone (Sls)
			Non-fissile along bedding planes, clay predominant over silt	Claystone
			Non-fissile along bedding planes, silt and clay fraction approximately equal or unknown	Mudstone (Ms)
			Fissile along bedding planes	Shale (Shl)
Chemical	***Chemical Classification***	***Principal Composition***	***Additional Identifying Characteristics***	***Name of Rock***
	Calcareous	Calcite (Calcium Carbonate)	Effervesces on contact with dilute HCl	Limestone (La)
		Dolomite (Calcium Magnesium Carbonate)	Pulverized sample effervesces on contact with dilute HCL	Dolomite (Dl), Dolostone
	Siliceous	Quartz (Silicon Dioxide)	Hard, dense, fractures conchoidally	Chert (Ch)
	Evaporites	Hydrated Calcium Sulfate	Earthy and crumbly	Gypsum (Gy)
		Calcium Sulfate	Usually exhibits indistinct stratification	Anhydrite
		Halite (Sodium Chloride)	Cubic cleavage	Rock Salt (Na)
Organic (Organogenetic or Biochemical)	***Chemical Classification***	***Principal Composition***	***Additional Identifying Characteristics***	***Name of Rock***
	Calcareous	Fossil shells and fragments	Loosely cemented fragmental limestone	Coquina (Cq)
		Foraminiferal shells	Soft, micritic limestone	Chalk (Chk)
		Calcite or aragonite	Derived from evaporation of spring water	Travertine (Tvr)
	Siliceous	Diatom shells (saltwater or freshwater organisms)	Light-colored, soft, friable, and porous siliceous deposit	Diatomite (Dm)
	Carbonaceous	Plant Remains	Degree of lithification varies-additional qualifiers such as peat, lignite, bituminous and anthracite may be necessary.	Coal (Cl)

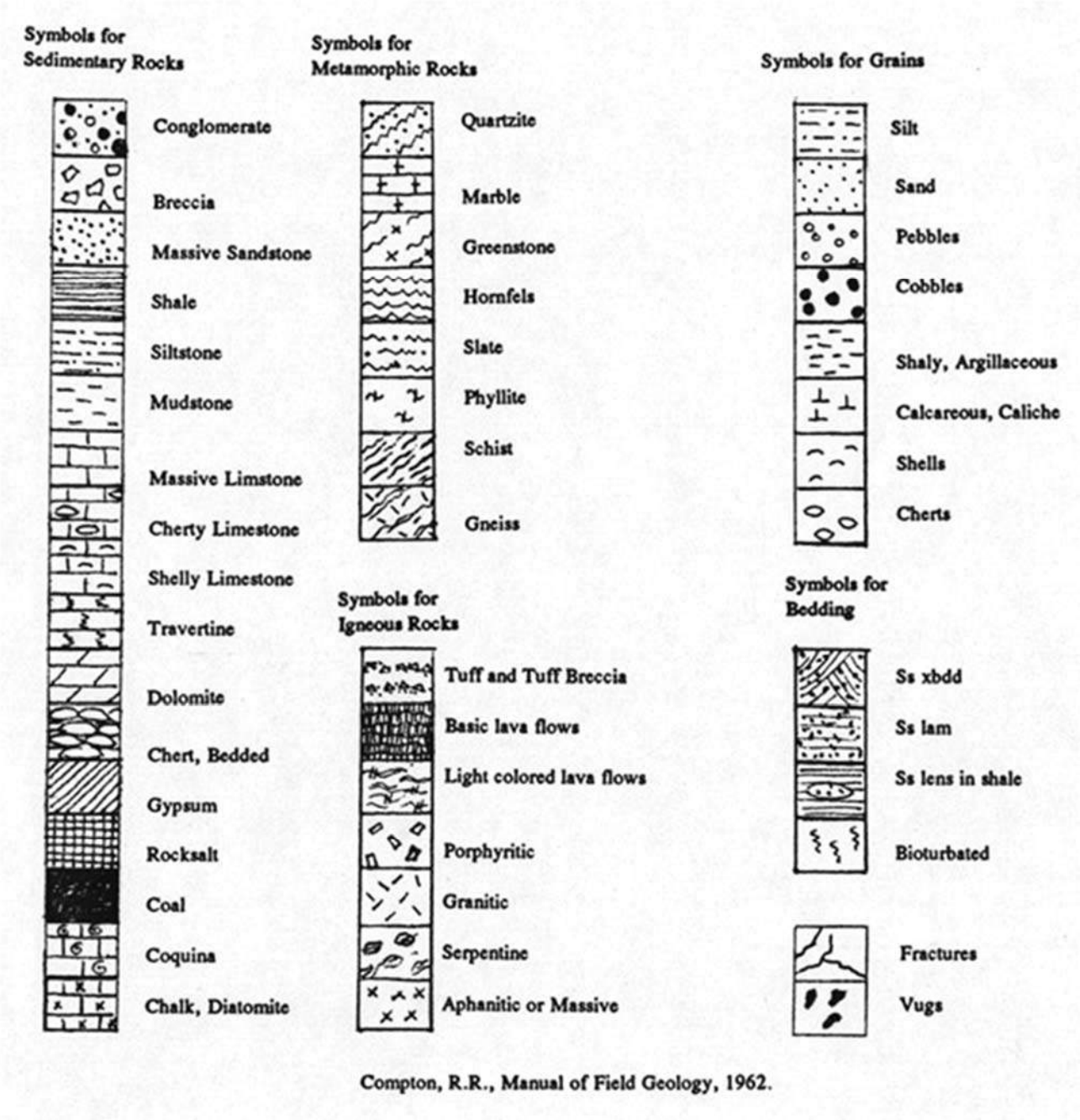
Attachment F

Classification of Metamorphic Rocks

Classification of Metamorphic Rocks			
Structure	**Texture**	**Chief Minerals**	**Name**
Non foliate	granular; breaks across grains	quartz	Quartzite (Qzt)
	granular; grains clearly visible	calcite	Marble (Mbl)
	granular; grains altered and indistinct	plagioclase, chlorite, epidote hornblende	Greenstone (Grs)
	very fine-grained	indistinguishable; mostly submicroscopic micas and clays	Hornfels (Hnf)
Foliate	slaty	submicroscopic mica, quartz	Slate (Slt)
	phyllitic	microscopic mica, quartz	Phyllite (Pyl)
	schistose	microscopic mica, quartz, amphibole	Blueschist
		chlorite, mica plagioclase	chlorite schist (CL-Sch)
		muscovite, quartz	Muscovite (Ms) Schist (Sch)
		garnet, muscovite	Garnet (G) Muscovite (Ms) Schist (Sch)
		hornblende, plagioclase	Amphibolite (Amp)
		staurolite, garnet, muscovite	Garnet (G) Staurolite (S) Muscovite (Ms) Schist (Sch)
	gneissose	plagioclase, hornblende	Amphibolite (Amp) Gneiss (Gns)
		feldspar, quartz	Granitie (Gr) Gneiss (Gns)
		eye-shaped feldspar, mica	Augen (Au) Gneiss (Gns)

Attachment G

Lithologic Symbol Chart



Compton, R.R., Manual of Field Geology, 1962.

Attachment H

Common Abbreviations for Lithologic Logging

Common Abbreviations		
Abundant – abnt	Diameter – dia	Laminated – lam
Amount – amt	Different – diff	Maximum – max
Approximate – approx	Disseminated – dissem	Pebble – pbl
Arenaceous – aren	Elevation – elev	Phenocryst – phen
Argillaceous – arg	Equivalent – equiv	Porphyritic – proph
Average – ave	foliated – fol	Probable – prob
Bedded – bdd	Formation frm	Quartz – qrz
Bedding – bdg	Fracture – frac	Regular – reg
Calcareous – calc	Fragmental – frag	Rocks – rx
Cemented – cmt	Granular – Gran	Rounded – rnd
Cobble – cbl	Gypsiferous – Gyp	Saturated – sat
Contact – ctc	Horizontal – hriz	Secondary – sec
Cross-bedded - xbdd	Igneous – ign	Siliceous – sil
Cross-bedding – xbdg	Inclusion – incl	Structure – struc
Cross-laminated – xlam	Interbedded – intbdd	Unconformity – uncnf
Crystal – xl	Irregular – ireg	Variegated – vrgt
Crystalline – xln	Joint – jnt	Vein – vn
Grain Size	**Contacts**	**Sorting**
grain – gn	gradational – grad	poor – pr
fine – f	erosional – er	moderate – mod
very fine – vf	abrupt – ab	well – well
medium – med		
coarse – crs	**Fabric**	
large – lg	grain supported – gs	
very large – vlg	matrix supported – ms	
small – sm	imbricate – im	

Adapted from, Compton, R.R., *Manual of Field Geology*, 1962.

Attachment I

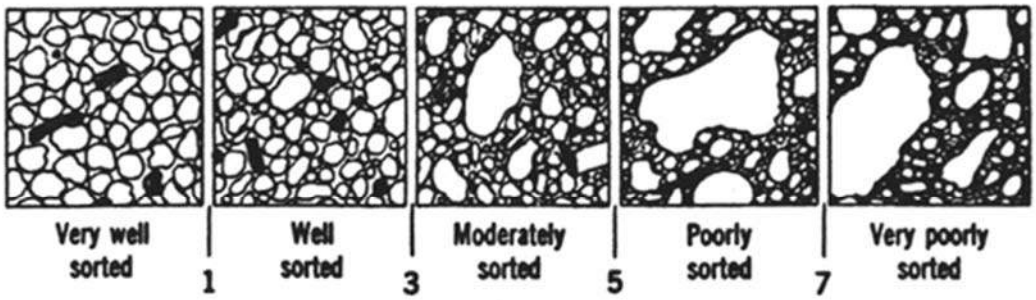
Naming of Unconsolidated Materials

Main Particle	Gravel	Sand	Silt	Clay
> 15 % gravel	Gravel	Gravelly Sand	Gravelly Silt	Gravelly Clay
> 15 % sand	Sandy Gravel	Sand	Sandy Silt	Sandy clay
> 15 % silt	Silty Gravel	Silty Sand	Silt	Silty Clay
> 15 % clay	Clayey Gravel	Clayey Sand	Clayey Silt	Clay
5-15 % gravel	Not Applicable	Sand with Gravel	Silt with Gravel	Clay with Gravel
5-15 % sand	Gravel with sand	Not applicable	Silt with Sand	Clay with sand
5-15 % silt	Gravel with silt	Sand with silt	Not applicable	Clay with silt
5-15 % clay	Gravel with clay	Sand with clay	Silt with clay	Not applicable
> 15% gravel plus 15% sand	Sandy Gravel	Gravelly Sand	Gravelly Sandy Silt	Gravelly Sandy Clay
> 15% gravel plus 15% silt	Silty Gravel	Gravelly Silty Sand	Gravelly Silt	Gravelly Silty Clay
> 15% gravel plus 15% clay	Clayey Gravel	Gravelly Clayey Sand	Gravelly Sandy Silt	Gravelly Clay
> 15% sand plus 15% silt	Silty Sand Gravel	Silty Sand	Sandy Silt	Sandy Silty Clay
> 15% sand plus 15% clay	Sandy Clayey Gravel	Clayey Sand	Sandy Clayey Silt	Sandy Clay
> 15% silt plus 15% clay	Silty Clayey Gravel	Silty Clayey Sand	Clayey Silt	Silty Clay
Note: Other combinations are possible when all particle sizes are present in greater than 15%. For example, a Silty Clayey Gravelly Sand. Other possible combinations exist such as a Gravelly Sand with silt.				

Compton, R.R., *Manual of Field Geology*, 1962.

Attachment J

Sorting Chart



Compton, R.R., Manual of Field Geology, 1962.

Attachment K

Example of Unified Soil Classification System (USCS)

Unified Soil Classification System (USCS)

	MILLIMETERS	INCHES	SIEVE SIZES
BOULDERS	> 300	> 11.8	-
COBBLES	75 - 300	2.9 - 11.8	-
GRAVEL: COARSE	75 - 19	2.9 - .75	-
FINE	19 - 4.8	.75 - .19	3/4" - No. 4
SAND: COARSE	4.8 - 2.0	.19 - .08	No. 4 - No. 10
MEDIUM	2.0 - .43	.08 - .02	No. 10 - No. 40
FINE	.43 - .08	.02 - .003	No. 40 - No. 200
FINES: SILTS	< .08	< .003	< No. 200
CLAYS	< .08	< .003	< No. 200

Attachment K

Example of Unified Soil Classification System (USCS)
(Continued)

CLAY

CLAY CONSISTENCY	THUMB PENETRATION	SPT, N BLOWS/ FT.	Undrained Shear Strength c (PSF) TORVANE	Unconfined Compressive Strength q_u Pocket Penetrometer
VERY SOFT	Easily penetrated several inches by thumb. Exudes between thumb and finger's when squeezed in hand.	< 2	250	500
SOFT	Easily penetrated one inch by thumb. Molded by light finger pressure.	2 - 4	250 - 500	500 - 1000
MEDIUM STIFF	Can be penetrated over 1/4 " by thumb with moderate effort. Molded by strong finger pressure.	4 - 8	500 - 1000	1000 - 2000
STIFF	Indented about 1/4" by thumb but penetrated only with great effort.	8 - 15	1000 - 2000	2000 - 4000
VERY STIFF	Readily indented by thumbnail.	15 - 30	2000 - 4000	4000 - 8000
HARD	Indented with difficulty by thumbnail.	> 30	> 4000	> 8000

SAND

SOILTYPE	SPT, N Blows/ft.	Relative Density,%	FIELD TEST
VERY LOOSE SAND	4	0 - 15	Easily penetrated with 1/2" reinforcing rod pushed by hand.
LOOSE SAND	4 - 10	15 - 35	Easily penetrated with 1/2" reinforcing rod pushed by hand.
MEDIUM DENSE SAND	10 - 30	35 - 65	Penetrated a foot with 1/2" reinforcing rod driven with 5-lb hammer.
DENSE SAND	30 - 50	65 - 85	Penetrated a foot with 1/2" reinforcing rod driven with 5-lb hammer.
VERY DENSE SAND	50	85 - 100	Penetrated only a few inches with 1/2" reinforcing rod driven with 5-lb hammer.

Attachment K

Example of Unified Soil Classification System (USCS) (Continued)

Summary of USCS Field Identification Tests							
Coarse-Grained Soils **More than half the material (by weight) is individual grains visible to the naked eye**	**Gravelly Soils** More than half of coarse fraction is larger than 4.75 mm		**Clean Gravels** Will not leave a stain on a wet palm	Substantial amounts of all grain particle sizes			GW
				Predominantly one size or range of sizes with some intermediate sizes missing			GP
			Dirty Gravels Will leave a stain on a wet palm	Non-plastic fines (to identify, see ML below)			GM
				Plastic fines (to identify, see CL below)			GC
	Sandy Soils More than half of coarse fraction is smaller than 4.75 mm		**Clean Sands** Will not leave a stain on a wet palm	Wide range in grain size and substantial amounts of all grain particle sizes.			SW
				Predominantly one size or a range of sizes with some intermediate sizes missing			SP
			Dirty Sands Will leave a stain on a wet palm	Non-plastic fines (to identify, see ML below)			SM
				Plastic fines (to identify, see CL below)			SC
Fine-Grained Soils **More than half the material (by weight) is individual grains not visible to the naked eye** **(<0.074 mm)**	**Ribbon**	**Liquid Limit**	**Dry Crushing Strength**	**Dilatancy Reaction**	**Toughness**	**Stickiness**	
	None	<50	None to Slight	Rapid	Low	None	ML
	Weak	<50	Medium to High	None to Very Slow	Medium to High	Medium	CL
	Strong	>50	Slight to Medium	Slow to None	Medium	Low	MH
	Very Strong	>50	High to Very High	None	High	Very High	CH
Highly Organic Soils	Readily identified by color, odor, spongy feel, and frequently by fibrous texture						OL OH Pt

Underground Facility Location*

Applicable only if CDM Smith, its subcontractor, or lower-tier subcontractor performs the intrusive work. Consult with responsible party for all other situations.

SOP 3-6
Revision: 2
Date: February 2015

Approved: Signature **Technical Review:** Stuart Barden

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to provide background information, resources, and guidance for locating underground facilities prior to performing excavations or other intrusive activities such as soil boring or demolition activities.

2.0 Background

To protect the public health and avoid serious and costly accidents that occur each year due to damage to underground utilities from excavation and other intrusive activities, most states have enacted laws and promulgated regulations that require notification and markout of underground utilities prior to performing excavation or other intrusive activities. These laws also require underground facility owners and operators to locate, markout, or otherwise provide the location of underground facilities that they operate so that contractors and others performing intrusive activities can take reasonable care to avoid damaging the facilities.

All states, and in some cases, localities within states, have established "one-call" type systems to facilitate location of underground facilities. These systems typically are operated by independent entities that are funded by the underground facility operators. They provide communication and verification services for locating underground facilities. One-call system operators are responsible for contacting the appropriate underground facility owners, ordering markouts, and tracking markout orders. The one-call system operator maintains records of requests for markouts and also maintains contact information for the field supervisor or other responsible person who requested the markout. This simplifies the process for contractors and allows them to take reasonable care to avoid accidental damage to underground utilities.

Most state laws require contractors to contact the one-call system prior to performing intrusive activities. The contractor (CDM Smith) must provide information including the site location (in some cases, the boundary of the excavation must be marked out), proposed start date, depth of the excavation, and contact information for a representative of the contractor, etc. Most laws provide for civil penalties for violation of the law and some provide for criminal penalties for negligence if an underground facility is damaged and the one-call system was not contacted.

Each state law is different, and it is the responsibility of the contractor to understand the law and activate the state's one-call system. Most states maintain web sites that provide one-call system information and offer downloadable forms for requesting subsurface facility markouts. Many of the one-call web sites also provide state regulations, procedures for using the system, procedures to follow if an underground facility is struck, and other important information. A list of telephone numbers to activate the one-call system in various states and localities is provided in Attachment A.

Often responsibility for contacting the one-call system and confirming the markout is transferred, by contract, to the subcontractor performing the intrusive work (i.e., driller, excavator, etc.). It is important to take reasonable care to ensure that the subcontractor has contacted the one-call system, that the markout has been completed, and that it includes the area where the intrusive activity will occur. No work should occur until the site manager or field manager is confident that the subcontractor has met their responsibility as defined in the subcontract statement of work.

Using a one-call system is not a guarantee that all underground facilities in an area have been identified. Not all facility operators participate in the one-call system or they may be exempt from participation by law. The one-call system operator can provide information on participating operators. Operators of exempted or nonparticipating underground facilities must be contacted directly to determine the location of their facilities. Responsibility for contacting these operators and locating the underground facilities may be transferred to a subcontractor via the subcontract statement of work. If responsibility is not transferred to a subcontractor, then it is the responsibility of the CDM Smith site manager or designee to contact the facility operator and determine the location of the underground facility.

Underground Facility Location*

Applicable only if CDM Smith, its subcontractor, or lower-tier subcontractor performs the intrusive work. Consult with responsible party for all other situations.

SOP 3-6
Revision: 2
Date: February 2015

2.1 Definitions

Excavation - State laws often define this term broadly to mean any movement of any earthen materials or rock, especially by means of machinery or explosives. Excavations below minimum depths (e.g., 12 inches) may trigger activation of the one-call system. For example, New Jersey's Underground Facility Protection Act defines excavation as:

> "...any operation in which earth, rock, or other material in the ground is moved, removed, or otherwise displaced by means of any tools, equipment, or explosive, and includes but is not limited to drilling, grading, boring, milling, to a depth greater than six inches, trenching, tunneling, scraping, tree and root removal, cable or pipe plowing, fence post or pile driving, and wrecking, razing, rending, or removing any structure or mass material, but does not include routine residential property or right of way landscaping activities performed with non-mechanized equipment, excavation within the flexible or rigid pavement box within the right-of-way, or the tilling of soil for agricultural purposes to a depth of 18 inches or less."

One-Call Operator - The person identified by the one-call system as the point of contact for location of underground facilities. The one-call operator is responsible for obtaining excavation location information from the excavator, contacting facility operators, ordering the markout, and providing verification that the markout has been completed.

One-Call System - Any of a number of communication systems established by state or local law to provide underground facility markout services to protect underground facilities. There are a number of state and regional one-call systems such as Dig Safe, Call Before You Dig, One Call, Miss Utility, and others.

Underground Facilities - Typically public or private facilities that are buried below ground or submerged on public or private property and used to convey water, sewage, telecommunications, cable television, electricity, oil, petroleum, gas, optical signals, or traffic control. Be aware that some regulations may exclude certain facilities such as storm drains and gravity sewers.

Operator - Person or entity that owns or operates an underground facility as defined above. One-call laws generally require operators to provide the location of their facilities and to perform the markout. Homeowners who operate underground facilities, such as lawn sprinklers, are usually not considered to be operators.

Waiting Period - Most one-call systems establish a minimum time to allow the markout to be completed before excavation activities can proceed. Lead times vary by state but usually are 2 to 3 business days.

Expiration Date - Some states require that the excavation activities must begin within a certain time period after the markout is completed or after the one-call system is contacted. The party responsible for locating the underground facilities is also responsible for maintaining the markouts (e.g., ensuring that markouts remain clearly visible for the duration of intrusive activities). If work is delayed, check with the one-call system to ensure that the markout remains valid.

2.2 Discussion

Excavation Activities on Residential Properties - Underground facility markouts often do not extend onto residential properties. Markouts usually show residential connections at the boundaries of the property, but end there. Excavation activities planned on residential properties require the use of other methods to determine the location of underground utilities such as water lines, electrical lines, septic systems, leach fields, and sewer lines. In addition, homeowners may install other underground facilities such as water sprinkler systems, low voltage lighting lines, well piping, and swimming pool plumbing.

The planning process for conducting work on residential properties must consider the activities that will be performed and their locations relative to the building footprint. Information about the location of underground utilities may be obtained from the residents before performing intrusive activities, but this information is often uncertain. Procurement of a geophysical subcontractor experienced in locating underground utilities is often the best choice to avoid damage to residential underground facilities. The responsibilities of the subcontractor for locating underground facilities should be clearly defined in the subcontract statement of work. Additional methods for avoiding utilities include hand digging and use of a vacuum system to remove soil from the excavation.

Use of an Underground Facility Location Subcontractor - In some situations or in particular areas of the country, it is customary to procure the services of a subcontractor experienced with providing utility markout services. This may be particularly useful for sites that cover large areas and require numerous markouts or in certain areas of the country where these services are used to identify underground facilities. For example, in some western states, the one-call systems are operated at the county or regional level and may require more coordination activities. The subcontractor must comply with all state and local regulations including activation of the one-call system. When this option is selected to identify underground facilities, the procedures described below for subcontractor as the excavator should be followed. The responsibilities of the subcontractor for locating underground facilities should be clearly defined in the subcontract statement of work.

2.3 Associated Procedures

- SOP 1-4, *Subsurface Soil Sampling*
- SOP 3-1, *Geoprobe® Sampling*
- SOP 4-4, *Design and Installation of Monitoring Wells in Aquifers*

3.0 Responsibilities

Project Manager - The project manager is responsible for ensuring that underground facility location responsibilities are clearly stated in subcontractor statements of work, as applicable.

Site Manager - The site manager is responsible for ensuring that the subcontractor has met their facility location responsibility as defined in the subcontract statement of work. If the underground facility location responsibility is not transferred to a subcontractor, it is the responsibility of the site manager to contact one-call and facility operators to determine locations.

Field Manager - The field manager is responsible for ensuring that work is not initiated until they are confident that underground facility location has occurred in the area of the work.

4.0 Procedures

General Procedures to Ensure that Underground Facilities are Identified Before Excavation

Note: The terms 'excavator' and 'excavation' used in the following procedures are broadly defined to include a variety of intrusive activities including excavation, soil boring, well drilling, test excavations, etc.

Subcontractor as the Excavator - Frequently CDM Smith transfers the responsibility for contacting the appropriate one-call system, via the subcontract statement of work, to a subcontractor such as an excavator or driller. The procedures below apply to the location of underground facilities, when a subcontractor is responsible for contacting the one-call system, and identifying underground facilities.

- Clearly define the subcontractor's responsibility in locating underground facilities in the subcontract statement of work.
- Obtain and be aware of the state or local regulations concerning excavation activities and underground facilities (contact the one-call system's website or call the system operator).
- Define areas for excavation. Be as specific as possible with the location and include address, block and lot, milepost markers, cross streets, drawings, etc. (***Note*** - State law may require that the proposed excavation area be physically delineated before the markout. Also be aware that methods for identifying locations vary from state to state).
- Provide the locations of the proposed excavations/borings to the subcontractor.

Underground Facility Location*

Applicable only if CDM Smith, its subcontractor, or lower-tier subcontractor performs the intrusive work. Consult with responsible party for all other situations.

SOP 3-6
Revision: 2
Date: February 2015

- Before beginning work, verify that the subcontractor has contacted the one-call system and that the required markouts have been completed. Obtain confirmation numbers or other documentation to verify that the subcontractor has contacted the one-call system or underground facility operator and that the markout has been completed. Obtain a list of the utilities that were contacted. Record all conversations with the subcontractor related to underground utility location in the field logbook.

- Before beginning the excavation activities, ensure that the appropriate area has been marked out and that the required waiting period has elapsed. Also, ensure that a delay in the start of the excavation activity has not invalidated the markout. If in doubt, contact the one-call system.

- Perform a walkover and visual inspection of the markout area. Identify any overhead hazards and establish appropriate offsets. Verify that the markout area covers the proposed excavation area. If any of the markouts are unclear or do not cover the excavation area, notify the subcontractor that the markout is not adequate. Request an additional markout if necessary.

- During the visual inspection, note the locations of features that may indicate underground facilities (manholes, sewer grates, vent pipes, electrical boxes, curb cuts, pavement patches, etc.). Depending on the state, not all utilities may be marked out (e.g., gravity sewers are excluded from New Jersey's underground facility protection law). Some facility operators may not have the resources to participate in the one-call system. The local municipality or utility must be contacted to determine the location of these underground facilities.

- Begin excavation activities. Be cautious during the initial stages of the intrusive activity. Be alert for signs that an underground facility may be present. Look for changes in the soil characteristics such as the presence of fill materials or non-native materials. Be alert for visual and aural cues that may indicate the presence of underground facilities (metallic sounds, changes in drilling progress, etc.). The party responsible for locating the underground facilities must maintain the markouts. Document maintenance of markouts in the field logbook and photographically.

- If an underground facility is struck or breeched, cease operations immediately. Remove all personnel from the area, inform the subcontractor, contact the facility operator, inform your supervisor, and follow the emergency procedures in the site-specific health and safety plan. The subcontractor should immediately contact the one-call system and identify the problem.

Note: **If CDM Smith takes an active role in contacting the one-call system, utility companies, or other operators of underground facilities, it may relieve the subcontractor of responsibility for locating the underground utilities. Before beginning intrusive activities, the site manager and subcontract manager should be consulted if it is believed that the subcontractor has not fulfilled his responsibility under the subcontract.**

CDM Smith as the Excavator - The procedures below define reasonable steps to be taken to avoid damage to underground facilities when CDM Smith is the excavator. In most cases it is advisable to designate a subcontractor (via contract) as the party responsible for location of underground facilities.

- Obtain and be aware of the state or local regulations concerning excavation activities and underground facilities.

- Obtain a copy of the appropriate one-call system underground facility location request form to determine what information is required to submit an underground facility location request.

- Define areas for excavation. Be as specific as possible with the location and include address, lot and block, milepost markers, cross streets, drawings, etc. (***Note*** - State law may require that the proposed excavation area be physically delineated before the markout).

- Contact the appropriate one-call system and provide information on the location of the proposed excavation and field manager or site manager contact information (provide the specific information required by the state's one-call system request form).

- Obtain a confirmation number or other documentation to verify that the markout has been completed. Obtain a list of the utilities that were contacted by the one-call system operator. Record all conversations with the one-call operator of underground utility operator in the field logbook.

- Before beginning the excavation activities, ensure that the appropriate area has been marked out and that the required waiting period has elapsed. Also, ensure that a delay in the start of the excavation activity has not invalidated the markout. If in doubt, contact the one-call system.

- Perform a walkover and visual inspection of the markout area. Identify any overhead hazards and establish appropriate offsets. Note the location of the marked out underground facilities and the proposed excavation location(s). If any of the markouts are unclear or do not cover the excavation area, contact the one-call system. Also during the visual inspection, note the locations of features that may indicate underground facilities (manholes, sewer grates, vent pipes, electrical boxes, curb cuts, pavement patches, etc.). Depending on the state, not all utilities may be marked out (e.g., gravity sewers are excluded from New Jersey's underground facility protection law). Other facility operators may not have the resources to participate in the one-call system. The local municipality or utility must be contacted to determine the location of these underground facilities.

- Determine what clearances are required between the location of the underground facility and the proposed excavation (clearances vary by state).

- Measure distance between proposed excavation and underground facility to ensure that proper clearance distance is maintained.

- Begin excavation activities. Be cautious during the initial stages of the excavation activity. Be alert for signs that an underground facility may be present. Look for changes in the soil characteristics such as the presence of fill materials or non-native materials. Be alert for visual and aural cues that may indicate the presence of an underground facility (metallic sounds, changes in drilling progress, etc.). The party responsible for locating the underground facilities must maintain the markouts. Document maintenance of markouts in the field logbook and photographically.

- If an underground facility is struck or breeched, cease operations immediately. Remove all personnel from the area, contact the one-call system, inform your supervisor, contact the facility operator, and follow the emergency procedures in the site-specific health and safety plan.

5.0 References

New Jersey Statute, Title 48. 1997. Public Utilities, Chapter 2. Board of Public Utility Commissioners, Article 9. Underground Facility Protection.

New York State Department of Public Service, Safety Section, New York Department of Public Service, Rule 753. *Duties of Excavators*, Sections 753-3.2 - 753-3.17.

Dig Safe, Inc. website, www.digsafe.com.

New Jersey One Call website, www.nj1-call.org.

New York State Electric and Gas (NYSEG) website, safety section, www.nyseg.org.

Dig Safely website, links to state laws and one-call contacts, www.digsafely.com.

Field Logbook Content and Control	SOP 4-1 Revision: 8 Date: February 2015

Approved: *Signature* **Technical Review:** Robert Alexander

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to set criteria for content entry and form of field logbooks. Field logbooks are an essential tool to document field activities for historical and legal purposes.

2.0 Background

2.1 Definitions

Biota - The flora and fauna of a region.

Magnetic Declination Corrections - Compass adjustments to correct for the angle between magnetic north and geographical meridians.

2.2 Discussion

Information recorded in field logbooks includes field team names; observations; data; calculations; date/time; weather; and description of the data collection activity, methods, instruments, and results. Additionally, the logbook may contain deviations from plans and descriptions of wastes, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

3.0 General Responsibilities

Field Team Leader (FTL) - The FTL is responsible for ensuring that the format and content of data entries are in accordance with this procedure.

Site Personnel - All CDM Smith employees who make entries in field logbooks during onsite activities are required to read this procedure before engaging in this activity. The FTL will assign field logbooks to site personnel who will be responsible for their care and maintenance. Site personnel will return field logbooks to the records file at the end of the assignment.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities should be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

4.0 Required Equipment

- Site-specific plans
- Field logbook
- Indelible black or blue ink pen
- Ruler or similar scale

5.0 Procedures

5.1 Preparation

In addition to this SOP, site personnel responsible for maintaining logbooks must be familiar with all procedures applicable to the field activity being performed. These procedures should be consulted as necessary to obtain specific information about equipment and supplies, health and safety, sample collection, packaging, decontamination, and documentation requirements. These procedures should be available electronically, or located at the field office or vehicle for easy reference.

Field logbooks shall be bound with lined, consecutively numbered pages. All pages must be numbered before initial use of the logbook. Before use in the field, each logbook will be marked with a specific document control number issued by the document control administrator, if required by the contract quality implementation plan (QIP). Not all contracts require document control numbers. The following information shall be recorded on the cover of the logbook:

- Field logbook document control number (if applicable).
- Start date of entries.

- Activity (if the logbook is to be activity-specific), site name, and location.
- Name of CDM Smith contact and phone number(s) (typically the project manager).
- End date of entries.
- In specific cases, special logbooks may be required (e.g., waterproof paper for stormwater monitoring).

The first few (approximately three) pages of the logbook will be reserved for a table of contents (TOC). Mark the first page with the heading and enter the following:

Table of Contents

Date/Description	Pages
(Start Date)/Reserved for TOC	1-3

The remaining pages of the table of contents will be designated as such with "TOC" written on the top center of each page. The table of contents should be completed as activities are completed and before placing the logbook in the records file.

5.2 Operation

Requirements that must be followed when using a logbook:

- Record work, observations, quantities of materials, calculations, drawings, and related information directly in the logbook. If data collection forms are specified by an activity-specific plan, this information does not need to be duplicated in the logbook. However, any forms used to record site information must be referenced in the logbook.
- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.
- Do not erase or blot out any entry at any time. Indicate any deletion by a single line through the material to be deleted. Initial and date each deletion. Take care to not obliterate what was written previously.
- Do not remove any pages from the book.
- All entries must be clearly written and legible.

Specific requirements for field logbook entries include:

- Initial and date each page.
- Sign and date the final page of entries for each day.
- Initial and date all changes.
- Multiple authors must sign out the logbook by inserting the following:
  Above notes authored by:
  - (Sign name)
  - (Print name)
  - (Date)
- A new author must sign and print his/her name before additional entries are made.
- Draw a diagonal line through the remainder of the final page at the end of the day.
- Record the following information on a daily basis:
  - Date and time
  - Name of individual making entry
  - Names of field team and other persons onsite
  - Description of activity being conducted including station or location (i.e., well, boring, sampling location number) if appropriate
  - Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction, and speed) and other pertinent data
  - Level of personal protection used
  - Serial numbers of instruments
  - Equipment calibration information
  - Serial/tracking numbers on documentation (e.g., carrier air bills)

Entries into the field logbook shall be preceded with the time (written in military units) of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g., data logger) or on a separate form required by an operating procedure. In these cases, the logbook must reference the automatic data record or form.

At each station where a sample is collected or an observation or measurement made, a detailed description of the location of the station is required. Use a compass (include a reference to magnetic declination corrections), scale, or nearby survey markers, as appropriate. A sketch of station location may be warranted. All maps or sketches made in the logbook should have descriptions of the features shown and a direction indicator. It is preferred that maps and sketches be oriented so that north is toward the top of the page. Maps, sketches, figures, or data that will not fit on a logbook page should be referenced and attached to the logbook to prevent separation.

Other events and observations that should be recorded include:

- Changes in weather that impact field activities.
- Deviations from procedures outlined in any governing documents. Also record the reason for any noted deviation.
- Problems, downtime, or delays.
- Upgrade or downgrade of personal protection equipment.
- Visitors to the site.

5.3 Post-Operation

To guard against loss of data as a result of damage or disappearance of logbooks, completed pages shall be periodically photocopied or scanned (weekly, at a minimum) and forwarded to the field or project office. Other field records shall be photocopied or scanned and submitted regularly and as promptly as possible to the office. When possible, electronic media such as flash drives or disks should be copied and forwarded to the project office. Follow the records control procedures specified in the site-specific plan.

At the conclusion of each activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated and that corrections were made properly (single lines drawn through incorrect information, then initialed and dated). The completed logbook shall be submitted to the records file.

6.0 Restrictions/Limitations

Field logbooks constitute the official record of onsite technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by CDM Smith personnel and their subcontractors. They are documents that may be used in court to indicate dates, personnel, procedures, and techniques employed during site activities. Entries made in these logbooks should be factual, clear, precise, and nonsubjective. Field logbooks, and entries within, are not to be used for personal use.

7.0 References

EPA 2007. Contract Laboratory Program Guidance for Field Samplers. Office of Superfund Remediation and Technology Innovation. OSWER 9240.0-44. EPA 540-R-07-06. July.

US EPA. 2014. Region 4. The Field Branches Quality System and Technical Procedures, Soil Sampling. SESDPROC-010-R5. May.

Photographic Documentation of Field Activities	SOP 4-2 Revision: 9 Date: February 2015
Approved: *Signature*	**Technical Review:** Robert R. Alexander

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to provide standard guidelines and methods for photographic documentation, which include digital photography and recordings of field activities and site features (geologic formations, core sections, lithologic samples, water samples, general site layout, etc.). This SOP is intended for circumstances when formal photographic documentation is required. Based on project requirements, it may not be applicable for all photographic activities.

2.0 Background

2.1 Definitions

Photographer - A photographer is the camera operator of digital photography or recordings whose primary function with regard to this SOP is to produce documentary or data-oriented visual media.

Identifier Component - Identifier components are visual components used within a photograph such as visual slates, reference markers, and pointers.

Standard Reference Marker - A standard reference marker is a reference marker that is used to indicate a feature size in the photograph and is a standard length of measure, such as a ruler, meter stick, etc. In limited instances, if a ruled marker is not available or its use is not feasible, it can be a common object of known size placed within the visual field and used for scale.

Slates - Slates are blank white index cards, marker boards, or paper used to present information pertaining to the subject/procedure being photographed. Letters and numbers on the slate will be bold and written with black indelible marking pens.

Arrows and Pointers - Arrows and pointers are markers/pointers used to indicate and/or draw attention to a special feature within the photograph.

Contrasting Backgrounds - Contrasting backgrounds are backdrops used to lay soil samples, cores, or other objects on for clearer viewing and to delineate features.

Date Stamp - A date stamp is a built-in feature that will record the date and time directly on a digital image or recording.

2.2 Associated Procedures

- SOP 4-1, *Field Logbook Content and Control*

2.3 Discussion

Digital photographs and recordings made during field investigations are used as an aid in documenting and describing site features, sample collection activities, equipment used, and possible lithologic interpretation. This SOP is designed to illustrate the format and desired placement of identifier components, such as visual slates, standard reference markers, and pointers. The use of a photographic logbook and standardized entry procedures are also outlined. These procedures and guidelines will minimize potential ambiguities that may arise when viewing the images or recordings and ensure the representative nature of the photographic documentation.

3.0 General Responsibilities

Field Team Leader - The field team leader (FTL) is responsible for ensuring that the format and content of photographic documentation are in accordance with this procedure. The FTL is responsible for directing the photographer to specific situations, site features, or operations that the photographer will be responsible for documenting.

Photographer - The photographer shall seek direction from the FTL and regularly discuss the visual documentation requirements and schedule. The photographer is responsible for maintaining a logbook per Sections 5.1, 5.2.4, and 5.3.1 of this SOP. Responsibilities will be defined in the project sampling plan.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

A general list of equipment that may be used:

- Digital camera and appropriate storage media
- Extra batteries for camera
- Digital video camera and appropriate storage media (e.g., SD card)
- Logbook
- Indelible black or blue ink pen
- Standard reference markers (e.g. ruler)
- Slates or marker boards
- Arrows or pointers
- Contrasting backgrounds
-
-
-

5.0 Procedures

5.1 Documentation

Documentation requirements for digital photographs and recordings should be specified in the site-specific plan. Otherwise, a commercially available, bound logbook will be used to log and document photographic activities. Review SOP 4-1, *Field Logbook Content and Control* and prepare all supplies needed for logbook entries.

Note: A separate photographic logbook is not required. A portion of the field logbook may be designated as the photographic log and documentation section.

Field Health and Safety Considerations

There are no hazards that an individual will be exposed to specific to photographic documentation. However, site-specific hazards may arise depending on location or operation. Personal protective equipment used in this operation will be site-specific and dictated through requirements set by the site safety officer, site health and safety plan, and/or prescribed by the CDM Smith Corporate Health and Safety Program. The photographer should contact the site safety officer for health and safety orientation before commencing field activities. The site health and safety plan must be read before entry to the site, and all individuals must sign the appropriate acknowledgement that this has been done.

The photographer should be aware of any potential physical hazards while photographing the subject (e.g., traffic, low overhead hazard, edge of excavation).

5.2 Operation

5.2.1 General Photographic Activities in the Field

The following sections provide general guidelines that should be followed to visually document field activities and site features using digital still and video cameras. Listed below are general suggestions that the photographer should consider when performing activities under this SOP:

- The photographer should be prepared to make a variety of shots, from close-up to wide-angle. Many shots will be repetitive in nature or format, especially close-up site feature photographs. Consideration should therefore be given to designing a system, camera settings, or technique that will provide a reliable repetition of performance.

- No preference of digital storage medium is specified and is left to the discretion of the photographer.

- Digital cameras have multiple photographic quality settings. A camera that obtains a higher resolution (quality) has a higher number of pixels and will store a fewer number of photographs per digital storage medium. Project resolution requirements should be determined prior to implementing field work.

5.2.2 General Guidelines for Still Photography

Caption Information
Unless otherwise specified in a site-specific plan, all still photographs will have a full caption added after the images are downloaded on a photo log sheet. The caption should contain the following information:

- Photograph sequence number
- Date and time
- Photographer
- Description of activity/item shown (e.g., name of facility/site, specific project name, project number)
- Direction (if applicable)

When directed by the sampling plan, a standard reference marker should be used in all documentary visual media. While the standard reference marker will be predominantly used in close-up feature documentation, inclusion in all scenes should be considered.

Digital images should be downloaded at least once each day to a personal computer; the files should be in either “JPEG” or “TIFF” format. Files should be renamed at the time of download to correspond to the logbook or as directed in the site-specific plan. It is recommended the electronic files be copied to a compact disc for backup.

Close-Up and Feature Photography
When directed by the sampling plan, close-up photographs should include a standard reference marker of appropriate size as an indication of the feature size and contain a slate or marker board marked with the site name and any identifying label, such as a well number or core depth, that clearly communicates to the viewer the specific feature being photographed.

Feature samples, core pieces, and other lithologic media should be photographed as soon as possible after they have been removed from their in situ locations. This enables a more accurate record of their initial condition and color. When directed by the sampling plan, include a standard reference color strip (color chart such as Munsell Soil Color Chart) within the scene. This is to be included for the benefit of the viewer of the photographic document and serves as a reference aid to the viewer for formal lithologic observations and interpretations.

Site Photography
Site photography, in general, will consist predominantly of medium- and wide-angle shots. If required by the sampling plan, a standard reference marker should be placed adjacent to the feature or, when this is not possible, within the same focal plane.

While it is encouraged that a standard reference marker and slate/marker board be included in the scene, it is understood that situations will arise that preclude their inclusion within the scene. This will be especially true of wide-angle shots. In such a case, the image number shall be entered in the photographic logbook along with other information pertinent to the scene.

5.2.3 General Photographic Documentation Using Digital Video Cameras
Documentary digital recordings of field activities may include an audio slate for all scenes. At the beginning of each video session, an announcer will recite the following information: date, time (in military units), photographer, site ID number, and site location. This oral account may include any additional information clarifying the subject matter being recorded.

A standard reference marker may be used when taking close-up shots of site features with a video camera. The scene may also include a caption/slate. It should be placed adjacent and parallel to the feature being photographed.

It is recommended that a standard reference marker and slate/marker board be included in all scenes. The caption information is vital to the value of the documentary visual media and should be included. If it is not included within the scene, it should be placed before the scene.

Original video recordings will not be edited. This will maintain the integrity of the information contained on the videotape or DVD. If editing is desired, a working copy of the original video recording can be made.

A digital recording filename should be created with the appropriate identifying information (project name, project number, date, location, etc.).

5.2.4 Photographic Documentation

If required by the site-specific plan, photographic activities must be documented in a photographic logbook or in a section of the field logbook. The photographer will be responsible for making proper entries.

In addition to following the technical standards for logbook entry as referenced in SOP 4-1, the following information should be maintained in the appropriate logbook:

- Photographer name.
- Sequential tracking number for each photograph taken (the camera-generated number may be used).
- Date and time (military time).
- Location.
- A description of the activity/item photographed.
- If needed, a description of the general setup, including approximate distance between the camera and the subject, may be recorded in the logbook.
- Record as much other information as possible to assist in the identification of the photographic document.

5.3 Post Operation

The photographer shall be responsible for downloading image files or recordings to the project files.

As required, the photographer(s) will ensure that the appropriate logbook has been completely filled out and maintained as outlined in SOP 4-1. Images and recordings will be handled according to contract records requirements. The project manager will ensure their proper distribution. Completed pages of the appropriate logbook will be copied weekly and submitted to the project files.

6.0 Restrictions/Limitations

This document is designed to provide a set of guidelines to ensure that an effective and standardized program of visual documentation is maintained.

It is not within the scope of this document to provide instruction in photographic procedures, nor is it within the scope of this document to set guidelines for presentation or “show” photography.

The procedures outlined herein are general by nature. The photographer is responsible for specific operational activity or procedure described in site-specific plans. Questions concerning specific procedures or requirements should be directed to the project manager or FTL.

Note: Some sites do not permit photographic documentation. Check with the site contact for any restrictions.

7.0 References

U. S. Army Corps of Engineers. 2001. *Requirements for the Preparation of Sampling and Analysis Plans*, EM 200-1-3. Appendix F. February.

U. S. Environmental Protection Agency. 1992. National Enforcement Investigations Center. *Multi-Media Investigation Manual*, EPA-330/9-89-003-R. p. 85. Revised March.

_____. 2013. Region 4. The Field Branches Quality System and Technical Procedures, Soil Sampling. SESDPROC-005-R2. January.

Well Development and Purging	SOP 4-3 Revision: 7 Date: February 2015

Approved: Signature **Technical Review:** David Schroeder

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define the procedural requirements for well development and purging.

2.0 Background

Monitoring wells are developed to repair damage to the formation caused by drilling activities and to settle and remove fines from the filter pack. Wells shall not be developed for at least 24 to 48 hours after completion when a cement bentonite grout is used to seal the annular space; however, wells may be developed before grouting if conditions warrant. Wells are purged immediately before groundwater sampling to remove stagnant water and to sample representative groundwater conditions. Wells shall be sampled within 3 hours of purging (optimum) to 24 hours after purging (maximum, for low recharge conditions).

2.1 Associated Procedures

- SOP 1-6, *Water Level Measurement*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that field personnel are trained in the use of this procedure and for verifying that development and purging are carried out in accordance with this procedure.

Field Team Leader - The field team leader is responsible for complying with this procedure.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Required Equipment

4-1 General

- Pump, pump tubing, or bailer and rope or wire line
- Power source (e.g., generator), if required
- Electronic water-level meter
- Temperature, conductivity, pH, and turbidity meters
- Personal protective equipment as specified in the site-specific health and safety plan
- Decontamination supplies, as required, according to SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*
- Disposal drums, if required
- Photoionization detector (PID) or equivalent as specified in site-specific health and safety plan

5.0 Procedures

5.1 Well Development

The following steps must be followed when developing wells:

1. Review site-specific health and safety plan and project plans before initiating sampling activity.

2. Don personal protective clothing and equipment as specified in the site-specific health and safety plan.

3. Open the well cover and check condition of the wellhead, including the condition of the surveyed reference mark, if any.

4. Monitor the air space at the wellhead, using a PID or equivalent, as soon as well cover is removed according to health and safety requirements.

5. Determine the depth to static water level and depth to bottom of the well (SOP 1-6).

6. Prepare the necessary equipment for developing the well. There are a number of techniques that can be used to develop a well. Some of the more common methods are bailing, overpumping, backwashing, mechanical surging, surge and pump, wire brush, swabbing, and high-velocity jetting. All of these procedures are acceptable; however, final approval of the development method rests with the appropriateness of a specific method to the geologic conditions, well type and approved work plans, field sampling plans, etc.

7. For screened intervals longer than 10 feet (3 meters [m]), develop the well in 2- or 3-foot (0.75- or 1-m) intervals from bottom to top. This will ensure proper packing of the filter pack.

8. Continue well development until produced water is clear and free of suspended solids, as determined by a turbidity meter or when pH, conductivity, and temperature have stabilized. Record pertinent data in the field logbook and on appropriate well development forms. Remove the pump assembly or bailers from the well, decontaminate (if required), and clean up the area. Lock the well cover before leaving. Containerize and/or dispose of development water as required by the site-specific plans.

5.2 Volumetric Method of Well Purging

The following steps shall be followed when purging a well by the volumetric method:

1. Review site-specific health and safety plan and project plans before initiating well purging activity.

2. Don personal protective clothing and equipment as specified in the site-specific health and safety plan.

3. Open the well cover and check condition of the wellhead, including the condition of the surveyed reference mark, if any.

4. Monitor the air space at the wellhead, using a PID or equivalent, as soon as well cover is removed according to health and safety requirements.

5. Determine the depth to static water level and depth to bottom of well casing according to SOP 1-6, *Water Level Measurement*. Calculate the volume of water within the well bore using the following formula (or equivalent):

$$7.4805 \left[\frac{D^2\pi}{(4)} \right] dH = \text{volume (in gallons)}$$

where
D = casing diameter in feet. (***Note***: This equation is used for grouted wells with short screens. For wells with long screens and/or ungrouted wells, the D = borehole diameter in feet).
dH = the distance from well bottom to static water level in feet.
π = 3.1416.

Alternatively, the volume may be determined using a casing volume per foot factor for the appropriate diameter well, similar to that in Table 1. The water level is subtracted from the total depth, providing the length of the water column. This length is multiplied by the factor in Table 1 corresponding to the appropriate well diameter, providing the amount of water, in gallons, contained in the well.

TABLE 1
WELL CASING DIAMETER vs. VOLUME

WELL CASING DIAMETER (inches) vs. VOLUME (gals.)/FEET of WATER	
CASING	GALLONS/FT
1	0.041
2	0.163
3	0.367
4	0.653
5	1.02
6	1.469
7	1.999
8	2.611
9	3.305
10	4.08
11	4.934
12	5.875

Note: Record all data and calculations in the field logbook.

6. Prepare the pump and tubing, or bailer, and lower it into the casing.

7. Remove the number of well volumes specified in the site-specific plans. Generally, three to five well volumes will be required. Conductivity, pH, temperature, and turbidity shall be measured and recorded. Purging shall continue until the field parameters have stabilized. Groundwater quality parameters are considered stable when three consecutive readings are within ± 0.1 for pH, ±3 percent for conductivity, ±10 mv for redox potential (ORP), and ±10 percent for turbidity if greater than 10 NTU, in accordance with site-specific plans. Efforts shall be made to get turbidity below 10 NTU, especially if groundwater samples are to be collected for metals or PCB analyses.

8. In low recharge aquifers, the following steps shall be followed: (1) If the initial water level is less then 10 feet above the top of the screen, then purge to dryness and allow sufficient recharge to collect samples. (2) If initial water level in the well is more than 10 feet above the top of the screen, then care shall be taken to prevent the dewatering of the screened interval. (3) Continue purging until the water level is between 1 and 5 feet above the top of the screen. (4) Allow well to recharge then continue purging until at least 1 full initial well volume has been purged. (5) Record pertinent data in the field logbook.

9. Groundwater sampling shall be performed immediately upon completion of purging (unless time for recharge is required for low-recharge wells) using the same equipment that was used for purging. Unfiltered samples shall be collected first, beginning with volatile organic compounds (VOCs). After all unfiltered samples have been collected, a 0.45 micron in-line filter shall be installed in the discharge line for collection of filtered samples, if required.

10. After sampling activities have been completed, remove the pump assembly or bailer from the well, decontaminate it (if required), and clean up the site. Lock the well cover before leaving. Containerize and/or dispose of development water as required by the site-specific plan.

5.3 Indicator Parameter Method of Well Purging

1. Review site-specific health and safety plan and project plans before initiating well purging activity.

2. Don personal protective clothing and equipment as specified in the site-specific health and safety plan.

3. Open the well cover and check the condition of the wellhead, including the condition of the surveyed reference mark, if any.

4. Monitor the air space at the wellhead, using a PID or equivalent, as soon as well cover is removed according to health and safety requirements.

5. Determine the depth to static water level and depth to bottom. Set up surface probe(s), (e.g., pH, conductivity) at the discharge orifice or dedicated probe port of the pump assembly or within the flow-through chamber. Allow probe(s) to equilibrate according to manufacturer's specifications. Record the equilibrated readings in the field logbook.

6. Assemble the pump and tubingand lower into the casing.

7. Begin pumping or bailing the well. Record indicator parameter readings for every 5 minutes or purge volume, whichever is sooner. Maintain a record of the approximate volumes of water produced. Care shall be taken to minimize drawdown (0 to 0.2 feet).

8. Continue pumping or bailing until indicator parameter readings remain stable within ± 0.1 for pH, ±3 percent for conductivity, ±10 mv for redox potential (ORP), and ±10 percent for turbidity if greater than 10 NTU for three consecutive recording intervals and a minimum of 1 well volume is removed, or in accordance with site-specific plans. Purging shall continue until the discharge stream is clear or turbidity becomes asymptotic-low or meets project requirements.

9. For a low recharge aquifer, follow the guidelines of Section 5.2, Paragraph 7.

10. Groundwater sampling shall be performed immediately upon completion of purging (unless time for recharge is required for low-recharge wells) using the same equipment that was used for purging. Unfiltered samples shall be collected first, beginning with VOCs. After all unfiltered samples have been collected, a 0.45 micron in-line filter shall be installed in the discharge line for collection of filtered samples, if required.

11. Remove the pump assembly or bailer from the well, decontaminate (if required), and clean up the site. Lock the well cover before leaving. Containerize and/or dispose of development water as required by the site-specific plans.

6.0 Restrictions/Limitations

Where flammable, free, or emulsified product is expected, or known to exist on or in groundwater, use intrinsically safe electrical devices only and place portable power sources (e.g., generators) 50 feet (15 m) or further from the wellhead and disposal drums.

7.0 References

American Society for Testing and Materials. 2005. Designation: D 5521, *Standard Guide for Development of Groundwater Monitoring Wells in Granular Aquifers,* Rev. 5, November.

U. S. Army Corps of Engineers. 1998. *Monitoring Well Design, Installation, and Documentation at Hazardous Toxic, and Radioactive Waste Sites, EM 1110-1-4000*, Chapter 6. November 1.

U. S. Environmental Protection Agency, Region III, 1997. *Low-Flow Purging and Sampling of Groundwater Monitoring Wells*, Bulletin No. QAD023, Philadelphia, Pennsylvania. October.

U. S. Environmental Protection Agency. 2002. *Groundwater Sampling Guidelines for Superfund and RCRA Project Managers.* Ground Water Forum Issue Paper, EPA 542-S-02-001, OSWER, Technology Innovative Office, Washington, D.C. May.

U. S. Environmental Protection Agency, Region IX, 2004. Groundwater Well Sampling, Field Sampling Guidance Document #1220.

Design and Installation of Monitoring Wells in Aquifers	SOP 4-4 Revision: 8 Date: February 2015

Approved: Signature **Technical Review:** Michael Valentino

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to provide guidelines for the installation of groundwater monitoring wells. These guidelines will help to produce consistency of approach in the design and installation of monitoring wells. Individual installations will probably vary in some respects since they may encounter differing hydrogeologic conditions.

2.0 Background

2.1 Definitions

Monitoring Well Installation - The act of installing well casing, screen, filter pack, bentonite seal, grout, and other specified materials in a borehole to construct a complete monitoring well.

2.2 Associated Procedures

- SOP 3-5, *Lithologic Logging*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-2, *Photographic Documentation of Field Activities*
- SOP 4-3, *Well Development and Purging*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.3 Discussion

This SOP is intended to cover the installation of monitoring wells for use in conducting a variety of environmental investigations. It is intended to be a general guideline listing the types of materials and methods to be considered when a well is installed. Materials are not specified in detail since it is likely there will be wide variability required to meet the needs of individual site conditions or specific clients. Ideally, the well shall not alter the medium that is being sampled.

3.0 General Responsibilities

Site Manager - Translates client's requirements into technical direction of project. Sets technical criteria, reviews and approves technical progress, and ensures that all participating personnel have proper training. ***Note***: Other titles such as project manager may be used.

Field Team Leader (FTL) - Supervises field operations. Ensures that all necessary equipment including safety equipment is available and functioning properly before project operations begin. Ensures that all necessary personnel are mobilized on time. Maintains daily log of activities each work day.

Field Geologist - Collects and maintains data and completes Monitoring Well Construction Forms. Coordinates and consults with site manager on decisions relative to unexpected encounters during well installation and deviation from this SOP. Directs overall activities of drill and support subcontractors.

Drilling Subcontractor - Provides necessary personnel, equipment, and services to meet terms of the contract.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Required Equipment and Materials

4.1 Required Equipment

- Field logbook
- Monitoring Well Construction Forms
- Measuring tape

4.2 Required Construction Materials

General - The materials that are used in the construction of a monitoring well and that come in contact with the groundwater shall not measurably alter the chemical quality of a groundwater sample. The well casing and well screen shall be steam cleaned (if appropriate for the selected material) before well installation or certified clean from the manufacturer and delivered to the site in protective wrapping. If required by the site specific plans, samples of the cleaning water, drilling fluids, filter pack, annular seal, and mixed grout may be retained to be analyzed if groundwater contamination as the result of well installation is suspected. If required, these samples can serve as quality control checks until the completion of at least one round of groundwater quality sampling and analysis.

Water - Water, which may be used in the well completion process, shall be obtained from a source that does not contain constituents that could compromise the integrity of the well installation. If required by the site specific plans, a certificate of analysis shall be provided with the water, or a sample of the water shall be analyzed and documented as contaminant-free.

Surface (Isolation) Casing - Surface, or isolation, casing may be required to isolate an upper aquifer while drilling and installing deep wells. The isolation casing usually consists of black steel or polyvinyl chloride (PVC). Surface casings shall be large enough to allow a minimum annular space of 2 inches between it and the well casing. Segments of black steel casings are typically welded together as the casing is lowered down the borehole. PVC isolation casings are either flush-threaded or have a bell shape at one end so that sections slip together and are held with small stainless steel screws. Casings shall be grouted in place and allowed to set for 12 to 24 hours before advancing the borehole below the surface casing.

Well Screen - The well screen shall be new and composed of materials most suited for the environment being monitored. The screened interval shall be plugged at the bottom. The plug shall be of the same material as the bottom section of screen and shall be securely attached, making a positive seal. This assembly must have the capability to withstand well installation and development stresses without becoming dislodged or damaged. The length of the well screen slotted area shall be appropriate for the interval to be monitored including some allowance for changes in elevation of the water table. Before installation, the casing string and associated equipment shall be cleaned with steam or high-pressure hot water, if not certified cleaned. Well screens shall be stainless steel or PVC, as appropriate. Fluoropolymer materials may be substituted if necessary because of the potential for incompatible chemical reactions between contaminants and the stainless steel screen, or if stainless steel constituents are possible site contaminants. The minimum inside diameter of the well screen shall be chosen based on the particular application. Well screens shall be flush threaded per American Society for Testing and Materials (ASTM) standards. Glued or solvent-welded joints may not be used since glues and solvents may alter the chemistry of the water samples.

The slot size of the well screen shall be determined relative to the grain-size analysis of the stratum to be monitored and the gradation of the filter pack material. In granular, noncohesive, strata that falls in easily around the screen, filter packs may not be necessary. In these cases of natural development, the slot size of the well screen is to be determined using the grain size of the surrounding strata. The slot size and arrangement shall retain at least 90 percent of the filter pack.

Casing - The well casing will be PVC, stainless steel, or some other appropriate material and will extend from the screen to the surface. The type of casing and wall thickness shall be adequate to withstand the forces of installation. Several different casing sizes may be required depending on the subsurface geologic conditions. The diameter of the casing for filter packed wells shall be selected so that a minimum annular space of 2 inches is maintained between the casing and the borehole wall. The diameter of the casings in multi-cased wells shall be selected so that a minimum annular space of 2 inches is maintained between casing strings and between the outer casing and the borehole (e.g., a 2-inch-diameter well screen will require first setting a 6-inch-diameter casing in a 10-inch-diameter boring). Under difficult drilling conditions (collapsing soils, rock, or cobbles), it may be necessary to advance temporary casing. Under these conditions, a smaller space may be maintained. The ends of each casing section shall be flush-threaded.

Primary Filter Pack - The primary filter pack (sand or gravel pack) consists of a clean, well-sorted, rounded granular material of selected grain size and gradation that is installed in the annulus between the screened interval and the borehole wall. The filter pack may be installed along the screened interval using a tremie pipe from the total depth of the well to the designated distance above the top of the screened interval. A filter pack material mostly consisting of siliceous, rather than calcareous, particles is preferred. Select the grading of the filter pack on the basis of the layer of finest material to be screened. A minimum filter pack thickness shall be between 2 to 3 inches and generally shall never be greater than 8 inches. The filter pack shall extend at least 2 to 3 feet above the screened interval or more depending on the screen length to provide for filter pack settlement.

Transition Sand - A layer of fine to very fine sand may be placed on top of the primary filter pack before emplacement of the bentonite seal. The sand shall be of sufficient thickness to prevent bentonite from penetrating to the vicinity of the well screen during placement of the bentonite seal.

Annular Sealants - The materials used to seal the annulus may be prepared as a slurry or used unmixed in a dry pellet form. Sealants shall be selected for compatibility with local geologic, hydrogeologic, climatic, and human-induced conditions anticipated to occur during the life of the well.

Grout - The grout backfill that is placed above the bentonite annular seal shall be a liquid slurry consisting of water, bentonite grout of Volclay or equivalent quality, and portland cement. Bentonite-based grouts are typically used when a more flexible grout is desired (i.e., freeze-thaw). Cement-based grout provides a more rigid installation. A typical bentonite grout mixture is 1 to 1.25 pounds bentonite to 2 pounds of Type I portland cement per gallon of water. Cement-based grout is typically 6 to 7 gallons of water per 94 pound bag of Type I portland cement and 2.7 percent bentonite powder.

Bentonite - Bentonite shall be powdered or pelletized sodium montmorillonite furnished in sacks or buckets from a commercial source and free of impurities that adversely impact water quality in the well. The diameter of pellets selected for monitoring well construction shall be less than one-fifth the width of the annular space into which they are placed to reduce the potential for bridging. Pellets are typically used for placing annular seals, and powdered bentonite is used for mixing in grout slurry.

Cement - Each type of cement has slightly different characteristics that may be appropriate under various physical and chemical conditions. Cement shall generally be portland Type I, Type II, or Type I/II as specified in ASTM C 150. Quick-setting cements containing additives are not allowable for use in monitoring well installation. Additives may leach from the cement and influence the chemistry of the groundwater.

Annular Seal Equipment (Tremie Pipe) - A tremie pipe is used to inject the annular seals and filter pack. Tremie pipes are typically constructed of PVC or galvanized steel. Associated equipment may include a trough or mixing box and “mud pump” to place the material.

Protective Casing - Protective casings are installed on wells placed in overgrown or nontraffic areas. The casings may be made of galvanized steel (or rarely stainless steel) and shall have a lid capable of being secured by a locking device. The inside dimensions of the protective casing shall be at a minimum 4 inches larger than the diameter of the casing to facilitate the installation and operation of sampling equipment. Protective casing shall extend approximately 2 to 3 feet into the ground to anchor it securely. Protective casings are typically set in concrete pads measuring 2 feet x 2 feet to 4 feet x 4 feet and 4 to 6 inches thick depending on client requirements.

Flush-Mount Protective Covers - Flush-mount covers (Christy boxes) are installed over wells placed in paved or manicured areas. The covers are typically 8 inches in diameter with a 1-foot galvanized steel skirt and cast iron bolt-down ring and lid. The covers are installed in concrete pads measuring 2 to 3 feet square that are constructed so as to not impede vehicle traffic and sloped from the center toward the edges to prevent infiltration of run-off water.

5.0 Procedures

5.1 Drilling Methods

The actual methods of drilling at a site will vary depending on site conditions. The method to be used at a site shall be stated in the site-specific plans. Deviations from the methods prescribed in these plans shall be approved by the FTL or designee. Typical drilling methods include air rotary or hammer, mud/fluid rotary, rotosonic, and hollow-stem auger. Drilling with mud or water is least desirable, but the driller shall have the capability to use this method if borehole conditions warrant it. Installation of isolation casing, if required, shall be done by either penetrating the outer casing into the ground by hammer blows or by drilling a borehole. The outer casing shall be set and secured by grouting or other means specified in the site-specific plans. The inner well borehole can then be drilled through the center of the outside casing. The monitoring wells shall be drilled vertical or at an angle if specified in the site-specific plans. The wells shall be drilled to a depth specified in the site-specific plans and may vary based on actual lithologic conditions. The depth to completion shall be approved by the FTL or designee before monitoring well construction. Drillers must prevent grease, oil, and other fluids from the drill rig from coming in contact with the ground around the area of well installation. If air is used during the drilling process (e.g., air rotary), the air supply must be free of oils to prevent introducing contamination to the borehole and groundwater system. This can be accomplished by installing an approved air filter in-line between the air compressor and the air discharge.

Collection of continuous core may be required during borehole drilling. Samples may be collected by several methods depending on project needs and the material being sampled including split-spoon sampler, direct-push, or sonic coring (unconsolidated material), or wire-line coring (bedrock). A description of soil/lithologic materials and drilling observations needs to be recorded on a boring log or in a logbook (SOP 3-5).

5.2 Monitoring Well Installation

5.2.1 Stable Borehole

A stable borehole must be constructed before attempting to install the monitoring well. Steps must be taken to stabilize the borehole before attempting installation if the borehole tends to cave or blow-in, or both. Boreholes that are not straight or are partially obstructed shall be corrected before attempting the installations described herein.

Although all monitoring wells will not be completed exactly alike, there are common elements among them. The Monitoring Well Construction Form(s) (Figures 1, 2, 3, and 4 or equivalent) must be completed by the end of the activity with data obtained through the installation process. Modification of the construction and dimensions on the Well Construction Forms may be needed depending on site-specific conditions. The well construction field form shall be reviewed before initiation of drilling activities to ensure that the required data are collected at appropriate times during drilling and installation.

5.2.2 Well Casing Assembly

The well screen, casing, and bottom plug shall be either certified clean from the manufacturer or decontaminated according to SOP 4-5.

The casing shall be flush-threaded, using Schedule 40 PVC or other suitable monitoring well casing. No adhesives, cements, or lubricants shall be used during casing make-up or during other drilling and well completion operations. Personnel shall take precautions to ensure that grease, oil, or other contaminants that may alter water samples do not contact any portion of the well casing assembly. As a precaution, personnel shall wear a pair of clean gloves while handling the assembly.

Normally, couplings are tightened by hand; however, steam- or high-pressure-cleaned strap wrenches may also be used. Use pipe wrenches with care as they may scar and weaken the pipe. Precautions shall be taken to prevent damage to the threaded joints during installation.

5.2.3 Setting the Well Screen and Casing Assembly in Fluid Filled Holes

When the well screen and casing assembly is lowered to the predetermined level and held in position, the assembly may require a ballast to counteract the tendency to float in the borehole. Ballasting may be accomplished by continuously filling the casing assembly with contaminant-free water. If fluid ballasts are used, the quantity introduced must be recorded in the field logbook. Alternatively, the casing assembly may be slowly pushed into the fluid in the borehole with the aid of hydraulic rams on the drill rig and held in place as additional sections of casing are added to the column. Care must be taken to secure the casing assembly so that personnel safety is ensured during the installation. For wells greater than 100 feet, the assembly shall be installed straight using centralizers at selected intervals.

The casing shall extend to grade or approximately 2 feet above grade, depending on the intended surface completion, and be capped or covered temporarily to deter entrance of foreign materials during completion operations.

5.2.4 Installation of the Primary Filter Pack

Placement of the casing assembly is followed by placing the primary filter pack (consisting of silica sand sized according to the average grain size of the screened formation) into the bottom of the borehole by using a tremie pipe. The primary filter pack is placed in increments as the tremie is gradually raised. The sand pack will be emplaced by the “washdown” gravity method and the depth to the top of the sand pack shall be determined and recorded frequently during the operation to ensure proper placement. The tremie pipe or a weighted line inserted through the tremie pipe can be used to measure the top of the primary filter pack as work progresses. As primary filter pack material is poured into the tremie pipe, potable water is used to help move the sand through the tremie pipe. The quantity of water introduced shall be recorded in a field logbook. If bridging of the primary filter pack occurs, the bridged material shall be broken mechanically before proceeding with the addition of more filter pack material. The depth, volume, and gradation of the primary filter pack will be recorded on the well construction diagram.

Some primary filter pack installations can be accomplished without the use of a tremie pipe. In these instances the filter pack is slowly poured into the borehole from the surface being careful to avoid bridging of the sand as it falls through the water column in the borehole. If this approach is used a weighted tape measure should be lowered into the borehole annulus to measure the increasing height of the filter pack as sand is added to the borehole, making sure to keep the tape measure moving in an up and down motion so the tape does not become buried in the filter pack.

If possible, measuring tape soundings shall be made to ensure the level of the filter pack is in agreement with the calculated volume and that the desired placement is achieved.

If used, temporary casing or auger sections will be withdrawn in increments as the sand pack is emplaced. Care shall be taken to minimize lifting the casing with the withdrawal of the temporary casing/augers. To limit borehole collapse, the temporary casing or hollow-stem auger is usually withdrawn until the lowermost point on the temporary casing or hollow-stem auger is at least 2 feet, but no more than 5 feet, above the filter pack for unconsolidated materials; or at least 5 feet, but no more than 10 feet, for consolidated materials. Ascertain the depth of the sand with an acceptable measuring device or with tremie pipe and verify the thickness of the sand pack. The primary filter pack is typically placed a minimum of 2 feet above the top of the well screen to account for settlement of the filter pack. The volume and depth of the filter pack material shall be measured and recorded on the well construction diagram.

5.2.5 Installation of the Bentonite Seal

A minimum 2-foot-thick bentonite seal shall be emplaced on top of the filter pack or transition sand (if used) and is generally emplaced by gravity feed. The bentonite shall be slowly fed into the annulus and carefully monitored to ensure that bridging is not taking place. Time-release pellets coated with a food-grade coating must be used in deep well applications to prevent premature expansion of the bentonite.

Some clients may require installing the bentonite seal by using a tremie pipe. This type of installation shall be accomplished by washing the bentonite pellets through the tremie pipe with potable water. If the tremie pipe becomes plugged, requiring an increase in pressure to clear it, not less than 20 feet of tremie pipe shall be pulled up to avoid jetting into the sand pack. If the seal is installed above the water level, water shall be added to allow proper hydration of the annular seal (approximately 1 gallon for each linear foot of annular seal). The volume and depth of the bentonite seal material shall be measured and recorded on the well construction diagram.

If possible, measuring tape soundings shall be made to ensure the level of the bentonite seal is in agreement with the calculated volume and that the desired placement is achieved.

5.2.6 Grouting the Annular Space

The following procedures apply to both single- and multi-cased monitoring wells. However, it shall be noted that grouting procedures will vary with the type of well design.

A sufficient volume of grout shall be premixed onsite, according to procedure stipulated by the manufacturer, to compensate for unexpected losses and checked against the known volume of annular space to ensure that bridging does not occur during emplacement. The use of alternate grout materials, including grout containing portland cement, may be necessary to control zones of high grout loss. The mixing (and placing) of grout shall be performed with recorded weights and volumes of materials, according to procedures stipulated by the manufacturer. Lumpy grout shall not be used in an effort to prevent bridging within the tremie and the well. Bentonite-based grout of Volclay or equivalent type shall be mixed to the manufacturer's specifications then pumped into place using minimum pump pressure. All additives to grouts shall be evaluated for their effects on subsequent water samples.

Depending upon the well design, grouting may be accomplished using a pressure grouting technique or by gravity feed through a tremie pipe. With either method, grout is introduced in one continuous operation until grout flows out at the ground surface without evidence of drill cuttings or fluid. The grout backfill shall be injected under pressure using a tremie pipe to reduce the possibility of leaving voids in the annular seal and to displace any liquids and drill cuttings that may remain in the annulus.

Grouting shall begin directly above the bentonite seal, after the bentonite has been adequately hydrated. Grout shall be injected using a tremie pipe. The tremie pipe shall be kept full of grout from start to finish with the discharge end of the pipe completely submerged as it is slowly and continuously lifted. Pump pressure shall be kept to a minimum. Approximately 5 to 10 feet of tremie pipe shall remain submerged during grout emplacement. A staged grouting procedure may be considered if the couplings of the selected casing cannot withstand the shear or if there is collapse stress exerted by the full column of grout as it sets. If used, the temporary casing or hollow-stem auger shall be removed in increments (immediately following each lift of grout installation) well in advance of the time when the grout begins to set. The initial grout mixture must be allowed to cure for at least 12 hours, before attempting to refill (top off) the grout to the surface.

The well shall not be developed until the grout sets and cures for the amount of time necessary to prevent a break in the seal between the grout and casing. The amount of time required (generally 24 to 48 hours) will vary with grout content and climate conditions and shall be documented on the well completion diagram along with the volume and depth of grout used to backfill the annular space.

5.3 Well Protection

Well protection refers specifically to installations made at or above the ground surface to deter unauthorized entry to the monitoring well, prevent damage, and to prevent surface water from entering the annulus.

The protective casing shall extend from below the frost line (at least 2 feet below grade) to slightly above the well casing top. The protective casing shall be sealed and immobilized in concrete that has been placed around the outside of the protective casing above the set grout backfill. The casing shall be positioned and stabilized in a position concentric with the casing. Clearance (usually 6 inches) shall be maintained between the lid of the protective casing and the top of the casing to accommodate sampling equipment. A ¼-inch-diameter weep hole shall be drilled in the protective casing at the ground surface to permit water to drain out of the annular space. This hole will also prevent water freezing between the well protector and the well casing.

All materials used shall be documented on the well construction diagram. The monitoring well identification number shall be clearly visible on the inside and outside of the lid of the protective casing and the outside of the protective casing.

A 3-feet x 3-feet x 6-inch-thick concrete pad, sloped to provide water drainage away from the well, may be placed around the installation. Pad size may vary according to site conditions or client specifications. Three to four 2½-inch-diameter concrete-filled steel posts set at least 24 inches below the surface in concrete shall be equally spaced around the well to protect against damage by vehicular traffic for aboveground well completions. The protective casing and steel posts may be primed and painted with rust-resistant yellow paint. The annulus between the well casing and the protective casing may be filled with sand to approximately 1 foot below the top of the well casing to help stabilize the well casing and prevent the loss of tools or equipment in the annular space.

A flush-mounted, traffic-rated casing or vault is typically used for the surface completion of monitoring wells installed in high-use paved or maintained grass (landscaped) areas. The well box cover shall be finished slightly above pavement or ground surface to prevent water entry. A layer of sand or gravel material shall be placed under the casing/vault to allow infiltrating surface water to drain out.

5.4 Post Operation

5.4.1 Field

At the conclusion of the monitoring well installation activities, all equipment must be decontaminated (according to SOP 4-5) before moving the equipment to a different work location. All water used in the decontamination of drilling equipment will be contained in an appropriate container, if required in the site-specific plans.

5.4.2 Documentation

The Groundwater Monitoring Well Construction Form (Figures 1, 2, 3, and 4 or equivalent) shall be completed by the FTL or designee at the conclusion of the field activity.

Copies of all field notes, the daily logs, and any completed Groundwater Monitoring Well Construction Forms shall be given to the site manager. These records shall be maintained in the project and document control files. At a minimum, all materials used for construction shall be documented by entering identifying numbers (lot numbers, manufacturer's identification, etc.) in the field logbook. Samples of well materials (including grout, sand, etc.) may be archived if specified in the project plans.

6.0 Restrictions and Limitations

None.

7.0 References

American Society for Testing and Materials. 2010. Designation: D6725 − 04 (Reapproved 2010), *Standard Practice for Direct-Push Installation of PrePacked Screen Monitoring Wells in Unconsolidated Aquifers*, Rev. 1. November.

_____. 2010. Designation: D5092 − 04 (Reapproved 2010)e1, *Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers*, Rev. 4. June.

_____. 2010. Designation: D6724 − 04 (Reapproved 2010), *Standard Guide for Installation of Direct-Push Groundwater Monitoring Wells*, Rev. 4. July.

Driscoll, F. G. 1986. *Groundwater and Wells*, 2nd Ed. Johnson Division. St. Paul, Minnesota.

U. S. Army Corps of Engineers. 1998. *Monitoring Well Design, Installation, and Documentation at Hazardous Toxic, and Radioactive Waste Sites*, EM 1110-1-4000. November 1.

Figure 1

Typical Construction Detail of Above-Grade Single-Cased Monitor Well (Not to Scale - Shown as an Example Only)

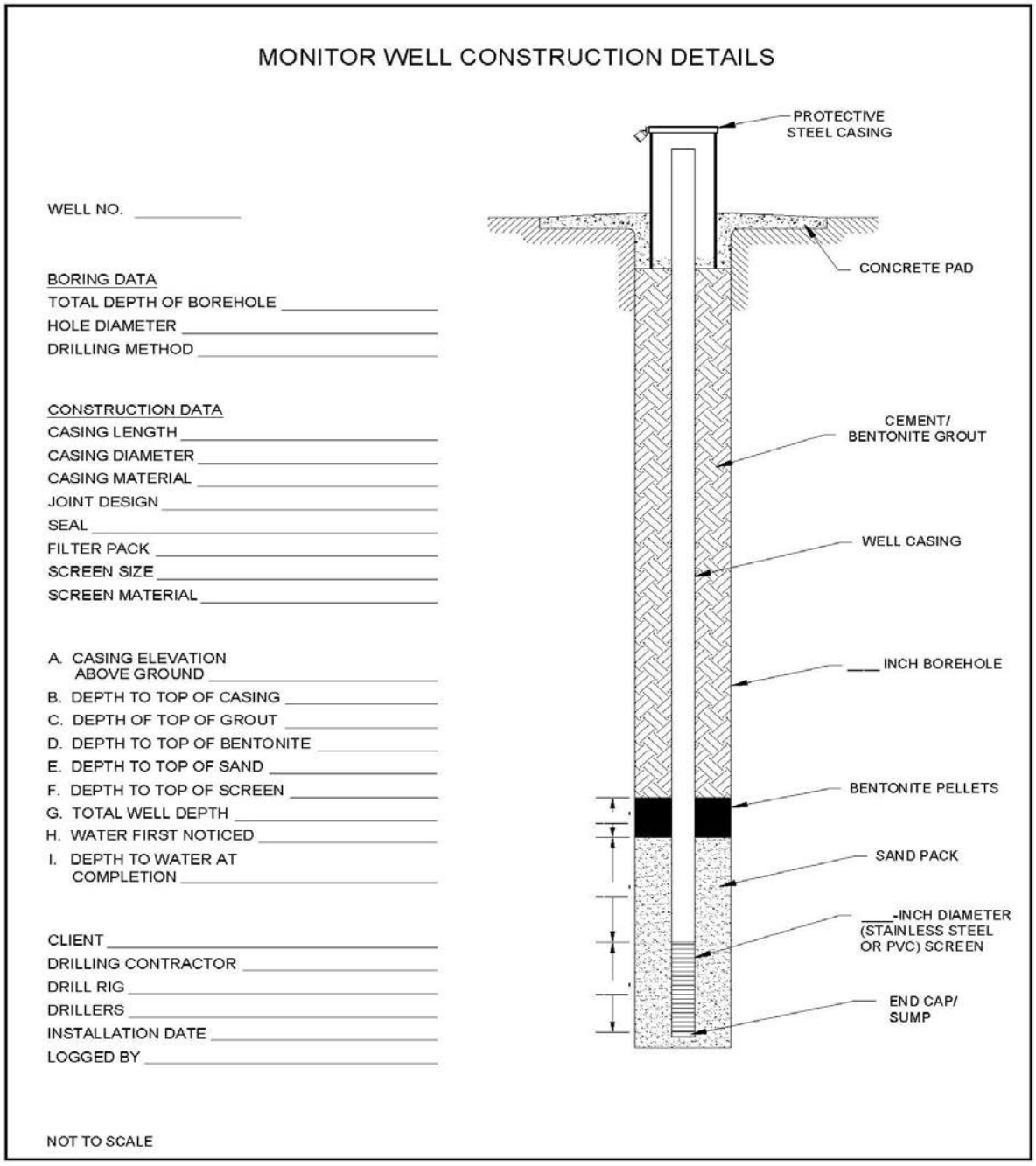


Figure 2

Typical Construction Detail of Flush-Mount Single-Cased Monitor Well
(Not to Scale - Shown as an Example Only)

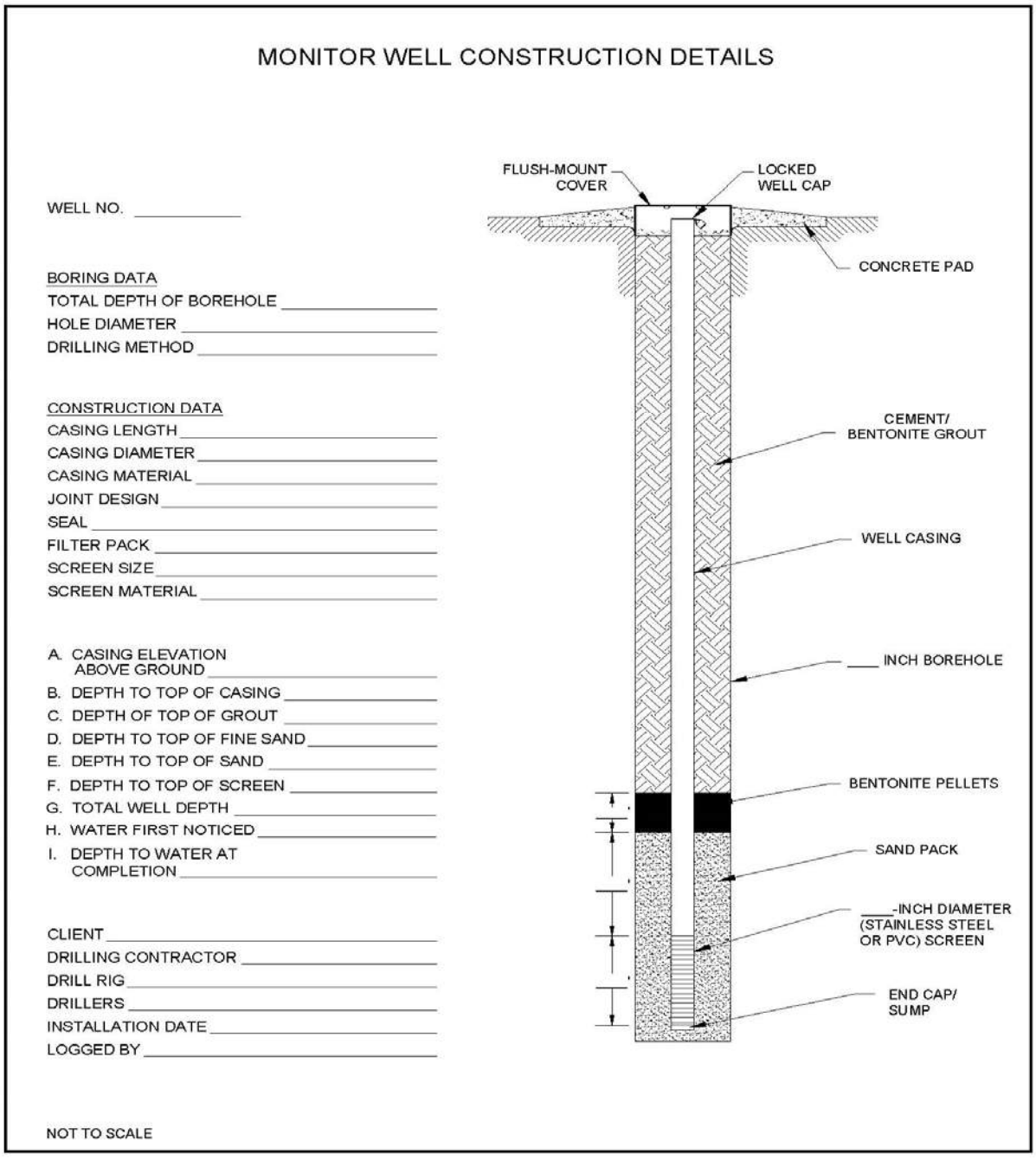


Figure 3

Typical Construction Detail of Above-Grade Double-Cased Monitor Well

(Not to Scale - Shown as an Example Only)

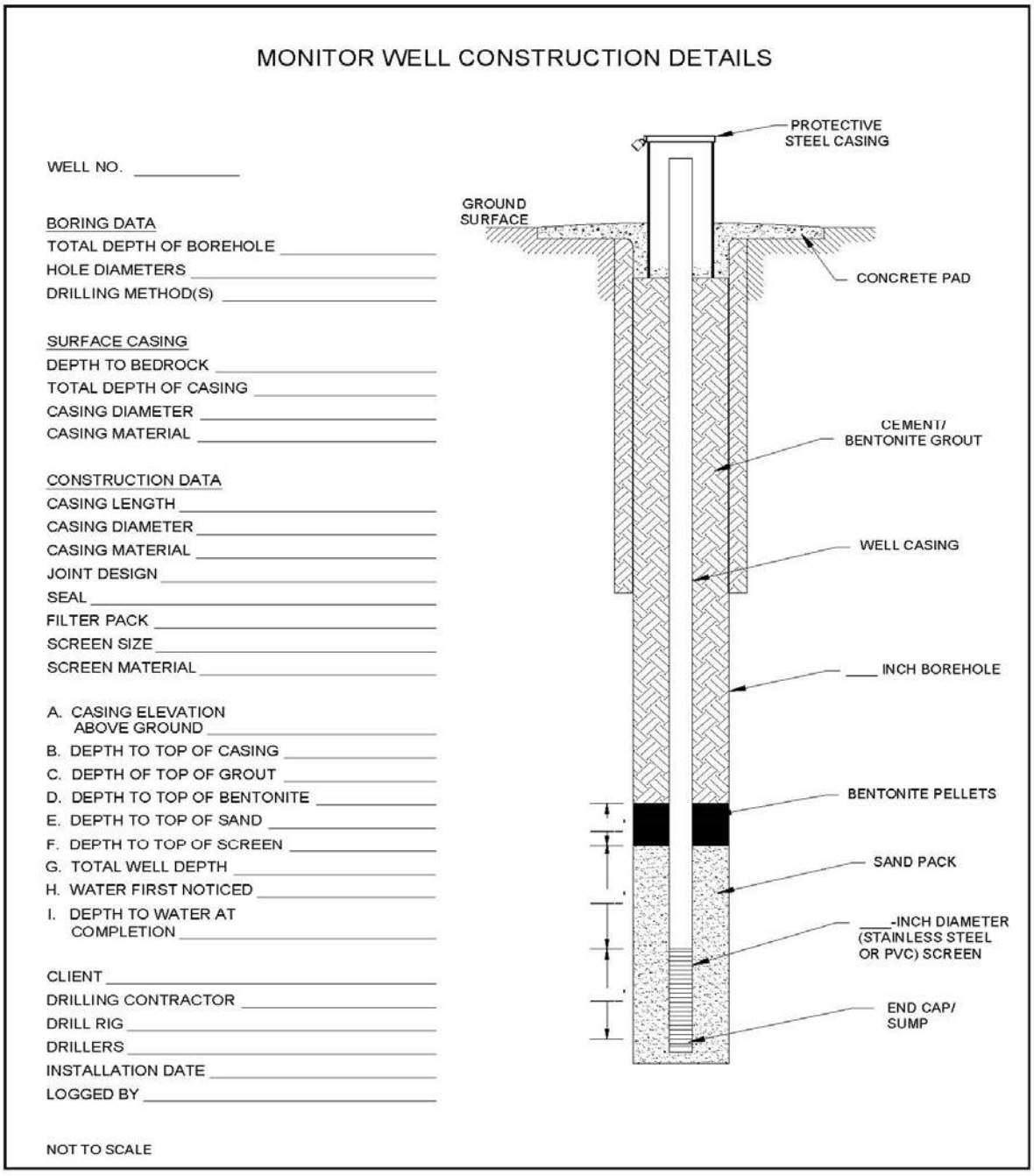


Figure 4

Design and Installation of Monitoring Wells in Aquifers	SOP 4-4 Revision: 8 Date: February 2015

Typical Construction Detail of Flush-Mount Double-Cased Monitor Well
(Not to Scale - Shown as an Example Only)

MONITOR WELL CONSTRUCTION DETAILS

WELL NO. _____

BORING DATA
TOTAL DEPTH OF BOREHOLE _____
HOLE DIAMETERS _____
DRILLING METHOD(S) _____

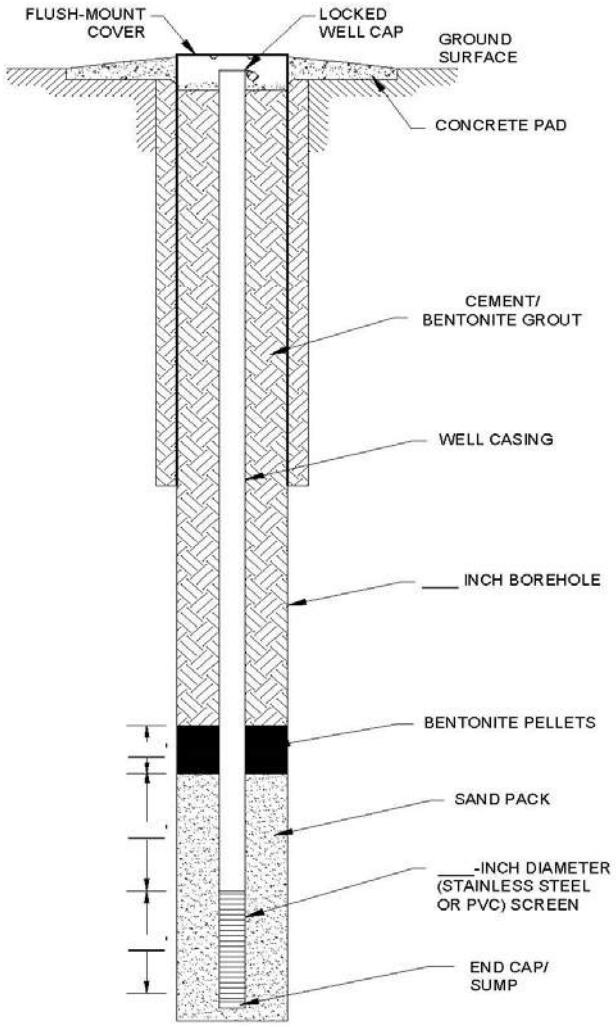
SURFACE CASING
DEPTH TO BEDROCK _____
TOTAL DEPTH OF CASING _____
CASING DIAMETER _____
CASING MATERIAL _____

CONSTRUCTION DATA
CASING LENGTH _____
CASING DIAMETER _____
CASING MATERIAL _____
JOINT DESIGN _____
SEAL _____
FILTER PACK _____
SCREEN SIZE _____
SCREEN MATERIAL _____

A. CASING ELEVATION ABOVE GROUND _____
B. DEPTH TO TOP OF CASING _____
C. DEPTH OF TOP OF GROUT _____
D. DEPTH TO TOP OF BENTONITE _____
E. DEPTH TO TOP OF SAND _____
F. DEPTH TO TOP OF SCREEN _____
G. TOTAL WELL DEPTH _____
H. WATER FIRST NOTICED _____
I. DEPTH TO WATER AT COMPLETION _____

CLIENT _____
DRILLING CONTRACTOR _____
DRILL RIG _____
DRILLERS _____
INSTALLATION DATE _____
LOGGED BY _____

NOT TO SCALE



Details of Monitoring Well _____
Project _____
Project Location _____

Field Equipment Decontamination at Nonradioactive Sites	SOP 4-5 Revision: 10 Date: February 2015

Approved: *Signature* **Technical Review:** Robert R. Alexander

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to describe the general procedures required for decontamination of field equipment at nonradioactive sites. This SOP serves as a general guide and is applicable at most sites; however, it shall be noted that site-specific conditions (i.e., type of contamination, type of media sampled), the governing agency (e.g., EPA, DOE, USACE), and site-specific work plans, sampling and analysis plans and/or quality assurance (QA) project plans may require modifications to the decontamination procedures provided in this SOP. Decontamination of field equipment is necessary to ensure acceptable quality of samples by preventing cross contamination. Further, decontamination reduces health hazards and prevents the spread of contaminants offsite.

2.0 Background

2.1 Definitions

Acid Rinse - A solution of 10 percent nitric or hydrochloric acid made from reagent grade acid and analyte-free water.

Analyte-Free Water - Tap water that has been treated so that the water contains no detectable heavy metals or other inorganic compounds. Analyte-free water shall be stored only in clean glass, stainless steel, or plastic containers that can be closed when not in use.

Clean - Free of contamination and when decontamination has been completed in accordance with this SOP.

Cross Contamination - The transfer of contaminants through equipment or personnel from the contamination source to less contaminated or noncontaminated samples or areas.

Decontamination - The process of rinsing or otherwise cleaning the surfaces of sampling or other equipment to rid them of contaminants and to minimize the potential for cross contamination of samples or exposure to personnel.

Safety Data Sheets (SDS) - These documents discuss the proper storage and physical and toxicological characteristics of a particular substance used during decontamination. These documents, generally included in site health and safety plans, shall be kept on site at all times during field operations.

Organic-Free/Analyte-Free Water - Tap water that has been treated so that the water meets the analyte-free water criteria and contains no detectable organic compounds. Organic-free/analyte-free water shall be stored only in clean glass, Teflon™, or stainless steel containers that can be closed when not in use.

Potable Water - Tap water may be obtained from any municipal system. Chemical analysis of the water source may be required before it is used for decontamination purposes.

Sampling Equipment - Equipment that comes into direct contact with the sample media. Such equipment includes split spoon samplers, well casing and screens, and spatulas or bowls used to homogenize samples.

Soap - Low-sudsing, nonphosphate detergent such as Liquinox™.

Solvent Rinse - Pesticide grade, or better, isopropanol, acetone, or methanol.

2.2 Associated Procedures

- SOP 1-1 – *Surface Water Sampling*
- SOP 1-3 – *Surface Soil Sampling*
- SOP 1-4 – *Subsurface Soil Sampling*
- SOP 1-5 – *Groundwater Sampling Using Bailers*
- SOP 1-7 – *Wipe Sampling*
- SOP 1-9 – *Tap Water Sampling*
- SOP 1-11 – *Sediment/Sludge Sampling*
- SOP 1-12 – *Low Flow (Low-Stress) Groundwater Sampling*
- SOP 1-13 – *Drum Sampling*
- SOP 1-14 – *Lagoon Sampling*
- SOP 1-15 – Procedures for Determination of Screening-Level Elemental Concentrations in Soil and Sediment using Field Portable X-Ray Fluorescence Spectrometry
- SOP 2-2 – *Guide to Handling Investigation-Derived Waste*
- SOP 3-1 – *Geoprobe® Sampling*

3.0 Responsibilities

The project manager or designee, generally the field team leader (FTL), ensures that field personnel are trained in the performance of this procedure and that decontamination is conducted in accordance with this SOP and site-specific work plans. The FTL may also be required to collect and document rinsate samples (also known as equipment blanks) to provide quantitative verification that these procedures have been correctly implemented.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

4.0 Required Equipment

- Stiff-bristle scrub brushes
- Plastic buckets and troughs
- Soap
- Nalgene or Teflon sprayers or wash bottles or 2- to 5-gallon, manual-pump sprayer (pump sprayer material must be compatible with the solution used)
- Plastic sheeting, plastic bags, and/or aluminum foil to keep decontaminated equipment clean between uses
- Disposable wipes, rags, or paper towels
- Potable water*
- Analyte-free water
- Organic-free/analyte-free water
- Gloves, safety glasses, and other protective clothing as specified in the site-specific health and safety plan
- High-pressure pump with soap dispenser or steam-spray unit (for large equipment only)
- Appropriate decontamination solutions pesticide grade or better and traceable to a source (e.g., 10 percent and/or 1 percent nitric acid [HNO_3], acetone, methanol, isopropanol, hexane)
- Tools for equipment assembly and disassembly (as required)
- 55-gallon drums or tanks for temporary storage of decontamination water (as required)
- Pallets for drums or tanks holding decontamination water (as required)

* Potable water may be required to be tested for contaminants before use. Check field plan for requirements.

5.0 Procedures

All reusable equipment (nondedicated) used to collect, handle, or measure samples shall be decontaminated before coming into contact with any sampled media or personnel using the equipment. Decontamination of equipment shall occur either at a central decontamination station or at portable decontamination stations set up at the sampling location, drill site, or monitoring well location. The centrally located decontamination station shall include an appropriately sized bermed and lined area on which equipment decontamination shall occur and shall be equipped with a collection system and storage vessels. In certain circumstances, berming is not required when small quantities of water are being generated and for some short duration field activities (i.e., pre-remedial sampling). Equipment shall be transported to and from the decontamination station in a manner to prevent cross contamination of equipment and/or area. For example, precautions taken may include enclosing augers in plastic wrap while being transported on a flatbed truck.

The decontamination area shall be constructed so that contaminated water is either collected directly into appropriate containers (5-gallon buckets or steel wash tubs) or within the berms of the decontamination area that then drains into a collection system. Water from the collection system shall be transferred into 55-gallon drums or portable tanks for temporary storage. Typically, decontamination water shall be staged until sampling results or waste characterization results are obtained and evaluated and the proper disposition of the waste is determined (SOP 2-2, *Guide to Handling Investigation-Derived Waste*). The exact procedure for decontamination waste disposal shall be discussed in the work plan. Also, solvent and acid rinse fluids may need to be segregated from other investigation-derived wastes.

All items that shall come into contact with potentially contaminated media shall be decontaminated before use and between sampling and/or drilling locations. If decontaminated items are not immediately used, they shall be covered either with clean plastic or aluminum foil depending on the size of the item. All decontamination procedures for the equipment being used are as follows:

General Guidelines

- Potable, analyte-free, and organic-free/analyte-free water shall be free of all contaminants of concern. Following the field QA sampling procedure described in the sampling plan, analytical data from the water source may be required.
- Sampling equipment that has come into contact with oil and grease shall be cleaned with methanol or other approved alternative to remove the oily material. This may be followed by a hexane rinse and then another methanol rinse. Regulatory or client requirements regarding solvent use shall be stated in the sampling plan.
- All solvents and acids shall be pesticide grade or better and traceable to a source. If provided, certificates of analyses should be placed in the project files. The corresponding lot numbers shall be recorded in the appropriate logbook.

Note: Solvents and acids are potentially hazardous materials and must be handled, stored, and transported accordingly. Solvents shall never be used in a closed building. See the site-specific health and safety plan and/or the chemical's MSDS for specific information regarding the safe use of the chemical.

- Decontaminated equipment shall be allowed to air dry before being used.
- Documentation of all equipment type, date, time, and method of decontamination along with associated field QA sampling shall be recorded in the appropriate logbook.
- Gloves, boots, safety glasses, and any other personnel protective clothing and equipment shall be used as specified in the site-specific health and safety plan.

5.1 Heavy Equipment Decontamination

Heavy equipment includes drilling rigs, well development rigs, and backhoes. Follow these steps when decontaminating this equipment:

1. Establish a bermed decontamination area that is large enough to fully contain the equipment to be cleaned. If available, an existing wash pad or appropriate paved and bermed area may be used; otherwise, use one or more layers of heavy plastic sheeting to cover the ground surface and berms. All decontamination pads shall be upwind of the area under investigation.

2. With the rig in place, spray areas (rear of rig or backhoe) exposed to contaminated media using a hot water high-pressure sprayer. Be sure to spray down all surfaces, including the undercarriage.

3. Use brushes, soap, and potable water to remove dirt whenever necessary.

4. Remove equipment from the decontamination pad and allow it to air dry before returning it to the work site.

5. After decontamination activities are completed, collect all contaminated wastewater, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal as detailed in the field plan. Liquids and solids must be drummed separately.

5.2 Downhole Equipment Decontamination

Downhole equipment includes hollow-stem augers, drill pipes, rods, stems, etc. Follow these steps when decontaminating this equipment:

1. Set up a centralized decontamination area, if possible. This area shall be set up to collect contaminated rinse waters and to minimize the spread of airborne spray.

2. Set up a “clean” area upwind of the decontamination area to receive cleaned equipment for air-drying. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or other surfaces on which decontaminated equipment is to be placed. All decontamination pads shall be upwind of any areas under investigation.

3. Place the object to be cleaned on aluminum foil or plastic-covered wooden sawhorses or other supports. The objects to be cleaned shall be at least 2 feet above the ground to avoid splashback when decontaminating.

4. Using soap and potable water in the hot water high-pressure sprayer (or steam unit), spray the contaminated equipment. Aim downward to avoid spraying outside the decontamination area. Be sure to spray inside corners and gaps especially well. Use a brush, if necessary, to dislodge dirt.

5. If using soapy water, rinse the equipment using clean, potable water. If using hot water, the rinse step is not necessary if the hot water does not contain a detergent. If the hot water contains a detergent, this final clean water rinse is required.

6. Using a suitable sprayer, rinse the equipment thoroughly with analyte-free water.

7. Remove the equipment from the decontamination area and place in a clean area upwind to air dry.

8. After decontamination activities are completed, collect all contaminated wastewaters, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal. Liquids and solids must be drummed separately.

5.3 Sampling Equipment Decontamination

Follow these steps when decontaminating sampling equipment:

1. Set up a decontamination line on plastic sheeting. The decontamination line shall progress from "dirty" to "clean." A clean area shall be established upwind of the decontamination wash/rinse activities to dry the equipment. At a minimum, clean plastic sheeting must be used to cover the ground, table, or other surfaces that the decontaminated equipment is placed for drying.

2. Disassemble any items that may trap contaminants internally. Do not reassemble the items until decontamination and air drying are complete.

3. Wash the items with potable water and soap using a stiff brush as necessary to remove particulate matter and surface films. The items may be steam cleaned using soap and hot water as an alternative to brushing. Note: Polyvinyl chloride or plastic items shall not be steam cleaned. Items that have come into contact with concentrated and/or oily contaminants may need to be rinsed with a solvent such as hexane and allowed to air dry prior to this washing step.

4. Thoroughly rinse the items with potable water.

5. The specific chemicals and/or fluids to be used in the decontamination process will be defined in the sampling plan. If sampling for metals, typically the potable water and soap wash is followed by a potable water rinse then an analyte-free water rinse; alternatively, an acid solution (e.g., 10 percent nitric acid) rinse followed by a rinse using analyte-free water may be specified in some instances. If sampling for organic compounds, thoroughly rinse the items with solvent (e.g., isopropanol) followed by a rinse using organic-free/analyte-free water. Again, the specific chemicals used in any acid rinse or solvent rinse phases shall be specified in the sampling plan. Any acid rinsate and solvent rinsate must each be containerized separately. Acids and solvents are potentially hazardous materials and care must be exercised when using these chemicals to prevent adverse health affects (e.g., skin burns, irritation to the eyes and respiratory system). Appropriate personal protective equipment must be worn when using these chemicals. The use of acids and solvents for decontamination should be carefully considered. These chemicals (including spent rinsate) must be managed and stored appropriately. Special measures such as proper labels, paperwork, notification, etc. may be required when transporting or shipping these chemicals.

6. Allow the items to air dry completely.

7. After drying, reassemble the parts as necessary and wrap the items in clean plastic wrap or in aluminum foil.

8. After decontamination activities are completed, collect all contaminated waters, used solvents and acids, plastic sheeting, and disposable personal protective equipment. Place the contaminated items in properly labeled drums for disposal. Liquids and solids must be drummed separately. Refer to site-specific plans for labeling and waste management requirements.

5.4 Pump Decontamination

Follow the manufacturer's recommendation for specified pump decontamination procedures. At a minimum, follow these steps when decontaminating pumps:

1. Set up the decontamination area and separate "clean" storage area using plastic sheeting to cover the ground, tables, and other surfaces. Set up four containers: the first container shall contain dilute (nonfoaming) soapy water, the second container shall contain potable water, the third container shall be empty to receive wastewater, and the fourth container shall contain analyte-free water.

2. The pump shall be set up in the same configuration as for sampling. Submerge the pump intake (or the pump, if submersible) and all downhole-wetted parts (tubing, piping, foot valve) in the soapy water of the first container. Place the discharge outlet in the wastewater container above the level of the wastewater. Pump soapy water through the pump assembly until it discharges to the waste container. Scrub the outside of the pump and other wetted parts with a metal brush.

3. Move the pump assembly to the potable water container while leaving discharge outlet in the waste container. All downhole-wetted parts must be immersed in the potable water rinse. Pump potable water through the pump assembly until it runs clear.

4. Move the pump intake to the analyte-free water container. Pump the water through the pump assembly. Pump the volume of water through the pump specified in the field plan. Usually, three pump-and-line-assembly volumes shall be required.

5. Decontaminate the discharge outlet by hand, following the steps outlined in Section 5.3.

6. Remove the decontaminated pump assembly to the clean area and allow it to air dry upwind of the decontamination area. Intake and outlet orifices shall be covered with aluminum foil to prevent the entry of airborne contaminants and particles.

5.5 Low Stress (Low Flow) Sampling Pump Decontamination

Sampling equipment used for Low Stress (Low Flow) Groundwater Sampling (SOP 1-12) must be decontaminated thoroughly each day before use (daily decontamination) and after each well is sampled (between-well decontamination). All non-disposable equipment, including the pump (support cable and electrical wires which are in contact with the sample) will be decontaminated as described below. Dedicated, in-place pumps and tubing must be thoroughly decontaminated using "daily decontamination" procedures prior to their initial use or installation.

5.5.1 Prior to Sampling Event Decontamination

Please Note: Steps 5 through 13 should only be performed once (for each pump that is to be used) before the commencement of a particular sampling event by a person qualified to disassemble pumps.

1. Pre-rinse: Operate pump in a deep basin containing 8 to 10 gallons of potable water for 5 minutes and thoroughly flush other equipment with potable water.

2. Wash: Operate pump in a deep basin containing 8 to 10 gallons of a non-phosphate detergent solution, such as Liquinox™, for 5 minutes and thoroughly flush other equipment with fresh detergent solution. Use the detergent sparingly.

3. Rinse: Operate pump in a deep basin of potable water for 5 minutes and thoroughly flush other equipment with potable water for 5 minutes.

4. Analyte-Free Rinse: Operate pump in a deep basin of analyte-free water to pump out 1 to 2 gallons of this final rinse water.

5. Disassemble pump.

6. Wash pump parts (inlet screen, shaft suction interconnector, motor lead assembly, stator house): Place the disassembled parts of the pump into a deep basin containing 8 to 10 gallons of non-phosphate detergent solution. Scrub all pump parts with a test tube brush.

7. Rinse pump parts with potable water for five minutes.

8. Rinse the pump parts with demonstrated analyte-free water.

9. If sampling for metals, an acid rinse may be specified in the sampling plan; if so, place impeller assembly in a large glass beaker and rinse with 1% nitric acid (HNO3).

10. Rinse impeller assembly with potable water for five minutes.

11. If sampling for organics, a solvent rinse may be specified; if so, place impeller assembly in a large glass bleaker and rinse with isopropanol or appropriate organic solvent specified in the site-specific plan.

12. Thoroughly rinse impeller assembly with demonstrated analyte-free water.

13. Reassemble pump.

5.5.2 Daily and Between-Well Decon

1. Pre-rinse: Operate pump in a deep basin containing 8 to 10 gallons of potable water for 5 minutes and thoroughly flush other equipment with potable water for five minutes.

2. Wash: Operate pump in a deep basin containing 8 to 10 gallons of a non-phosphate detergent solution, such as Liquinox™, for 5 minutes and thoroughly flush other equipment with fresh detergent solution. Use the detergent sparingly.
3. Rinse: Operate pump in a deep basin of potable water for 5 minutes and thoroughly flush other equipment with potable water for five minutes.
4. Final Rinse: Operate pump in a deep basin of analyte-free water to pump out 1 to 2 gallons of this final rinse water.

5.6 Instrument Probe Decontamination

Instrument probes used for field measurements such as pH meters, conductivity meters, etc. shall be decontaminated between samples and after use with analyte-free, or better, water.

5.7 Waste Disposal

Refer to site-specific plans and SOP 2-2 for waste disposal requirements. The following are guidelines for disposing of wastes:

- All wash water, rinse water, and decontamination solutions that have come in contact with contaminated equipment are to be handled, packaged, labeled, marked, stored, and disposed of as investigation-derived waste.
- Small quantities of decontamination solutions may be allowed to evaporate to dryness.
- If large quantities of used decontamination solutions shall be generated, each type of waste shall be contained in separate containers.
- Unless otherwise required, plastic sheeting and disposable protective clothing may be treated as solid, nonhazardous waste.
- Waste liquids shall be sampled, analyzed for contaminants of concern in accordance with disposal regulations, and disposed of accordingly.

6.0 Restrictions/Limitations

Nitric acid and polar solvent rinses are necessary only when sampling for metals or organics, respectively. These steps shall not be used, unless required, because of the potential for acid burns and ignitability hazards.

If the field equipment is not thoroughly rinsed and allowed to completely air dry before use, volatile organic residue, which interferes with the analysis, may be detected in the samples. The occurrence of residual organic solvents is often dependent on the time of year sampling is conducted. In the summer, volatilization is rapid, and in the winter, volatilization is slow. Check with your EPA region, state, and client for approved decontamination solvents.

7.0 References

American Society for Testing and Materials. 2015. *Standard Practice for Decontamination of Field Equipment at Waste Sites*, ASTM D5088-15. January 15.

Puls, R.W. and M.J. Barcelona. 1996. Low-Flow (Minimal Drawdown) Ground-water Sampling Procedures, EPA/540/S-95/504.

U. S. Environmental Protection Agency. 1987. *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001.1.

_____. 1998. EPA Region 2, Ground Water Sampling Procedure Low Stress (Low Flow) Purging and Sampling, March 16.

_____. 1992. *Standard Operating Safety Guidelines*; Publication 9285.1-03. June.

_____. 2011. Region 4. The Field Branches Quality System and Technical Procedures, Soil Sampling. SESDPROC-205-R2. December. http://www.epa.gov/region4/sesd/fbqstp/

Hydraulic Conductivity Testing	SOP 4-6 Revision: 5 Date: February 2015
Approved: Signature	**Technical Review:** John Dougherty

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to define requirements for conducting and analyzing in situ hydraulic conductivity (slug) tests in small, developed wells.

2.0 Background

2.1 Definitions

Slug Testing - A rapid and easy means of estimating the hydraulic conductivity of an aquifer. If the thickness of the aquifer is known, then the transmissivity can also be determined. Slug testing is accomplished by adding (or removing/ displacing) a known volume to (or from) the monitoring well to create a rapid rise (or fall) in water level. Water levels are then measured as the water level in the well returns to static (pre-test) conditions. American Society for Testing and Materials method D4044 provides an overview of slug testing (ASTM 1996). Butler (1998) is a good reference for the design and analysis of slug tests over a full range of aquifer conditions.

Slug Bar - A weighted cylinder that is used in displacing water in a well of known volume. A bailer may be used in place of a slug bar under low-recharge aquifer conditions.

Pneumatic System - A system that uses an air pump, compressor, or compressed air cylinder to increase the air pressure in the well, which is sealed with an air-tight cap that has ports through which the compressed air is introduced and a water level indicator or pressure transducer can be inserted. This displacement method is commonly employed in high transmissivity aquifers where aquifer response is rapid and it is difficult to achieve the rapid initial displacement required using a slug bar or bailer. In all cases, the rate of water level recovery is then measured using a pressure transducer and data recorder or a water level meter and stopwatch (the former method is preferable in most environments). Data, as displacement-time pairs, are then graphed and used in equations to determine hydraulic conductivity.

2.2 Associated Procedures

- SOP 1-5, *Groundwater Sampling with Bailers*
- SOP 1-6, *Water Level Measurement*
- SOP 2-6, *Handling Investigative-Derived Waste*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-3, *Well Development and Purging*
- SOP 4-4, *Design and Installation of Monitoring Wells in Aquifers*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.3 Discussion

Advantages of slug testing over pump testing include the fact that little or no contaminated water is produced requiring containment and disposal as well as that several areas can be tested in a relatively short period of time. A disadvantage of slug testing is that the resulting estimate of hydraulic conductivity is limited to a small volume of the aquifer around the tested well and care must be taken in extrapolating the results from one well to other areas or intervals of the aquifer.

If possible, when designing the field program or considering in which interval to place a well screen, try to screen only one formation type. If a well is screened across more than one formation (such as fine sand and coarse sand or overburden and bedrock) the results must be analyzed and interpreted in the hydrogeologic context.

3.0 Responsibilities

Site Manager - The site manager is responsible for ensuring that field personnel have been trained in conducting slug tests and for ensuring that slug tests are conducted in accordance with this procedure.

Field Team Leader - The field team leader is responsible for performing slug tests in accordance with this procedure and for verifying that the data collected are adequate and of high quality. The project field geologist shall perform a field calculation to check data quality.

***Note*:** Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Required Equipment

The following equipment shall be used when performing a rising or falling-head slug test in a monitoring well. Site-specific conditions may warrant the use of additional equipment.

- Pressure transducer and data recorder, if data are to be automatically recorded (recommended) and manufacturers' instructions
- Laptop or hand-held computer for downloading and viewing data (field printer optional)
- Water level measuring device
- Stopwatch, if measurements collected manually (not recommended)
- Slug device of known volume
- Rope or wire
- Duct tape
- Field logbook
- Decontamination equipment and supplies
- Data on the construction of the well: depth to screen, screen length, well drilled diameter, riser diameter, height of sandpack above screen and length of riser above ground surface

Note that the well construction data shall be used so that the slug test data being collected are appropriate and of acceptable quality. Additional information (e.g., distance from screen to confining layer) may be necessary to analyze the data and determine the hydraulic conductivity. Data analysis is not covered under this procedure.

The slug bar shall be constructed of plastic, such as polyvinyl chloride (PVC), or metal such as aluminum or steel (depending upon the chemical environment in the well) and have no buoyancy. For example, a standard slug is constructed with a PVC pipe filled with sand and capped at both ends. The slug bar shall be of sufficient size to cause a recommended minimum of 1 to 3 feet of displacement in a well. A slightly lesser or greater head change is acceptable so long as a sufficient response curve is recorded that can be applied in a subsequent analysis. For a 2-inch diameter monitoring well, the slug bar shall be no more than 1.5 inches in diameter and a minimum of 5 feet long. For a 4-inch diameter well, the slug bar shall be no more than 3 inches in diameter and a minimum of 5 feet long. The slug bar shall be securely fastened to a nylon rope or braided metal wire.

A standard sampling or well development bailer may be used in place of the slug bar, as long as the volume of water displaced by the bailer is sufficient to change the water level in the well a minimum of 1 to 3 feet. If the bailer is to be used for a falling-head test, it shall be filled with analyte-free water so that the bailer will not have any buoyancy.

5.0 Procedures

5.1 Preparation

The following steps must be followed when preparing for slug testing:

1. Lay plastic sheeting around the wellhead. Arrange needed equipment and decontamination materials on the sheet or on a table.

2. Put on personnel protective clothing, as specified in the site-specific health and safety plan.

3. Open the protective casing locking lid and vented riser caps following the procedures outlined in SOP 1-6. Note the physical condition of the well, including damage, deterioration and signs of tampering. Note any unusual odors, sounds, or difficulties in opening the well. Record organic vapor readings with a suitable organic vapor screening device.

4. Measure and record the static water level, the depth to the bottom of the well and inside diameter of the well casing. Record these data in the appropriate logbook.

5. If using a pressure transducer and data logger (transducers with built-in data loggers are commonly used for slug tests), lower the pressure transducer into the well to a sufficient depth so that the transducer will be below the maximum depth reached by the bottom of the slug bar or other displacement device. If necessary, calibrate the transducer as specified by the manufacturer. Allow the transducer to temperature equilibrate in the well for approximately 15 minutes (or as recommended by the manufacturer) after insertion and before any calibration or test procedure to ensure that it will accurately record water level changes. Make sure that the transducer is not placed below its maximum operating depth, or it will not be able to detect any change in pressure. For example, pressure increases 1 pound per square inch (psi) per 2.3 feet of head; therefore, a 10 psi transducer will function to a depth of 23 feet below the water level in the well.

6. Secure the pressure transducer cable using a Kellems grip or similar device. The transducer cable shall lay flat along side the well riser, so that disturbance by the slug bar will be avoided.

   ***Note*:** Do not kink the transducer cable, otherwise the pressure equalization vent tube in the cable will be damaged and the transducer will not function properly.

7. Allow the water level in the well to recover to static after emplacement of the pressure transducer, before starting the test. Measure and record this water level.

8. Program the data logger to record logarithmically, with a maximum time interval of no more than 1 minute between readings. If the formation is expected to have low hydraulic conductivity, the maximum interval between readings can be set to a longer time interval, such as 10 minutes.

9. Confirm and/or set the transducer and logger parameters as recommended by the manufacturer. This task may also be performed before placing the instrument in the well.

10. Determine the distance from the top of the well riser to the water surface in the well and add 1 foot to this length. The resulting length is the amount of wire or rope needed so that the slug bar or bailer will be submerged a minimum of 1 foot when it is placed in the well. A loop shall be placed in the rope or wire at this length and a strong metal rod or wooden stick placed and secured through the loop. When inserted into the well, the slug bar shall be a distance (more than 1 foot) above the transducer to avoid disturbing the measuring device.

11. If depth readings are to be recorded manually (this procedure is not recommended but may be used in formations suspected of having low hydraulic conductivity, less than 1 foot per day), readings shall be taken every 10 seconds for the first minute of the test, every 30 seconds for the next 4 minutes and every minute until 10 minutes. Thereafter, readings shall be taken every 5 minutes for the duration of the test. If the well has not recovered within 1 hour, readings shall be taken every 0.5 hours until 6 hours and 1 hour every hour thereafter. This process will require two personnel during the first 10 minutes of the test: one to act as time keeper/data recorder and one to measure depth to water in the well.

5.2 Standard Displacement Slug Tests

5.2.1 Falling-Head Slug Test Procedure

This test can only be conducted in wells whose screens are fully submerged, otherwise, displaced water will be introduced into the unsaturated zone and recovery rates will be due to flow in both the unsaturated and saturated zones. All slug test analytical procedures assume flow in the saturated zone only. The following steps must be followed when performing falling-head slug tests:

1. Place the slug or bailer in the well until the bottom of the displacement device is no more than 6 inches to 1 foot above the water level in the well. The person holding the device shall be holding the rope or wire by the rod or stick described in Section 5.1, ninth bullet.
2. Switch on the data recorder, or set the water level meter probe near the level at which water is expected to rise.
3. To start the test, the person holding the slug bar will signal the person operating the data logger or water level indicator, then rapidly lower the displacement device into the well until the stick or rod is resting horizontally on top of the well riser. The slug bar shall not be dropped, to minimize sloshing in the well. The data logger is turned on immediately prior to the slug bottom entering the water.
4. Continue recording depth-time data until the well has recovered to at least 90 percent of the static water level. When using data recorders, it is advisable to check and record the reading every few minutes to ensure that data are being properly recorded. If 90 percent recovery has not occurred within 12 hours, the test may be stopped. Field conditions and time constraints may warrant stopping the test in less than 12 hours. The final decisions under these circumstances will be the responsibility of the field team leader.
5. Record the time of test completion and file name in the logbook.
6. Review the response curve. If a sufficient response curve was not recorded (e.g., logging was not started soon enough to identify maximum water level displacement), then the test shall be repeated. If an acceptable response curve is not being recorded due to field conditions (e.g., no water level response due to high hydraulic conductivity) the project manager shall be notified and a determination on the well test shall be made.
7. Decontaminate all equipment according to SOP 4-5. Clean up the site, and close and lock the well before leaving. Contaminated plastic sheeting and disposable protective clothing shall be taken to designated disposal containers.
8. Download the data logger to a computer or to hardcopy to ensure that the data is not inadvertently lost. If the data were recorded manually, calculate the relative change in head by subtracting the recorded depths to water during recovery from the initial static depth to water reading and record the absolute value of that change, for each depth-time data pair.

Note: Both rising- and falling-head slug tests may be carried out in the same operation by first measuring the rate of water level fall immediately after slug insertion, then measuring the rate of water level rise after slug withdrawal. Be sure that the well has recovered to the static water level before conducting the rising-head test. If using a data logger, the recovery tests needs to be set up and run as a separate test.

5.2.2 Rising-Head Slug Test Procedure

The steps for a rising-head test are essentially the same as those for a falling-head test. In a well screened across the water table, a rising-head test is the only test that is valid. The following steps must be followed when performing rising- head slug tests:

1. Lower the slug bar or bailer of known volume into the well until it is fully submerged. Allow the well to re-equilibrate to static water level. In formations of suspected low hydraulic conductivity, re-equilibration may take several hours or overnight. In such cases, it is suggested that the displacement device be placed in the well at the end of a field day and the test conducted the following day.
2. Turn on the data recorder, if used, or verify that static water level has been re-established with a water level meter.

3. To start the test, the person holding the slug bar will signal the person operating the data logger or water level indicator, then rapidly and smoothly raise the displacement device from the well until the bottom of the slug bar is above the water level in the well. The data logger is turned on or manual measurements commence at the moment the slug bar is raised and before it (or any portion of it) is removed from the water. If a data logger is being used, the slug bar wire or rope shall be secured to the well casing or riser for the duration of the test and only removed from the well after the test has been completed, to avoid disturbing or dislocating the pressure transducer.

4. Continue recording depth-time data until the well has recovered to at least 90 percent of the static water level. When using data recorders, it is advisable to check and record the reading every few minutes to ensure that data are being properly recorded. If 90 percent recovery has not occurred within 12 hours, the test may be stopped. Field conditions

   and time constraints may warrant stopping the test in less than 12 hours. The final decisions under these circumstances will be the responsibility of the field team leader.

5. Record the time of test completion and file name in the logbook.

6. Review the response curve. If a sufficient response curve was not recorded (e.g., logging was not started soon enough to identify maximum water level displacement), then the test shall be repeated. If an acceptable response curve is not being recorded due to field conditions (e.g., no water level response due to high hydraulic conductivity), the project manager shall be notified and a determination on the well test shall be made.

7. Decontaminate all equipment according to SOP 4-5. Clean up the site, and close and lock the well before leaving. Contaminated plastic sheeting and disposable protective clothing shall be taken to designated disposal containers.

8. Download the data logger to a computer or to hardcopy to ensure that the data is not inadvertently lost. If the data were recorded manually, calculate the relative change in head by subtracting the recorded depths to water during recovery from the initial static depth to water reading and record the absolute value of that change, for each depth-time data pair.

5.3 Pneumatic Rising-Head Tests

This test can be performed in aquifers of high hydraulic conductivity that are expected to respond very rapidly to slug displacement. It can only be performed in wells where the screen is substantially below the water table, otherwise, increased air pressure in the well casing will be able to bleed off to the unsaturated zone through the well screen and the test will not be successful.

5.3.1 Required Equipment

In addition to the required equipment outlined in Section 4.0, the following equipment shall be used when conducting a pneumatic rising-head slug test.

- Minimum 30-psi rated transducer and data logger
- Electric water level indicator with on/off switch
- Pressure-tight "tree" assembly, as described below
- Short length (6 inches) of flexible rubber hose whose inside diameter is the same as the outside diameter of the well riser
- Two 2- or 4-inch diameter hose clamps
- Compressor, air pump, or compressed air tank with hose and appropriate adapters

The pressure-tight tree assembly is a device placed on the top of the well that will accomplish the following:

- Form a pressure-tight seal between the well and the atmosphere
- Allow the injection of compressed air into the well via an air hose connected to the pump, compressor, or air supply
- Provide a pressure-tight passage for a pressure transducer cable and a water level meter
- Allow for rapid well depressurization

The tree is illustrated in Figure 1. If the top of the riser is threaded, the device may be screwed onto the riser if the threads are compatible (Teflon™ tape shall be used to ensure a good seal). If the threaded end of the riser has been cut off, a slip coupling will need to be placed over the base of the tree and the top of the riser. A small length of flexible rubber hose the same inside diameter as the outside diameter of the coupling will need to be slipped over the coupling and secured in place with tightly closed hose clamps to form a pressure-tight seal between the riser and the well.

The simplest method for providing access through the tree for the pressure transducer cable indicator is to use a modified standard large diameter black rubber cork. A hole that is the same diameter as the cable shall be drilled through the cork's axis and a vertical slit shall be cut radially from the hole to an edge of the cork. The pressure transducer cable shall be threaded through the hole and the water level indicator tape shall be placed flat in the slit. The cork shall be firmly placed in the top of the tree to form a pressure-tight seal. To ensure that the cork does not pop out while the well is under pressure, it can be secured in place with duct tape or a friction fit plastic cap placed over the cork and onto the tree.

The tree will have a standard ball valve with an inside valve orifice diameter no less than the diameter of the well riser as shown in Figure 1. In addition, a pressure-tight coupling (swage-loc, quick-connect, or Schrader valve) will be attached to the side of the tree to act as a compressed air inlet.

5.3.2 Preparation

Preparation procedures for the pneumatic test are similar to those for the standard slug bar displacement test, with the exception that an electronic data logger is a necessity for this procedure.

5.3.3 Pneumatic Slug Test Procedure

1. Install the test tree to the top of the well, using a method appropriate to the type of riser present (threaded or unthreaded). Make sure that the seal to the riser top is pressure-tight.

2. Lower the pressure transducer into the well through the top of the tree to a minimum of 10 feet below the water table. The pressure transducer shall be rated no less than 30 psi. Allow the transducer to equilibrate at least 15 minutes before initiating any calibration or test procedure.

3. Turn on and insert a water level indicator into the well to approximately 5 feet depth below the water table. Turn off the indicator.

4. Secure the water level indicator and pressure transducer to the test tree using the rubber cork described in Section 5.3.1. Insert the transducer cable into the hole in the rubber cork via the slit and place the water level indicator tape flat in the slit. Place the cork firmly in the top of the tree so that no gaps are left in the cork. Place small strips of duct tape over the assembly to ensure that the seal is airtight and that the cork cannot loosen when the well is pressurized.

   ***Note*:** During this procedure, do not kink the transducer cable or the pressure equalization vent tube in the cable will be damaged and the transducer will not function.

5. Connect the pressure transducer to the data logger and calibrate the system according to manufacturer's instructions. Set the data logger to record logarithmically with a maximum recording interval of no more than 1 minute. Set the logger to record relative change in head only.

6. Connect the air hose to the compressed air supply, pump, or compressor and to the tree. Make sure the ball valve is securely closed.

7. Turn on the water level indicator and start feeding compressed air to the well. When the water level in the well has been depressed sufficiently, the water level indicator submergence tone will stop sounding. The pressure required shall be no more than 2 or 3 pounds over atmospheric pressure.

8. Simultaneously open the ball valve and activate the data logger. Open the ball valve quickly so that the pressure is released at once.

9. In highly permeable aquifers, the water level shall recover to pre-test water levels within a few seconds. Full recovery shall be accomplished in no more than 1 minute. In any event, do not stop the test until a minimum of 90 percent recovery can be confirmed with the data logger.

10. Review the response curve. If a sufficient response curve was not recorded (e.g., logging was not started soon enough to identify maximum water level displacement), then the test shall be repeated. If an acceptable response curve is not being recorded due to field conditions (e.g., no water level response due to high hydraulic conductivity) the project manager shall be notified and a determination on the well test shall be made.

11. Record the time of test completion and file name in the logbook.

12. Decontaminate all equipment according to SOP 4-5. Clean up the site, and close and lock the well before leaving. Contaminated plastic sheeting and disposable protective clothing shall be taken to designated disposal containers.
13. Download the data logger to a computer or to hardcopy to ensure that the data is not inadvertently lost.

6.0 Data Reduction and Analysis Procedures

6.1 General

The following slug test data reduction procedure and report is recommended.

- All raw data shall be printed out and listed as an appendix to the analysis report.

- All data shall be plotted using the graphing method of the accepted analytical solution. These plots shall be included as an appendix to the analysis report.

- All well geometry data shall be tabulated and included in the analysis report. Most of these data must be known before the start of testing, except for items related to the water level in the well at the time of testing. The purpose of this tabulation is to ensure consistent calculation of all variables required in the data analysis, make input into a data analysis computer program an easier task, and to make technical review of the analyses and input values easier. This table shall include the following items for each tested well or piezometer (the list of items may vary depending on the analytical method employed):

  - Well ground surface elevation
  - Well reference elevation (i.e., top of riser)
  - Depth to static water level at start of test
  - Elevation of static water level at start of test
  - Depth to top of screen or open interval from ground surface or top of casing
  - Depth to bottom of screen or open interval from ground surface or top of casing
  - Elevation of top of screen or open interval
  - Elevation of bottom of screen or open interval
  - Depth to base of aquifer (if available)
  - Elevation of base of aquifer (if available)
  - Aquifer saturated thickness
  - Depth to top of screen or open interval relative to the top of the aquifer
  - Depth to bottom of screen or open interval relative to the top of the aquifer
  - Length of saturated well screen
  - Length of saturated riser
  - Diameter of well riser and screen (or open interval)
  - Diameter of borehole
  - Grain-size of filter pack

- The report shall include a detailed description of the data collection procedures and test methods.

- The report shall include a detailed listing of all analysis results.

- When reviewing the data for analysis, note that if the water level recovered to the static level (or close to it) before the test was stopped, only the data before 100 percent recovery shall be included in the data plot. Plotting 100 minutes of data when the recovery occurred rapidly (e.g., 30 seconds or 2 minutes) will make analysis of the actual response very difficult and often lead to a substantial underestimate of the formation hydraulic conductivity. Raw data plots shall also be examined for evidence of sloshing of the water level in the well caused by insertion or removal of the slug bar. In most cases, these early data points can also be removed from the data set and time values reset to the new starting point represented by the remaining data. This evaluation is shown on Figure 2. The data may also be removed using common software packages developed for analyzing slug tests.

6.2 Review and Analysis of Data

Slug test response generally falls into three categories illustrated on Figure 3. Overdamped or normal response occurs where the well recovers to static level without exceeding that level. Critically damped response occurs where the well recovers to static level and the water level flows above (rising-head test) or below (falling-head test) then recovers to static in a sinusoidal manner within one cycle, as shown in Figure 3. The third category is underdamped harmonic oscillatory response, where the water level in the well oscillates around the static water level as a sine wave of decreasing amplitude.

Slug test data are recommended to be analyzed with computer software; however, data may also be analyzed manually. The groundwater modeling tool kit contains Aquifer^{WIN32} (ESI International), which is a program that may be used for analyzing slug test data. Other programs are also available. Software packages are useful since they can be used to manage a significant amount of data in short time periods and contain many different confined and unconfined slug test solutions. The trained user can use these benefits to generate detailed response curve graphs, precise hydraulic conductivity values, and insights into the hydrogeologic framework near the well. Regardless of the analytical method employed or whether the data is analyzed manually or by computer, the analyst shall review the original technical paper or textbook summary of the method to understand the mechanics and assumptions underlying the method before attempting any analysis.

Slug test data analyses and hydraulic conductivity calculations shall be performed by an experienced professional. Data analysis and parameter calculations are beyond the scope of this SOP and, therefore, are not discussed here.

7.0 Restrictions and Limitations

In wells in which the static water level and water levels induced during testing are above the top of the screened or open hole interval, both rising-head and falling-head tests shall be conducted to provide a redundancy check of results. However, in most cases, rising-head tests provide more consistent data, less subject to sloshing of the water level due to displacement by the slug bar than is often observed in falling-head tests. Falling-head slug tests are invalid in wells where the static water level is at or below the top of the screened or open-hole interval.

Regardless of which testing method is used, it is recommended that the hydraulic conductivity testing be performed three times in each well, if time constraints such as recovery time or the project schedule will allow multiple tests. The purpose of multiple testing is to demonstrate the precision of the test results. Ideally, the test results will be similar, which results in an increased level of confidence in the data. In addition, if one of the data sets is bad, there is additional data available for analysis.

8.0 References

American Society for Testing and Materials. 1996. *Standard Test Method (Field Procedure) for Instantaneous Change in Head (Slug) Tests for Determining Hydraulic Properties of Aquifers.* D4044 – 96 (Reapproved 2008).

Butler, James. 1998. The Design, Performance, and Analysis of Slug Tests. Lewis Publishers. 252 pp.

ESI International, see their website, http://esinternational.com, for current information on Aquifer^{win32}

Figure 1

Pneumatic Slug Test "Tree" Schematic

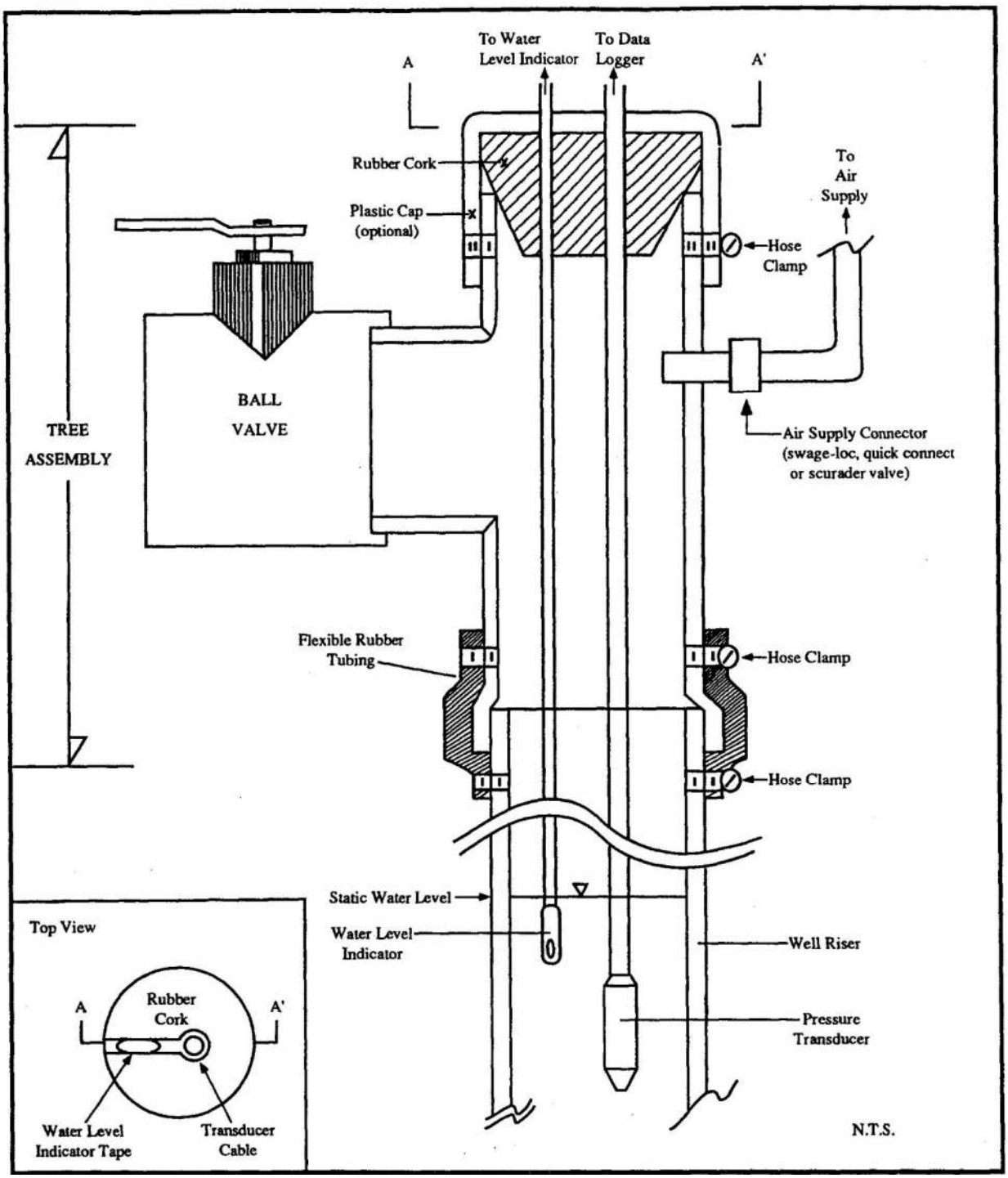
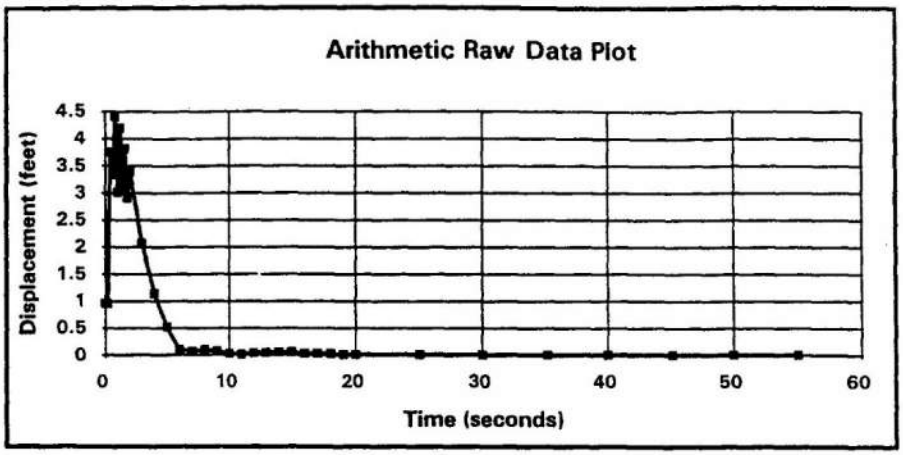
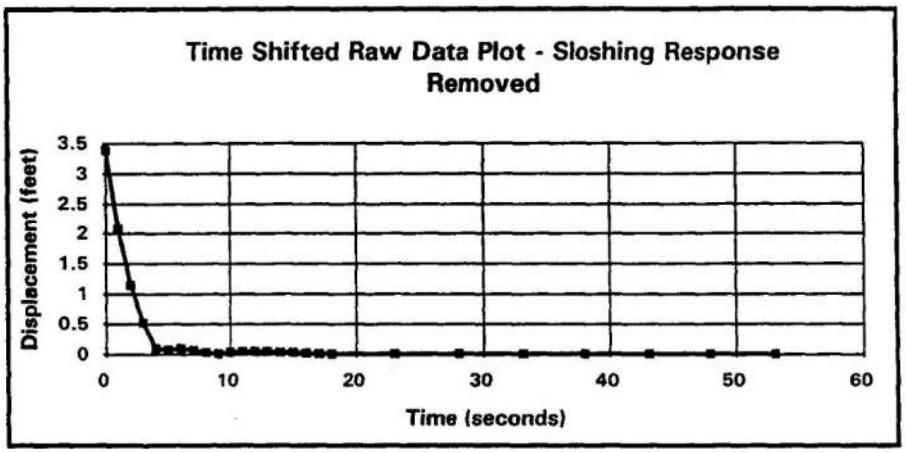


Figure 2

Deletion of Nonessential Data





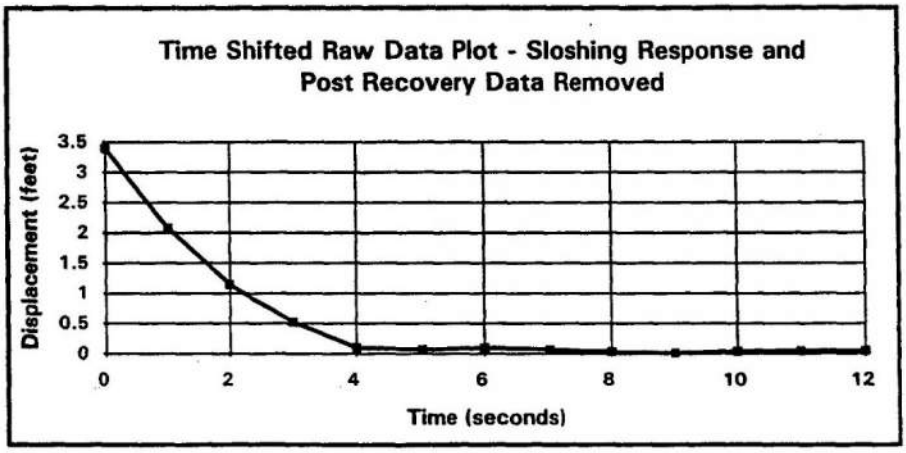
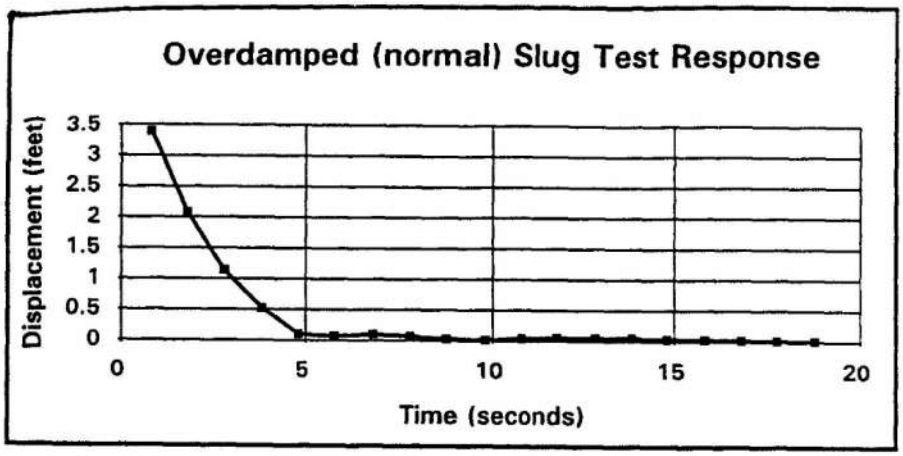
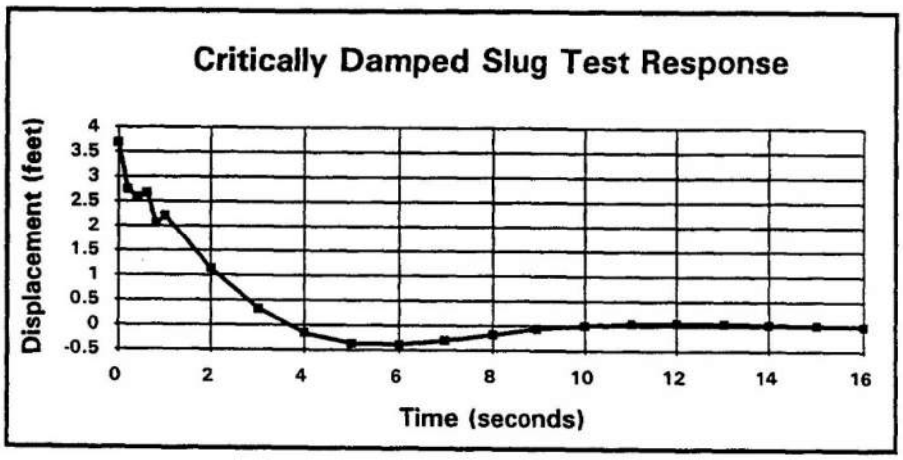
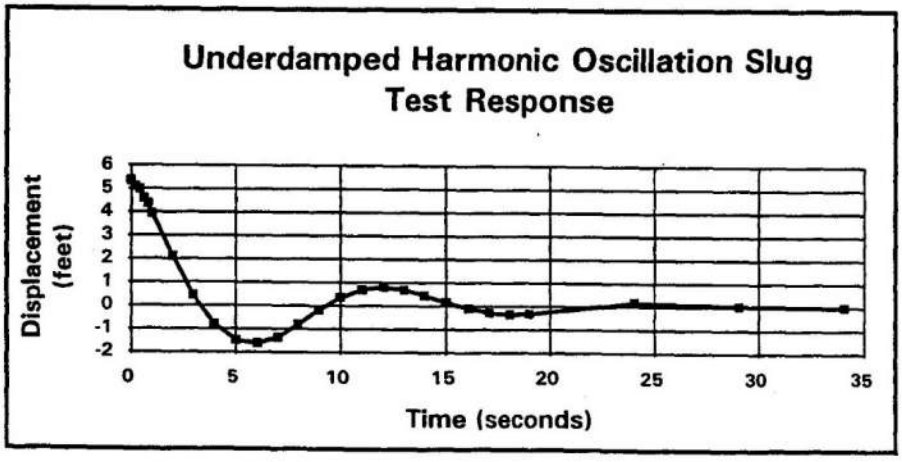


Figure 3

Typical Slug Test Responses







Aquifer Performance Tests	SOP 4-9 Revision: 2 Date: February 2015

Approved: Signature **Technical Review:** John Dougherty

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define requirements for conducting a constant rate aquifer performance test (APT).

2.0 Background

Many different methods and techniques are performed to determine hydraulic properties of an aquifer (American Society for Testing and Materials 2010). The methods and techniques in this procedure are for a standard constant-rate withdrawal test to be conducted at a nonflowing well. APTs are commonly performed in wells that will ultimately be used to withdraw groundwater for an extended period of time. These wells are typically 6 inches or more in diameter and are used for purposes such as drinking water (a supply well), contaminated groundwater removal (a recovery well), and industrial processes (production well). However, tests can be run in other well types and sizes (e.g., a monitoring well that is 4 inches in diameter). The information collected during an APT is used for defining the hydraulic characteristics of the aquifer. Data collected during an APT can also be used to assess pump selection and water delivery piping.

2.1 Definitions

Pumping well - The well from which water is withdrawn during an APT.

Observation well - A well that is used to monitor the groundwater level at some distance from the pumping well during an APT.

Stilling pipe - A small diameter (about 1 inch) pipe that is installed in the pumping well from the top of the pump to the surface; the transducer is placed in the pipe.

2.2 Discussion

In general, APTs consist of withdrawing water from a pumping well for a specified time period and monitoring the water level in the pumping well and observation wells. The recorded time-drawdown data are then reduced and analyzed to:

- Determine the specific capacity and safe yield of the well
- Calculate the properties (transmissivity [T] and storativity [S]) of the aquifer (T may be estimated from pumping well and observation well data; S may be estimated from observation well data)
- Characterize the hydrogeologic framework at and near the investigation area

These three items, or one of the items at a minimum, are typically evaluated with APT data. However, other ancillary but useful information (e.g., water quality changes under stressed conditions) may also be obtained from the APT data. During the planning stages of the APT, the objectives of the test shall be specified so that the necessary data to reach the objectives are collected when the test is performed.

2.3 Associated SOPs

- SOP 1-6, *Water Level Measurement*
- SOP 1-10, *Field Measurement of Organic Vapors*
- SOP 2-2, *Guide to Handling of Investigation-Derived Waste*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-4, *Design and Installation of Monitoring Wells in Aquifers*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

3.0 Roles and Responsibilities

Site Manager - Translates client's requirements into technical direction of project. Sets technical criteria, reviews, and approves technical progress. Ensures that all participating personnel have proper training. ***Note***: Other titles such as project manager may be used.

Field Team Leader (FTL) - Supervises field operations. Ensures that all necessary equipment, including safety equipment, is available and functioning properly before project operations begin. Ensures that all necessary personnel are mobilized on time. Maintains daily log of activities each work day.

Field Geologist - Collects and maintains data. Coordinates and consults with site manager on decisions relative to unexpected encounters during testing and deviations from this SOP. Directs overall activities of testing procedures and support subcontractors.

4.0 Required Equipment

Water measuring and recording:

- Pressure transducers and data logger
- Personal computer for viewing and downloading data
- Water level measuring device
- Stopwatch
- Field logbook
- Decontamination equipment and supplies
- Data on construction of the pumping well (depth to screen and screen length)

Water pumping, treating, storing, and discharging:

- Pump (sufficient capacity to withdraw at the required rate) with electric wiring
- Discharge hosing/piping
- Electrical source (e.g., generator)
- Flowmeter with totalizer
- Sampling valve
- Water treatment unit (if required)
- Water storage container (if required)
- Ancillary equipment and supplies to install and/or operate the main equipment

A field service subcontractor will typically be responsible for providing and operating the equipment for pumping, treating, storing, and discharging water. However, in some cases, it may be appropriate for the pumping, treating, storing, and/or discharging equipment to be provided and operated by those that also provide and operate the water measuring and recording equipment. The project requirements and structure will need to be evaluated to determine the most suitable arrangement for providing and operating the necessary equipment.

5.0 Procedures

An APT has five main components:

- Preparation
- Continuous background monitoring
- Step-drawdown test
- Long-term constant rate test
- Discharge water management

Sometimes only the long-term constant rate test is performed and the background monitoring and the step-drawdown tests are omitted. Therefore, the long-term test is sometimes referred to as an APT.

A form that provides typical general information that should be recorded for each test is provided at the end of this SOP.

5.1 Preparation

Adequate attention to the planning and design of the APT is a significant phase of the procedure and will ensure that useful results are produced (U. S. Geological Survey 1976, U. S. Environmental Protection Agency 1993). A planning meeting shall be held to identify the objectives of the APT and then the scope of the APT shall be developed. After the objectives are identified and the scope is developed, an APT plan shall be prepared that describes the procedures to be followed. The plan shall identify and describe the details to be followed for each component of the APT.

5.2 Continuous Background Monitoring

Water levels shall be collected continuously prior to performing the long-term test. Adjacent surface water bodies should also be monitored. The water levels shall be used to reduce and analyze the data collected during the long-term test. The background data is also useful in characterizing the hydrogeologic framework.

Transducers/loggers shall be installed in the pumping well and the observation wells. Each transducer/logger shall be checked and set following the manufacturer's manual, including setting the internal clock to a common external standard. Each transducer shall be installed to a depth that does not exceed the working capacity of the transducer and where the water level will not drop below the transducer during ambient water level changes. After the selected depth is reached with the transducer:

- Securely attach the cable to the well head and mark a reference point with electrical tape to allow verification that the transducer position does not change during the test
- Read the depth of water using the transducer (note that the transducer may need to equilibrate with the water temperature following the manufacturer's specifications and recover from displacement of water caused by submersion of the transducer)
- Collect a manual water level measurement from the well's measuring point
- Begin recording water levels on a linearly rate of 1 reading per 30 minutes

Transducers shall be programmed so that water level recording begins at the same time at each well. Having water levels recorded at the same time for each well simplifies the data reduction and evaluation activity contrasted to having water levels recorded at different times for different wells.

Background water levels shall be recorded for 7 days. During the monitoring period, the transducers/loggers should be occasionally checked (e.g., check the transducers on day two and day five) to verify that the equipment is working properly. Manual water level measurements should be taken and recorded during this check. Replace any transducer that is identified to be not operating correctly.

At the end of the monitoring period, stop the test recording and download the recorded data.

Barometric pressure (BP) and precipitation shall be recorded during the background monitoring period. These two elements are commonly considered the main natural factors to impact groundwater levels. If publicly available data can be obtained from a weather station located nearby (within approximately 5 miles of the project), the data from that station may be used. BP and precipitation data shall also be recorded during the long-term test.

5.3 Step Drawdown Test

The step drawdown test (or simply, step test) is required to determine the constant pumping rate that will be used for the subsequent long-term test and to assess well efficiency. Step test data may also be used to evaluate the hydrogeologic characteristics. The step test is performed at the pumping well. In summary, the step drawdown test consists of pumping water from the well at short incrementally increased rates (steps) so that a withdrawal rate can be determined for the long-term test.

Aquifer Performance Tests

SOP 4-9
Revision: 2
Date: February 2015

A pump capable of yielding 1.5 times the estimated yield of the pumping well shall be installed to the specified depth. A vertical check valve will be placed in the discharge line immediately above the pump or intake to prohibit water from draining into the well when the pumping ceases. A 1-inch diameter polyvinyl chloride line will be placed in the well with the bottom end open to a depth within 1 foot from the top of the pump. Several ¼-inch diameter holes should be drilled in the bottom 5 feet of this stilling pipe. The water level transducer will be installed in the pipe. After the pumping equipment and transducer are installed, the following steps will be followed:

- Connect a flow meter/totalizer and sample tap with valve to the discharge line from the pump; direct the discharge line to the system to handle the water. Care must be taken to provide sufficient straight sections of pipe above and below the flow meter to obtain accurate measurements. Recent calibration certificates should be obtained for the flow meter.
- Record the volumetric reading on the totalizer (***Note***: Prior to pumping and increasing pumping rate and after ending pumping, the volumetric reading should be recorded).
- Measure and record the static water level in the pumping well.
- Begin logging with the transducer and then start pumping water from the pumping well at a relatively low (approximately ½ of the estimated yield) but steady rate (STEP 1); logging should be started approximately 2 to 5 seconds prior to starting pumping. Flow should be adjusted to maintain a constant rate, noting when changes are made.
- Record the time at which pumping is started, using a clock that is synchronized with the transducer clocks, and the flow rate; check operation of the transducer.
- Monitor the water level in the pumping well with the transducer and confirm periodically with manual measurements.
- After approximately 1½ hours, increase the pumping rate to approximately ¾ of the estimated yield, and continue to monitor the water level for approximately 2 hours (STEP 2).
- Record the time at which the pumping rate is increased and the new flow rate; check operation of the transducer.
- Approximately 2 hours after increasing the pumping rate for STEP 2, increase the pumping rate to approximately equal to the estimated yield, and continue to monitor the water level for approximately 2 hours (STEP 3).
- Record the time at which the pumping rate is increased and the new flow rate.
- Approximately 2 hours after increasing the pumping rate for STEP 3, increase the pumping rate to approximately 1.5 x the estimated yield, and continue to monitor the water level for approximately 2 hours (STEP 4).
- Record the time at which the pumping rate is increased and the new flow rate.
- Shut off the pump at the end of STEP 4 (maximum of 8 hours has elapsed since pumping started at the beginning of the test) and download data. The transducer should continue recording during the recovery period.

A step test is dynamic. During each step the operator will gain more information on how the well's water level responds to specified pumping rates. The estimated increases identified above for each step should only be used as a guide. Each successive increase should be based on the operator's general understanding of well hydraulics, observations made while installing and developing the well, and on the well's response during the previous step(s). The goal, in summary, is to achieve the well yield at STEP 3 and exceed the well yield at STEP 4.

During the test, water levels at the pumping well shall be recorded logarithmically following the recommended schedule in the following chart. Typical data loggers have default sample intervals except for the largest sample interval, which is set by the user (in the table below, the 10-minute sample interval is set by the user). The default sample intervals shall be equal to or similar to the table below.

Log Cycle	*Elapsed Time*	*Sample Interval*	*Points/Cycle*
1	0 to 20 seconds	0.2 second	101
2	20 to 60 seconds	1 second	40
3	1 to 10 minutes	10 seconds	54
4	10 to 100 minutes	2 minutes	45
5	100 to 1,000 minutes	10 minutes	90

The drawdown-time data shall be plotted semi-logarithmically. The drawdown (y-axis) shall be plotted on a linear scale and time (x-axis) shall be plotted on a logarithmic scale. The drawdown curves shall be extrapolated to the specified time of the proposed long-term test. The rate that results in the maximum drawdown without dropping the water level below the design pumping level within the time period of the long-term test shall be considered the flow rate to be used for the long-term test. The specific capacity versus pumping rate should also be plotted to determine if excessive well losses occur at the selected rate.

5.4 Long-Term Constant Rate Test

The long-term constant rate test will be performed at the pumping well. Water levels will be monitored in the pumping well and the observation wells. The same pumping equipment used for the step test will be used for the long-term test. BP and precipitation shall be recorded during the long-term test. If publicly available data can be obtained from a weather station located nearby (within approximately 5 miles of the project), the data from that station may be used. Adjacent surface water bodies should also be monitored if the surface water is potentially connected to the groundwater system.

The time interval for the long-term constant rate test shall be specific to the project. However, at a minimum, a confined aquifer should be pumped for 24 hours and an unconfined aquifer to be pumped for 72 hours (American Water Works Association 1997). The project objectives will need to be reviewed and aquifer test solution requirements considered so that the correct pumping period is selected. The following steps shall be followed to conduct the long-term test after the step test is completed.

- Install transducers in the pumping well and the observation wells (note that transducers can be installed in observation wells prior to the day the long-term test starts).
- Read the water level depths with the transducers and record the values; measure and record the static water levels with the electronic water level meter from the wells' measuring points.
- Record the volumetric reading on the totalizer.
- Begin logging water level data with the transducers and then start pumping at the predetermined rate (determined based on the step-drawdown test results).
- Periodically monitor discharge rate and transducers; maintain constant pumping rate.
- Stop pumping at the end of the specified time, record volumetric reading on the totalizer.
- Continue to record water level data with transducers until the water level in the pumping well has recovered so that sufficient data are collected to adequately analyze the recovery or a maximum of 24 hours has elapsed.

The water level data will be transferred to disk form so that it may be reduced, analyzed, and put into report format.

The water levels in the wells will be recorded logarithmically following the recommended schedule in the following chart:

Log Cycle	*Elapsed Time*	*Sample Interval*	*Points/Cycle*
1	0 to 20 seconds	0.2 second	101
2	20 to 60 seconds	1 second	40
3	1 to 10 minutes	10 seconds	54
4	10 to 100 minutes	2 minutes	45
5	>100 minutes	10 minutes	unspecified

When the pump is shut off and recovery begins, a new logarithmic series will be started for the transducer in the pumping well. The series shall be started 1 to 5 seconds prior to ending the pumping activity. The transducers in the observation wells will continue to monitor on the first logarithmic cycle series. If the aquifer is expected to recover quickly, the observation well transducers may also be restarted on a new series. Data will be recorded until the water level in the pumping well has returned so that sufficient data are collected to adequately analyze the recovery or until a maximum of 24 hours has elapsed. A manual water level measurement shall be collected from the wells, measuring points, and a reading should be taken with the transducers during recovery.

At the conclusion of the recovery test, the data logging shall be stopped at each well and the transducers shall be removed and the data downloaded.

5.5 Discharge Water Management

The water pumped from the well shall be discharged and managed following the plan specific to the project. Several methods may be used to handle the discharge water from an APT. The water may be discharged:

- Directly to the ground surface or a water body, if permitted by the regulatory agencies. Such discharge should be at a sufficient distance from the pumping and observation wells so that the test is not impacted if water infiltrates to the aquifer.
- To a holding tank, sampled and analyzed after the test, and then released to the ground surface or water body after analytical results prove that discharge requirements are met.
- To a unit designed and constructed to treat the water to meet discharge criteria; treated and then released to the ground surface or water body.

Also, a combination of the three options above may be used. Other discharge options may also be available and followed.

In summary, several different methods are typically available to handle discharge water. The governing agency shall be contacted so that required water handling practices are followed and discharge criteria are met.

6.0 Data Reduction and Analysis

The data sets from an APT are typically very robust. The data may be reduced and analyzed to:

- Determine the specific capacity and safe yield of the well
- Calculate the properties (T and S) of the aquifer
- Characterize the hydrogeologic framework at and near the investigation area

These three items, or one of the items at a minimum, are typically evaluated with APT data. Other pumping test data may also be available and evaluated.

APT data are recommended to be analyzed with computer software; however, data may also be analyzed manually. The groundwater modeling tool kit contains Aquifer^{Win32} (ESI International), which is a program that may be used to assist in analyzing test data. Other programs are also available. Software packages are useful since they can be used to manage a significant amount of data in short time periods and contain many different confined and unconfined test solutions. The trained user can use these benefits to generate detailed response curve graphs, precise hydraulic values, and insights into the hydrogeologic framework near the well. Regardless of the analytical method employed or whether the data is analyzed manually or by computer, the analyst should review the original technical paper or textbook summary of the method in order to understand the mechanics and assumptions underlying the method prior to attempting any analysis and verify the method is appropriate for the site conditions.

APT data analyses and hydraulic property calculations shall be performed by an experienced professional, documented in a calculation brief, and reviewed. Data analysis and parameter calculations are beyond the scope of this SOP and, therefore, are not discussed here.

7.0 Restrictions/Limitations

This procedure describes the standard steps used to conduct a constant rate APT. Since APTs are complex and project objectives and site requirements vary, not every step or possible method was incorporated into the procedure.

A planning meeting shall be held to identify the objectives of the APT, then the scope of the APT shall be developed. After the objectives are identified and the scope is developed, an APT plan shall be prepared that describes the project-specific procedures to be followed. The plan shall describe the details to be followed for each component of the APT. The objectives of the test shall be specified so that the necessary data to reach the objectives are collected when the test is performed.

8.0 References

American Society for Testing and Materials. 2010. Standard Guide for Selection of Aquifer Test Method in Determining Hydraulic Properties by Well Techniques. D 4043-96 (Reapproved 2010).

American Water Works Association. 1997. AWWA Standard for Water Wells (ANSI/AWWA A 100-97).

ESI International, see their website, http://esinternational.com, for current information on Aquifer^{win32}

U. S. Environmental Protection Agency. 1993. Ground Water Issue Suggested Operating Procedures for Aquifer Pumping Tests (EPA/540/S-93/503). February.

U. S. Geological Survey. 1976. Techniques of Water-Resources Investigations of the United States Geological Survey: Chapter B1 Aquifer Test Design, Observation and Data Analysis.

Aquifer Performance Tests

SOP 4-9
Revision: 2
Date: February 2015

Aquifer Test Data

Project Name:	Date:
Pumped Well ID:	Weather:
Observation Well ID:	Personnel:
Well locations (*provide sketch or attach map*):	
Include: Scale/dimensions, north arrow, and significant features (*e.g., surface water*)	
This sheet records data for (*well ID*):	
Measuring Point: (*e.g., notch or inner casing*)	
Static Water Level:	(*feet below measuring point [ft BMP]*)
Static Water Level Date:	Time:
Interval Open/Screened to Aquifer (*ft BMP*):	
Pump Setting Depth (*ft BMP*):	
Pump Model:	Serial No.:
Flow Meter Model:	Serial No.:
Logger/Transducer Model:	Serial No.:
Totalizer Reading before Pumping:	
Date/Time Pumping Started:	
Discharge Rate (*gpm*):	

Date	*Time*	*Manual Water Level Measure (ft BMP)*	*Discharge (gpm)*	*Comments*

*Use more sheets if more rows are needed.

Date/Time pumping ended: _____

Totalizer reading at end of pumping: _____

Borehole and Well Decommissioning

SOP 4-10
Revision: 2
Date: February 2015

Approved: Signature **Technical Review:** David Schroeder

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to provide guidelines for the decommissioning of boreholes and wells. These guidelines help to produce consistency of approach in the planning and implementation of borehole and well decommissioning. Individual decommissioning procedures shall probably vary in some respects as a result of varying borehole or well construction materials and methods and regulatory framework.

2.0 Background

2.1 Definitions

Note: The definitions provided are generalized. Definitions are often codified in state or local regulations and should be verified against the definitions presented in this SOP before commencement of work.

Decommissioning - The engineered closure of a well, borehole, or other subsurface monitoring device sealed with plugging materials. Decommissioning also includes the planning and documentation of all associated activities. Common synonyms for "decommissioning" include "destroying," "plugging," and "abandoning," and the term used can vary between regulatory agencies.

Grout - Material consisting of bentonite, cement, or a cement-bentonite mixture.

Overdrilling - The process of drilling out a well casing and any material placed in the annular space.

Plugging Material - A material that has a hydraulic conductivity equal to or less than that of the geologic formation(s) to be sealed. Typical materials include portland cement and bentonite.

Tremie Pipe - A pipe or tube that is used to transport cement, bentonite, or other plugging materials from the ground surface to a specified depth in a borehole or well. The material may be allowed to flow freely or it may be injected under pressure.

Well Casing - Impervious durable pipe placed in a well to prevent the borehole walls from caving and to help seal off surface drainage or undesirable water, gas, or other fluids from entering the well.

2.2 Associated Procedures

- SOP 2-2, *Guide to Handling of Investigation-Derived Waste*
- SOP 3-5, *Lithologic Logging*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-2, *Photographic Documentation of Field Activities*
- SOP 4-4, *Design and Installation of Monitoring Wells in Aquifers*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.3 Discussion

This SOP is intended to cover the decommissioning of boreholes during environmental investigations and remediation efforts. It is intended to be a general guideline listing the types of materials and methods to be considered when a borehole or well is decommissioned. Materials are not specified in detail since it is likely there will be wide variability required to meet the needs of individual site conditions, specific clients, and regulatory requirements. If there is a conflict between this

SOP and procedures given by the federal, state, and/or local regulatory agencies involved in the decommissioning process, the materials and procedures given by the regulatory agencies shall take precedence.

3.0 General Responsibilities

Field Team Leader - The field team leader (FTL) is responsible for ensuring that borehole and well decommissioning activities are conducted in accordance with this SOP and any site-specific work plans, scopes of work, quality assurance plan, and site health and safety plan.

Field Geologist - The field geologist is responsible for understanding and implementing this SOP during all field activities, as well as obtaining the appropriate permits, field logbooks, forms, and records necessary to complete the field activities. The field geologist collects and maintains data by providing oversight of the decommissioning procedures by the drilling subcontractor and completing the CDM Smith Borehole/Well Decommissioning Record (Figure 1). The field geologist coordinates and consults with the FTL on decisions relative to unexpected encounters during field activities and deviations from this SOP. The field geologist directs overall activities of drill and support subcontractors.

Drilling Subcontractor - The drilling subcontractor provides the necessary equipment and materials for borehole and monitoring well decommissioning. This generally includes a drill rig or service truck with grouting equipment and appropriate backfill materials such as portland cement, potable water, and bentonite powder and/or chips. The drilling subcontractor is responsible for submitting forms and records required by law or regulations to the applicable state and/or local regulatory agency and as required in the subcontractor statement of work.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Required Equipment and Materials

4.1 Required Field Equipment and Supplies

- Water level indicator
- Personal protective equipment (PPE)
- Tape measure (weighted tip)
- Camera
- Health and safety equipment
- Explosimeter, OVA (FID) or OVM per health and safety plan
- Bound field logbook
- CDM Smith Borehole/Well Abandonment Form
- Backfill materials (e.g., portland cement, potable water, and bentonite powder and/or chips)
- Global positioning system (GPS) equipment (if required)
- Downhole video equipment (if required)

4.2 Required Construction Materials

Note: The materials that are used in the decommissioning of a borehole or well and that come in contact with groundwater shall not measurably alter the chemical quality of the groundwater source.

Water - Water, which may be used in the well decommissioning process, shall be obtained from a source that does not contain constituents that could compromise groundwater quality. A certificate of analysis should be provided with the water, potable water from an approved source should be used, or a sample of the water should be analyzed and documented as contaminant-free.

Plugging Materials - The materials used to plug the borehole or well casing may be prepared as a slurry or used unmixed in a dry pellet form, depending on the application. Plugging materials shall be selected for compatibility with local geologic, hydrogeologic, climatic, and human-induced conditions in the subsurface.

Grout - The grout used for decommissioning boreholes or wells shall be a liquid slurry consisting of water, bentonite of Volclay or equivalent quality, and/or portland cement. Bentonite-based grout is typically used when a more flexible grout is desired (i.e., freeze-thaw). Cement-based grout provides a more rigid plugging material. A typical bentonite slurry mixture is a minimum 20 percent solids slurry by weight. Cement-bentonite grout is typically 6 to 7 gallons of water per 94 pound bag of Type I portland cement and 2.7 percent bentonite powder.

Bentonite - Bentonite shall be powdered, chipped, or pelletized sodium montmorillonite furnished in sacks or buckets from a commercial source and free of impurities that adversely impact water quality in the subsurface. Pellets or chips are typically used for plugging shallow boreholes and powdered bentonite is used for mixing in grout.

Cement - Each type of cement has slightly different characteristics that may be appropriate under various physical and chemical conditions. Cement shall generally be portland Type I, Type II, or Type I/II as specified in ASTM C 150. Quicksetting cements containing additives are not allowable for use in decommissioning. Additives may leach from the cement and influence the chemistry of the groundwater.

Tremie Pipe - A tremie pipe is used to inject grout-based plugging materials. Tremie pipes are typically constructed of PVC or galvanized steel. Associated equipment may include a trough or mixing box and “mud pump” to place the material.

5.0 Procedures

The actual methods of decommissioning boreholes or wells at a site vary depending on site conditions and the construction materials and methods used during the original installation. The method to be used at a site shall be stated in the site-specific plans. Deviations from the methods prescribed in these plans shall be approved by the FTL or designee.

A description of plugging materials, volume calculations, and/or overdrilling observations needs to be recorded on the CDM Smith Borehole/Well Decommissioning Form (Figure 1) or in a logbook (SOP 4-1).

5.1 Pre-Decommissioning Preparation

Well construction records (if available) should be obtained before field mobilization to understand the construction of the wells requiring decommissioning. Records are used to verify depths and construction materials within the well before start of decommissioning activities and as decommissioning activities are completed. The records may also be required by state and/or local regulatory agencies as part of the decommissioning permitting process.

State or local regulations may require either pre- and post-notification or a permit during borehole or well decommissioning activities; each borehole or well at a site may require individual notification and/or permits. Before initiating field work, state and local regulations shall be researched and the notification and/or permitting procedures provided by the regulatory agencies shall be followed. Some state and local regulations require the driller or drilling company licensed in the jurisdiction to submit the required decommissioning paperwork and obtain the permits from the regulatory agencies. The drilling subcontractor statement of work should clearly designate whether the subcontractor or CDM Smith is responsible for submitting decommissioning paperwork to the regulatory agencies in accordance with the regulations.

5.2 Borehole or Well Inspection

Before decommissioning, each borehole or well shall be inspected for obstructions (including but not limited to pumps, sampling equipment, debris, and other undesirable materials that might interfere with decommissioning) and remove them. Disposal of materials removed from boreholes or wells shall be in accordance with the site-specific plans and SOP 2-2.

Total depth of the borehole or well should be verified using a weighted tape, compared to any records obtained from original borehole or well installation, and recorded on the CDM Smith Borehole/Well Decommissioning Form (Figure 1) or in a logbook (SOP 4-1). The preexisting condition of the borehole or well prior to decommissioning shall be recorded in the logbook and photographed in accordance with SOP 4-2. Some state or local regulatory agencies may have additional pre-decommissioning requirements such as field location of the borehole or well using GPS equipment or downhole video inspection of the well before decommissioning. This information should also be recorded on the borehole/well decommissioning form and in the logbook.

5.3 Well Overdrilling (Not Applicable to Borehole Decommissioning)

The requirement for overdrilling wells during decommissioning shall vary to meet the needs of individual site conditions, specific clients, and regulatory requirements. Some state and local regulatory agencies require all well construction materials to be removed from the borehole using an overdrilling method before the remaining borehole can be plugged. It is generally preferable to pull the well casing from the subsurface by overdrilling the annular space of the well and remove the casing in whole sections. Site-specific conditions and well construction materials may preclude removal of all casing materials using an overdrilling/pulling method.

The actual methods for overdrilling wells at a site shall vary depending on site conditions and the construction materials and methods used during the original well installation. The method to be used at a site shall be stated in the site-specific plans. Deviations from the methods prescribed in these plans shall be approved by the FTL or designee. Typical overdrilling methods include air rotary, mud/fluid rotary, rotosonic, and hollow-stem auger. Drilling with mud or water is least desirable, but the driller shall have the capability to use this method if well conditions warrant it. The wells shall be overdrilled to the well depth specified in the site-specific plans but may vary based on actual well construction depth (determined in the field) required to remove well construction materials. Drillers must prevent grease, oil, and other fluids from the drill rig from coming in contact with the ground around the area of well installation. Disposal of materials removed from wells shall be in accordance with the site-specific plans and SOP 2-2.

A description of overdrilling observations needs to be recorded on the CDM Smith Borehole/Well Decommissioning Form (Figure 1) or in a logbook (SOP 4-1). Unusual conditions or materials observed during overdrilling shall be photographed in accordance with SOP 4-2.

5.4 Borehole or Well Plugging

The following processes apply to both boreholes and wells. It should be noted that plugging processes shall vary with the total depth of the borehole or well and/or the type of well construction. Generally, plugging using bentonite pellets or chips is only allowable for shallow boreholes less than 10 feet below ground surface (bgs). Chips should not be used for plugging small-diameter boreholes because of bridging concerns due to their angular shape. The method to be used at a site for a particular borehole or well shall be stated in the site-specific plans. Deviations from the methods prescribed in these plans shall be approved by the FTL or designee.

The CDM Smith field geologist shall calculate the amount of plugging material needed to fill the borehole or well casing to complete decommissioning. These calculations should be completed in the field after borehole or well depths and dimensions have been inspected and verified. The volume and depth of plugging materials used for borehole or well decommissioning shall be documented on the CDM Smith Borehole/Well Decommissioning Form (Figure 1) or in a logbook (SOP 4-1).

5.4.1 Plugging Using Bentonite Pellets or Chips

Boreholes completed to a depth less than 10 feet may be backfilled with bentonite pellets or chips and hydrated until the pellets or chips come within 6 inches of the surface. The chips shall be installed by gravity feed down the center of the borehole. The depth of the pellets or chips shall be tracked using a weighted tape to ensure there is no bridging. The chips shall be hydrated at 2-foot intervals using sufficient quantities of clean water to allow for adequate saturation and expansion of the bentonite.

5.4.2 Plugging Using Grout

Boreholes greater than 10 feet in depth and wells shall be plugged using grouting methods. A sufficient volume of grout shall be premixed onsite, according to procedure stipulated by the manufacturer, to compensate for unexpected losses to the formation. This process shall be checked against the calculated volume of the borehole or well casing to be decommissioned to ensure that bridging does not occur during emplacement. The use of alternate grout materials, including grout containing portland cement, may be necessary to control zones of high grout loss. The mixing (and placing) of grout shall be performed with recorded weights and volumes of materials, according to procedures stipulated by the manufacturer. Lumpy grout shall not be used in an effort to prevent bridging within the tremie and the well. Bentonite-based grout of Volclay or equivalent type shall be mixed to the manufacturer's specifications and then pumped into place using minimum pump pressure. All additives to grouts shall be evaluated for their effects on the subsurface.

Depending upon the borehole or well depth and/or construction, plugging may be accomplished using a pressure grouting technique or by gravity feed through a tremie pipe. With either method, grout is introduced in one continuous operation until grout flows out at the ground surface without evidence of drill cuttings or fluid. The grout shall be injected under pressure using a tremie pipe when possible to reduce the possibility of leaving voids in the annular seal and to displace any liquids or drill cuttings that may remain in the borehole or well casing. The tremie pipe shall be kept full of grout from start to finish with the discharge end of the pipe completely submerged as it is slowly and continuously lifted. Pump pressure shall be kept to a minimum.

Approximately 5 to 10 feet of tremie pipe shall remain submerged during group emplacement. If possible, steel tape soundings shall be made to ensure the level of the tremie material is in agreement with the calculated volume and that the desired placement of plugging materials is achieved. A staged grouting procedure may be considered if there is significant grout loss into the formation because of the weight exerted by the full column of grout as it sets. If used for borehole or well drilling, temporary casing or hollow-stem augers shall be removed in increments (immediately following each lift of grout installation) well in advance of the time when the grout begins to set. The initial grout mixture must be allowed to cure for approximately 24 hours, then checked and refilled to within 6 inches of the surface.

5.5 Surface Restoration

Surface completion materials for wells (such as protective casing, bumper posts, and concrete pads) should be removed to the extent possible. If overdrilling is required, removal of surface completion materials shall be performed before the overdrilling operation. Well and surface casings (if left in the subsurface) should be removed to an approximate depth of 2 feet bgs before surface restoration. Disposal of materials removed from wells shall be in accordance with the site-specific plans and SOP 2-2.

The upper 6 inches of the borehole or well that has been decommissioned shall be completed with similar surface material (i.e., sod, asphalt, concrete) as the surrounding area. In the case of soil boreholes completed in an area absent of surface material, the overlying soil shall be used to backfill the remaining 6 inches of the borehole until flush with the surrounding surface.

5.6 Post Operation Procedures

5.6.1 Field Procedures

At the conclusion of the borehole or well decommissioning activities, all equipment coming in contact with the subsurface must be decontaminated (according to SOP 4-5) before moving the equipment to a different work location. All water used in the decontamination of equipment shall be contained in an appropriate container, if required in the site-specific plans. Disposal of decontamination water shall be in accordance with the site-specific plans and SOP 2-2.

5.6.2 Documentation

The CDM Smith Borehole/Well Decommissioning Form (Figure 1) shall be completed by the CDM Smith field geologist or designee at the conclusion of the field activity. Any post-notification or decommissioning paperwork to be submitted to regulatory agencies shall be completed by the drilling subcontractor or CDM Smith designee as required by regulations.

Copies of all field notes, the daily logs, and the CDM Smith Borehole/Well Decommissioning Forms shall be given to the FTL. These records shall be maintained in the project and document control files. At a minimum, all materials used for decommissioning shall be documented by entering identifying numbers (lot numbers, manufacturer's identification, etc.) in the field logbook and on the CDM Smith Borehole/Well Decommissioning Form. Samples of decommissioning materials (including grout, sand, etc.) may be archived if specified in the site-specific plans.

6.0 Restrictions/Limitations

Check with the federal, state, and local agencies for specific decommissioning procedures. If no specific decommissioning procedures are identified, the decommissioning materials and methods provided in this procedure shall be followed.

7.0 References

American Society for Testing and Materials (ASTM). 1999 (Reapproved 2012)e1. *Standard Guide for Decommissioning Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities*. Standard Method D 5299-99.

U. S. Army Corps of Engineers (USACE). Nov. 1, 1998. *Engineering Manual (EM) 1110-1-4000. Monitoring Well Design, Installation, and Documentation at Hazardous, Toxic, and Radioactive Waste Sites.*

Example

CDM Smith Borehole/Well Decommissioning Record

Borehole/Well Identification No. _____

Facility or Project Area:	**Site:**	**Job No:**
Recorded By:	**Date:**	**Checked By:**

Borehole/Well Decommissioning Permit No. (if applicable): _____

Regulatory/Permitting Agency: _____ Contact: _____ Phone No.: _____

Original Borehole/Well Construction Records Available and Reviewed? (Attach) Yes ___ No ___

Condition of Borehole/Well at Ground Surface before Decommissioning: _____

Maximum Depth Measured in Borehole/Well: _____ Datum: _____

Original Borehole/Well Bore Depth from Records (if available): _____

Downhole Obstruction Indicated? Yes _____ No _____

If yes, describe the method(s) used to assess the nature of the obstruction and/or methods to remove it: _____

Depth (feet) to Water before Borehole/Well Decommissioning: _____ Datum: _____

Date of water level measurement: _____

Any Indications of Borehole/Well Contamination? Yes _____ No _____

If yes, describe field evidence suggesting borehole/well contamination: _____

Plugging/Sealing Material Used for Decommisioning and Corresponding Depth Intervals:

Plugging Material	**From**	**To**	**Qty. Sacks Cement**	**Qty. Sacks Sand**	**Qty. Sacks Bentonite**	**Qty. Sacks Aggregate**	**Qty. Gal. Water**

Manufacturer/Supplier Information for Plugging Materials:

Surface Restoration Materials (describe): _____ From: _____ To: _____

Comments:

Control of Measurement and Test Equipment

SOP 5-1
Revision: 10
Date: February 2015

Approved: Signature **Technical Review:** Stuart Barden

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to establish the baseline requirements, procedures, and responsibilities inherent to the control and use of all measurement and test equipment (M&TE). Contractual obligations may require more specific or stringent requirements that must also be implemented.

2.0 Background

2.1 Definitions

Traceability - The ability to trace the history, application, or location of an item and like items or activities by means of recorded identification.

2.2 Associated Procedures

- SOP 4-1, *Field Logbook Content and Control*
- CDM Smith Quality Procedures (QPs) 2.1 and QP 6.3
- Manufacturer's operating and maintenance and calibration procedures

2.3 Discussion

M&TE may be government furnished (GF), rented or leased from an outside vendor, or purchased. It is essential that measurements and tests resulting from the use of this equipment be of the highest accountability and integrity. To facilitate that, the equipment shall be used in full understanding and compliance with the instructions and specifications included in the manufacturer's operations and maintenance and calibration procedures and in accordance with any other related project-specific requirements.

3.0 Responsibilities

All staff with responsibility for the direct control and/or use of M&TE are responsible for being knowledgeable of and understanding and implementing the requirements contained herein as well as any other related project-specific requirements.

The project manager (PM) or designee (equipment coordinator, quality assurance coordinator, field team leader, etc.) is responsible for initiating and tracking the requirements contained herein.

***Note*:** Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Requirements for M&TE

- Determine and implement M&TE related project-specific requirements
- The maintenance and calibration procedures must be followed when using M&TE
- Obtain the maintenance and calibration procedures if they are missing or incomplete
- Attach or include the maintenance and calibration procedures with the M&TE
- Prepare and record maintenance and calibration in an equipment log or a field log as appropriate (Figure 1)
- Maintain M&TE records
- Label M&TE requiring routine or scheduled calibration (when required)
- Perform maintenance and calibration using the appropriate procedure and calibration standards
- Identify and take action on nonconforming M&TE

5.0 Procedures

5.1 Determine if Other Related Project-Specific Requirements Apply

For all M&TE:

The PM or designee shall determine if M&TE related project-specific requirements apply. If M&TE related project-specific requirements apply, obtain a copy of them and review and implement as appropriate.

5.2 Obtain the Operating and Maintenance and Calibration Documents

For GF M&TE that is to be procured:

Requisitioner - Specify that the maintenance and calibration procedures be included.

For GF M&TE that is acquired as a result of a property transfer:

Receiver - Inspect the M&TE to determine whether maintenance and calibration procedures are included with the item. If missing or incomplete, order the appropriate documentation from the manufacturer.

For M&TE that is to be rented or leased from an outside vendor:

Requisitioner - Specify that the maintenance and calibration procedures, the latest calibration record, and the calibration standards certification be included. If this information is not delivered with the M&TE, ask the procurement division to request it from the vendor.

5.3 Prepare and Record Maintenance and Calibration Records

For all M&TE:

PM or Designee - Record all maintenance and calibration events in a field log unless other project-specific requirements apply.

For GF M&TE only (does not apply to rented or leased M&TE):

If an equipment log is a project specific requirement, perform the following:

Receiver - Notify the PM or designee for the overall property control of the equipment upon receipt of an item of M&TE.

PM or Designee and User:

- Prepare a sequentially page numbered equipment log for the item using the maintenance and calibration form (or equivalent) (Figure 1).
- Record all maintenance and calibration events in an equipment log.

5.4 Label M&TE Requiring Calibration

For GF M&TE only (does not apply to rented or leased M&TE):

If calibration labeling is a project specific requirement, perform the following:

PM or Designee:

- Read the maintenance and calibration procedures to determine the frequency of calibration required.
- If an M&TE item requires calibration before use, affix a label to the item stating “Calibrate Before Use.”
- If an M&TE item requires calibration at other scheduled intervals, e.g., monthly, annually, etc., affix a label listing the date of the last calibration, the date the item is next due for a calibration, the initials of the person who performed the calibration, and a space for the initials of the person who shall perform the next calibration.

5.5 Operating, Maintaining or Calibrating an M&TE Item

For all M&TE:

PM or Designee and User - Operate, maintain, and calibrate M&TE in accordance with the manufacturer’s maintenance and calibration procedures. Record maintenance and calibration actions in the equipment log or field log.

5.6 Shipment

For GF M&TE:

Shipper - Inspect the item to ensure that the maintenance and calibration procedures are attached to the shipping case, or included, and that a copy of the most recent equipment log entry page (if required) is included with the shipment. If the maintenance and calibration procedures and/or the current equipment log page (if required) is missing or incomplete, do not ship the item. Immediately contact the PM or designee and request a replacement.

For M&TE that is rented or leased from an outside vendor:

Shipper - Inspect the item to ensure that the maintenance and calibration procedures and latest calibration and standards certification records are included prior to shipment. If any documentation is missing or incomplete, do not ship the item. Immediately contact the procurement division and request that they obtain the documentation from the vendor.

Note that some M&TE equipment must remain upright to maintain calibration. Such M&TE should be shipped only in containers labeled appropriately with "This End Up" labels.

Receiver – Some M&TE equipment must remain upright to maintain calibration. Upon receipt, inspect the container to verify that it is upright per "This End Up" labels. If it is not upright, notify the PM and vendor.

5.7 Records Maintenance

For GF M&TE:

PM or Designee - Create a file upon the initial receipt of an item of M&TE or calibration standard. Organize the files by contract origin and by M&TE item and calibration standard. Store all files in a cabinet, file drawer, or other appropriate storage media at the pertinent warehouse or office location.

Receiver - Forward the original packing slip to the procurement division and a photocopy to the PM or designee.

PM or Designee and User:

- Maintain all original documents in the equipment file except for the packing slip and field log.
- File the photocopy of the packing slip in the M&TE file.
- Record all maintenance and calibration in an equipment log or field log (as appropriate). File the completed equipment logs in the M&TE records. Forward completed field logs to the PM for inclusion in the project files.

For M&TE rented or leased from an outside vendor:

Receiver - Forward the packing slip to the procurement division.

User:

- Forward the completed field log to the PM for inclusion in the project files.
- Retain the most current maintenance and calibration record and calibration standards certifications with the M&TE item and forward previous versions to the PM for inclusion in the project files.

5.8 Traceability of Calibration Standards

For all items of M&TE:

PM or Designee and User:

- When ordering calibration standards, request nationally recognized standards as specified or required. Request commercially available standards when not otherwise specified or required. Or, request standards in accordance with other related project-specific requirements.
- Require certifications for standards that clearly state the traceability.
- Require Material Safety Data Sheets to be provided with standards.
- Note standards that are perishable and consume or dispose of them on or before the expiration date.

5.9 M&TE That Fails Calibration

For any M&TE item that cannot be calibrated or adjusted to perform accurately:

PM or Designee

- Immediately discontinue use and segregate the item from other equipment. Notify the appropriate PM and take appropriate action. in accordance with the CDM Smith QP 6.3 for nonconforming items.
- Review the current and previous maintenance and calibration records to determine if the validity of current or previous measurement and test results could have been affected and notify the appropriate PM(s) of the results of the review.

6.0 Restrictions/Limitations

On an item-by-item basis, exemptions from the requirements of this SOP may be granted by the Headquarters health and safety manager and/or Headquarters quality assurance director. All exemptions shall be documented by the grantor and included in the equipment records as appropriate.

7.0 References

CDM Federal Programs Corporation. 2012. *Quality Assurance Manual.* Rev. 13.

American National Standard. 2004. Quality Systems for Environmental Data and Technology Programs – Requirements with Guidance for Use. ANSI/ASQ E4-2004

Figure 1

CDM Smith A subsidiary of Camp Dresser & McKee Inc.

Maintenance and Calibration

Date: Time: (a.m./p.m.)	
Employee Name:	Equipment Description:
Contract/Project:	Equipment ID No.:
Activity:	Equipment Serial No.:

Maintenance

Maintenance Performed:

Comments:

Signature: Date:

Calibration/Field Check

Calibration Standard:	Concentration of Standard:
Lot No. of Calibration Standard:	Expiration Date of Calibration Standard:
Pre-Calibration Reading:	Post-Calibration Reading:
Additional Readings:	Additional Readings:
Additional Readings:	Additional Readings:
Pre-Field Check Reading:	Post-Field Check Reading:

Adjustment(s):

Calibration: □ Passed □ Failed

Comments:

Signature: Date:

Vapor Intrusion Analysis by HAPSITE GC-MS

Project-Specific SOP 6-1
Revision: 0
Date: April 23, 2020

Prepared:	Todd Burgesser	**Technical Review:**	Dung Nguyen
PM Approval:	Nathan Smith	**Editorial Review:**	Melissa Vagi
Project Name:	700 South 1600 East PCE Plume Site, Salt Lake City, Utah		

1.0 Objective

This technical standard operating procedure (SOP) describes the procedures that will be followed to measure volatile organic compounds (cis-1,2-dichlorethene, trichloroethene [TCE], and tetrachloroethene [PCE]) concentration in vapor samples collected as part of the vapor intrusion study and aqueous samples, including groundwater, surface water, and tap water. This procedure will be performed at locations at the 700 South 1600 East PCE Plume Site in Salt Lake City, Utah.

2.0 Background

The Salt Lake City Healthcare System George E. Wahlen Veterans Administration Medical Center (VAMC) in Salt Lake City, Utah, operated a part-time dry cleaning operation that used PCE over a 6-year period in the late 1970s and early 1980s. During this period, dry cleaning residuals were disposed in the sanitary sewer. The PCE Plume was first identified in 1990 during routine monitoring of the Mt. Olivet Cemetery irrigation well. The PCE Plume is in an approximately 300-acre commercial and residential area. PCE levels below the maximum contaminant level of 5 micrograms per liter (µg/L) also were reported in Salt Lake City's secondary drinking water well #18 (closed in 2004). In 2010, PCE was detected up to 40 µg/L in surface water in an area locally identified as the East Side Springs (ESS). In 2018, PCE was detected up to 96 µg/L in shallow groundwater along a transect of monitoring wells at 1400 East in addition to the ESS area. Volatile organic compounds (VOCs), including PCE and TCE, also have been detected in the water emanating from seeps and spring at the site.

2.1 Associated Procedures

- SOP 1-1 Surface Water Sampling, Rev. 10, February 2015
- SOP 1-5 Groundwater Sampling Using Bailers, Rev. 9, February 2015
- SOP 1-9 Tap Water Sampling, Rev. 7, February 2015
- SOP 1-10 Field Measurement of Total Organic Vapors, Rev. 7, February 2015
- SOP 1-12 Low-Stress (Low-Flow) Groundwater Sampling, Rev. 2, February 2015
- SOP 1-14 Lagoon Sampling, Rev. 2, February 2015
- SOP 4-1 Field Logbook Content and Control, Rev. 8, February 2015

3.0 General Responsibilities

HAPSITE Operator – The HAPSITE operator is responsible for receiving proper training in the use of this procedure, the required equipment, and health and safety procedures. The HAPSITE operator must ensure that the quantity and type of quality assurance (QA) samples collected meet the requirements of the work plans.

4.0 Project Planning

This section provides a list of general equipment used during vapor analysis, HAPSITE operations, and health and safety procedures.

4.1 General Equipment

- INFILCON HAPSITE GC/MS
- INFILCON Headspace Sampling System
- Supelco SB1, 30m x .32mm id z 1.0µm film column or equivalent
- Syringes (1 mL, 5 mL and 10 mL, 100 mL) Teflon Luer Lock gastight
- Nitrogen Carrier Gas 99.999%, INFILCON 930-430 or standalone cylinder

- Toughbook Computer (provided with the instrument)
- 500 mL Tedlar Bags
- Liquid Standard at 2,000 µg/mL containing cis-1,2-DCE, TCE and PCE
- Mass Calibration Gas: Internal Standard 1, INFILCON 930-431
- 1/8" and 1/4" stainless steel or Teflon tubing
- Internal Standard (aqueous analysis only) INFILCON 071-747 (fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4 all at 250 µg/L and bromopentafluorobenzene at 500 µL)

5.0 Instrument Parameters

The parameters listed are for general information. Individual parameters may be adjusted based on environmental conditions (temperature and humidity) at the analysis locations.

1. GC Conditions:
    a. Column Temperature – 65°C (ramped to 180°C)
    b. Head Pressure – 105 pa
    c. Inlet Temperature – 60°C
    d. Probe Temperature – 40°C
    e. Valve Temperature – 60°C
    f. Run Time – <8 minutes
2. MS Conditions
    a. Scan/Second – 1.04
    b. Getter Pump Temperature – 400 to 480°C
    c. Scan Range – 45 to 300 amu

6.0 Procedure

6.1 Vapor Analysis

Two strategies for the analysis of vapor may be employed: in situ analysis and indirect analysis. In situ analysis involves the direct analysis of the vapor from a specific location within a building (residence, commercial building, or municipal building). Indirect analysis or off-site analysis requires the collection of a vapor sample in a Tedlar bag (500 mL sample size) and analysis of the vapor at a separate location.

1. All samples that will be received in Tedlar bags should be accompanied with a chain of custody or a field sampling form. Samples analyzed in situ will have the location and identifying information documents in the HAPSITE logbook.
2. For samples collected off-site in Tedlar bags, connect the Tedlar bag to the gas chromatography/mass spectrometry (GC-MS) sampling probe and tighten the Swagelok fitting. To ensure a tight fit and seal, pull on the Tedlar bag to make sure it does not freely move.
3. Open the valve on the Tedlar bag one full turn.
4. Log sample information into the computer and the HAPSITE logbook.
5. Ensure the correct and most up-to-date method is selected (e.g., VA_SG_SIM_PPB_20191206)
6. For in situ sample analysis, position the GC-MS sampling probe in a direction and position to ensure representative analysis of the target air space (e.g., breathing zone, crawl space)
7. Press the start button on the sampling probe or select "Start Run" on the computer. The internal pump pulls the sample through the instrument.
8. The sample pump will turn of after the designated sampling time has been achieved. At this point either close the valve on the Tedlar bag and remove it or place the sampling probe on the cart used to transport the instrumentation.

Note: After 60 seconds of purge, the valve is automatically switched to the fill position, which sweeps the sample across a microtrap concentrator. The microtrap concentrator uses a small bed of absorbent material to trap VOCs. The trap is heated, and the trapped VOCs are carried through the GC column and into the MS for identification and quantitation.

6.1 Aqueous Analysis

Analysis of aqueous sample (groundwater, surface water, tap water) requires the addition of the HAPSITE Headspace Sampling System. All aqueous samples will be analyzed at the VAMC campus. Similar to the HAPSITE instrument, the HAPSITE Headspace Sampling System can be operated using a small on-board source of carrier gas (nitrogen) or an external tank of carrier gas. The system includes a four-sample auto sampler.

1. Connect the Headspace Sampling System to the HAPSITE instrument as described in chapter 3 of the Headspace Sampling System Operating Manual.
2. Prepare the collected samples for analysis by quantitatively transferring 20 mL of sample to a 100 mL vial. Transfer of the water into the syringe should be accomplished by removing the barrel of the syringe and pouring the water directly into the syringe while capping the small open end with a gloved finger.
3. Prepare the internal standard for addition by rinsing a 10 µL syringe three times with organic-free water followed by three rinses with the internal standard. Expel the water and waste internal standard into a sealed waste container (labelled 40 mL vial).
4. Add the internal standards to the sample by transferring 1 µL of internal standard into a 10 µL syringe. Inject the internal standard into the sample through the small opening of the 100 mL syringe.
5. Uncap a clean 40 mL sample vial, tilt the vial, and slowly transfer the sample to the vials down the side of the vial. When transferring, avoid aerating the sample.
6. Cap the vial tightly and label the vial with the sample information.
7. Load the sample into a sample well of the Headspace Sampling System and place another clean and empty vial into an adjacent sample well (used for purge).
8. Position the needle above the clean and empty purge vial by pulling down on the needle until it can freely rotate.
9. Gently lower the needle to allow the needle into the purge vial through the cap septum.
10. Close the black cover to prevent heat loss and promote thorough heating of the assembly.
11. Record the time when each sample was inserted into the well to monitor equilibrium time (approximately 20 minutes).
12. From the computer select the desired method (e.g., VA_VOC_PPT_061919)
13. Click the “Run” button on the computer.
14. When the analysis is completed, a “METHOD FINISHED” message will be displayed.
15. Document all results, including internal standard recoveries, in the HAPSITE logbook.

7.0 Data Analysis and Quantitation

Quantitative analysis is performed by integrating the area of the identified quantitation ion. The quantitation ion for each target analyte has been selected to provide interference free quantitation in the presence of known analytes. The concentration of each analyte is calculated using the response factor from the initial calibration for each compound being measured. Typical detection limits for the analytes of interest are listed in the table below.

Table 1: Detection limits for analytes of interest

Analyte	Detection Limit in Vapor (µg/m^3)	Detection Limit in Water (µg/L)
cis-1,2-dichloroethene	1	10
Trichloroethene	1	10
Tetrachloroethene	1	10

8.0 Quality Control

Quality control procedures consisting of tune checks, initial calibration (three-point), calibration verifications, method blanks, and duplicate measurements (field duplicates and measurement duplicates) are discussed below.

8.1 Initial Calibration

The initial calibration will contain a minimum of three standards with varying concentrations of the analytes of interest listed in Table 1. The lowest concentration standard will be 1 µg/m^3 for vapor and 10 µg/L for water, which is equal to the detection limit. The highest concentration should encompass the linear range of the instrument or the highest concentration of the samples expected. Acceptance criteria for the initial calibration are 25% relative standard deviation of curve fit (%RSD). For vapor analysis the standards

Corrective action for the initial calibration is to investigate the outlying standard and reanalyze. If the problem is not corrected, it may be necessary to remake the standard or correct the problem with the instrument and reanalyze all standards.

8.2 Tune Checks

The tune verification should meet manufacturer specifications and should be repeated every 12 hours before the analysis of any samples. The tune is performed automatically through the system software, and tune failure prevents further analysis until the tune criteria are achieved per manufacturer recommended guidance (HAPSITE Smart Plus Operating Manual INFICON 2008). The tune verification will be repeated each time the instrument is moved or powered down for any reason. Corrective action for the tune verification is to reanalyze the tune. If it continues to not meet criteria, then the tune will be adjusted and saved manually.

8.3 Calibration Verification

A mid-level calibration standard will be analyzed at the beginning of the day prior to analyzing samples. Recovery should be ± 30% of initial calibration.

8.4 Method Blank

The method blank will be analyzed prior to analyzing samples. For aqueous samples, transfer 20 mL of organic-free water to the 100 mL syringe and follow the steps outlined in section 6.2. For vapor samples, fill a Tedlar bag with nitrogen gas and follow the steps outlined in section 6.1. The method blank should be analyzed after the highest calibration standard and after any sample with elevated detections of target compounds. The blank acceptance criteria are that no compounds are detected above the reporting limit. Corrective action for the method blank is to reanalyze. If continuing detections in the method blank occur, clean the system and reanalyze the blank.

8.5 Duplicates

Duplicates should be analyzed on a frequency of 5% of the total samples when sample volume allows. The samples chosen to be analyzed in duplicate should contain detectable concentrations of analytes of interest if possible. Both measurement duplicates (vapor analyzed from the same Tedlar bag if volume is available) and field duplicates will be measured. The acceptance criteria are 30% absolute relative percent difference (%RPD). Corrective action for the duplicate is to reanalyze the sample. If criteria are still not met, results must be flagged as estimated. The absolute RPD is calculated using the following formula:

$$\%\boldsymbol{RPD} = \boldsymbol{abs}\left[\frac{(\boldsymbol{A}-\boldsymbol{B})}{(\boldsymbol{A}+\boldsymbol{B})\ \div\ \mathbf{2}}\right] \times \mathbf{100}$$

Where: A = Original sample result
B = Duplicate sample result

9.0 Health and Safety

When handling any samples, standards, or chemicals the technician should follow standard health and safety procedures, including the donning of nitrile gloves and the proper use of eye protection. Thoroughly wash hands and any exposed skin prior to and after the days work. All liquid and solid waste should be disposed of at the water management area located at the Conex staging area.

10.0 Documentation

Bound field logbooks shall be used for the maintenance of HAPSITE records. All aspects of sample preparation, such as sample observations, deviations to procedures, laboratory duplicate precision, decontamination, and calibrations, will be documented in the laboratory logbooks. The logs documenting the analysis sequence will be recorded daily.

All entries in laboratory logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's activities. Corrections to logbook entries will be accomplished by a single cross out with the date and initials of the person making the entry. Wite-Out or correction fluid or tape is not permitted.

KD ANALYTICAL by FEDERAL RESOURCES

KD ANALYTICAL by FEDERAL RESOURCES

Certificate of Completion

THIS ACKNOWLEDGES THAT

Todd Burgesser

HAS SUCCESSFULLY COMPLETED
HAPSITE BASIC & ADVANCED OPERATOR TRAINING

DAN SCHENK, TRAINER

NOVEMBER 21, 2017

Low-Stress (Low-Flow) Groundwater Sampling

Project-Specific SOP 6-2
Revision: 0
Date: June 5, 2020

Prepared:	Karla Leslie	**Technical Review:**	Robert Alexander
PM Approval:	Nathan Smith	**Editorial Review:**	Terry Crowell
Project Name:	700 South 1600 East PCE Plume Site, Salt Lake City, Utah		

1.0 Objective

This technical standard operating procedure (SOP) describes the procedures that will be followed to collect groundwater samples using a low-flow (minimal drawdown) approach at the 700 South 1600 East PCE Plume Site in Salt Lake City, Utah.

2.0 Background

2.1 Site History

The Salt Lake City Healthcare System George E. Wahlen Veterans Administration Medical Center in Salt Lake City, Utah, operated a part-time dry cleaning operation that used tetrachloroethene (PCE) over a 6-year period in the late 1970s and early 1980s. During this period, dry cleaning residuals were disposed of in the sanitary sewer. The PCE plume was first identified in 1990 during routine monitoring of the Mount Olivet Cemetery irrigation well. The PCE plume is in an approximately 300-acre commercial and residential area. PCE levels below the maximum contaminant level of 5 micrograms per liter (µg/L) were also reported in Salt Lake City's secondary drinking water well #18 (closed in 2004). In 2010, PCE was detected at levels up to 40 µg/L in surface water in an area locally identified as the East Side Springs (ESS). In 2018, PCE was detected at levels up to 96 µg/L in shallow groundwater along a transect of monitoring wells at 1400 East, in addition to the ESS area. Volatile organic compounds (VOCs), including PCE and trichloroethene, have also been detected in the water emanating from seeps and spring at the site.

2.2 Procedure Background

Low-flow groundwater sampling is a method of collecting samples from a well that unlike traditional purging methods does not require the removal of large volumes of water from the well. The objective of low-flow groundwater sampling is to collect samples with minimal alterations to water chemistry through pumping the well at a rate low enough to minimize drawdown and avoid disturbance in the well. Water level drawdown provides the best indication of the stress imparted by a given flow-rate for a given hydrological situation.

Minimal drawdown must be stabilized such that the water to be sampled is representative of the formation surrounding the screened interval and is not from the stagnant water column above the screened interval. Minimal drawdown is achieved, to the extent practical, taking site sampling objectives into account. Typically, flow rates on the order of 50 to 500 milliliters per minute (mL/min) are used. Dedicated bladder pumps or zone isolation sampling technology (ZIST) systems are installed in all wells at the site, except for two artesian wells. The bladder pumps are installed at appropriate depths to ensure pumps remain submerged during sampling, typically at the approximate midpoint of the screened interval, or at a depth interval where lithologic logs indicate groundwater-bearing units are present. The ZIST pumps are installed in the pump receiver at the top of the screened interval provided they have at least 10 feet of submergence, otherwise they are installed without a pump receiver in the well screen like bladder pumps. Minimum purge volume, calculated based on the volume in the sampling system (tubing and pump), has been calculated for each well and is provided to the field sampling team, along with pump depth, screened interval, and previous flow rate and sampling conditions. During pump start-up,

drawdown may exceed the 0.3 feet target and then recover somewhat as pump flow adjustments are made. If the drawdown exceeds 0.3 feet and stabilizes, a new minimum purge volume is calculated, which includes the volume of water in the pump's tubing and the volume of water in the excess drawdown.

From the time the pump starts purging until samples are collected, the purged water is discharged into a graduated bucket to determine the total volume of groundwater purged. Once the minimum purge volume has been met, it is recorded on the purge form. Flow rate is recorded using a separate, smaller graduated cup and recorded in mL/min on the purge form every 5 minutes. The water level is also recorded on the purge form every 5 minutes. The flow rate should match that used during the previous sampling event(s) and should minimize water level drawdown.

A multiparameter instrument capable of measuring pH, oxidation-reduction potential (ORP), dissolved oxygen (DO), specific conductance, and temperature, coupled with a flow-through cell and separate turbidimeter, is used to monitor water quality parameters during purging, which are recorded every 5 minutes. The flow rate must be able to "turn over" at least one flow-through-cell volume between measurements (i.e., a 250-milliliter flow-through cell with a flow rate of 50 mL/min should be monitored every 5 minutes). Stabilization is achieved after all parameters fall within established limits for three successive readings, as discussed in Section 5.

2.3 Associated Procedures

- CDM Smith SOP 1-2, Sample Custody Rev. 8, February 2015
- CDM Smith SOP 1-6, Water Level Measurement Rev. 9, February 2015
- CDM Smith SOP 1-10, Field Measurement of Total Organic Vapors, Rev. 7, February 2015
- CDM Smith SOP 2-1, Packaging and Shipping Environmental Samples, Rev. 6, February 2015
- CDM Smith SOP 2-2, Guide to Handling Investigation-Derived Waste, Rev. 8, February 2015
- CDM Smith SOP 4-1, Field Logbook Content and Control, Rev. 8, February 2015
- CDM Smith SOP 4-2, Photographic Documentation of Field Activities, Rev. 9, February 2015
- CDM Smith SOP 4-5, Field Equipment Decontamination at Nonradioactive Sites, Rev. 10, February 2015
- CDM Smith SOP 5-1, Control of Measurement and Test Equipment, Rev. 10, February 2015

3.0 General Responsibilities

Project Manager (PM) and Primary Technical Lead (PTL) – The PM and PTL are responsible for ensuring that field personnel are trained in the use of this procedure and for verifying that groundwater sampling is carried out in accordance with this procedure.

Field Team Leader (FTL) – The FTL is responsible for complying with this procedure.

4.0 Required Equipment

- Pumps (dedicated bladder pumps and ZIST systems)
- MP-50 compressor/controller
- ZIST controller
- MP-10(H)/Solinst controller
- Compressed gas
- Pump tubing (airline/polyethylene and waterline/Teflon-lined polyethylene)
- Power source (batteries and/or field vehicles)
- Electronic water level meter (according to CDM Smith SOP 1-6)
- Volume measuring device (e.g., graduated cylinder or measuring cup)
- Personal protective equipment, as specified in the health and safety plan
- Sample containers, including packaging supplies and associated paperwork (according to CDM Smith SOP 2-1)
- Decontamination supplies (according to CDM Smith SOP 4-5)
- Turbidity meter

- Standards for calibration and field checking, as needed, of water quality and turbidity meters
- Water quality meter (e.g., YSI 600 Series) with a closed flow-through cell for continuous in-line measurement of temperature, pH, specific conductance, ORP, and DO
- Photoionization detector (PID), as specified in the health and safety plan
- Barometer for calibrating water quality meter DO measurements

5.0 Procedure

The following steps must be followed for low-flow groundwater sampling activities:

1. Review the site-specific health and safety plan, and site-specific project and sampling plans (including previous sampling conditions and well completion details) before initiating sampling activities.

2. Prior to sampling, calibrate all sampling devices and monitoring equipment according to manufacturer's recommendations and the site-specific sampling plan. Calibration of pH should be performed using at least two buffers that bracket the expected pH range. Dissolved oxygen (DO) calibrations should be corrected for local barometric pressure readings and altitude. Calibration is conducted each day and recorded on the calibration log.

3. Put on personal protective clothing and equipment as specified in the site-specific health and safety plan.

4. Open the well cover at each artesian well and monitor the air space at the wellhead for VOCs using a PID immediately upon removal of the well cover. Remove the K-packer from all artesian wells at the site and install the dedicated threaded well cap with gauge, valve, and tubing.

5. Collect synoptic water levels for all wells according to SOP 1-6 Water Level Measurement. All water level measurements must be completed within a 24 hour period. Open the well cover and check condition of the wellhead, including the condition of the surveyed reference mark. Monitor the air space at the wellhead for VOCs using a PID immediately upon removal of the well cap and according to health and safety requirements. Take precautions to minimize disturbance of the stagnant water column above the screened interval during water level measurements. Well depth should be obtained from review of the well completion logs or from previous work. Insertion of a water level measuring device to the bottom of the well casing will result in resuspension of settled solids from the formation surrounding the screened interval, thus requiring longer purging times for turbidity equilibration.

6. Record the pressure reading on the gauge at each artesian well (in pounds per square inch [psi]). The pressure reading will be converted to head as follows:

$$h = 2.31 \times p$$

where,

h = head (feet)
p = pressure (psi)

7. Dedicated sampling devices (either bladder pumps or ZIST systems) are installed in all wells at the site. The bladder pumps are installed at appropriate depths to ensure pumps remain submerged during sampling, typically at the approximate midpoint of the screened interval or at a depth interval where lithologic logs indicate groundwater-bearing units are present. Shallow wells are sampled with the MP-50 controller/compressor or equivalent, deep wells are sampled with the MP-10(H), or equivalent, controller/compressed gas, and the ZIST systems are sampled with the ZIST controller/compressed gas.

8. To achieve low-flow purging conditions, the purge rate should generally not exceed 500 mL/min. Adjust the pump control to stabilize the flow rate and therefore minimize drawdown (less than 0.3 feet during purging activities). The

water level in the well should be measured throughout the purging process to monitor drawdown. Flow rate can be measured from the discharge tube using a volumetric measuring device (e.g., a graduated cylinder or measuring cup) and a watch. Determine the flow rate by measuring volume in 0.5-minute or 1-minute increments. Try to match the flow rates from previous sampling events, using previous controller settings.

9. Record water level measurements and field parameters, including pH, temperature, specific conductance, ORP, DO, turbidity, and flow rate every 3 to 5 minutes during the purging process. Record all measurements and observations in the logbook or on a Groundwater Purging and Sampling Form (Attachment 1). Purging shall continue until the field parameters have stabilized. Parameters are considered stable when three consecutive readings are within the limits of the criteria defined in Table 5.1.

Table 5.1 Stabilization of Water Quality Parameters

Parameter	Units	Stabilization Criteria
Water Level	feet (ft)	less than 0.3 ft
Temperature	degrees Celsius (°C)	±1°C
pH	standard units (su)	±0.1 su
Specific Conductivity	milliSiemens per centimeter (mS/cm)	±3 percent (%)
ORP	millivolts (mV)	±10 mV
DO	milligrams per liter (mg/L)	±10% or ±0.2 mg/L (whichever is greater)
Turbidity	nephelometric turbidity units (NTU)*	greater than 50 NTU AND ±10% OR <10 NTU
Flow Rate	milliliters per minute (mL/min)	50 to 500 mL/min

*****High turbidity is measured in attenuation units (AU); low levels of turbidity, appropriate for stabilization evaluation, are measured in NTU. If AU turbidity is measured, purging will continue until turbidity decreases and the NTU criteria is reached.

10. From the time the pump starts purging until samples are collected, the purged water is discharged into a graduated bucket to determine total volume of groundwater purged. Minimum purge volume, which is three times the volume in the sampling system (tubing and pump), has been calculated for each well and is provided on the purge forms (Attachment A). If drawdown stabilizes at a level greater than 0.3 feet, a new minimum purge volume must be calculated to account for the volume of the drawdown. Volume (of either water in the tubing or water in the well) is calculated as follows:

$$V = \pi r^2 h$$

where,

$\pi = 3.1416$
r = radius (inches)
h = height (inches)
V = volume (cubic inches) (1 gallon contains 231 cubic inches)

11. Wells at the site with low recharge rates can be purged and sampled at the minimum flow rate of 50 mL/min. If the drawdown at the minimum flow rate does not stabilize, a new minimum purge volume must be calculated that includes the volume of water in the drawdown.

12. Artesian wells should be sampled with the valve fully opened to minimize restriction of groundwater flow, even if the free-flowing flow rate of the artesian wells is greater than 500 mL/min. Water levels (recording drawdown) will not be collected during purging and sampling.

13. If either artesian well is found to not be free-flowing at the time of the sampling event (as revealed by the lack of pressure after the installation of the gauge and well cap), a nondedicated portable bladder pump will be rented and

installed at the midpoint of the screened interval. Low-flow groundwater sampling will then proceed as described in Section 5.0.

14. After field parameters have stabilized and minimum purge volume has been met, disconnect the flow-through cell and collect groundwater samples directly from the discharge tubing into an appropriate sample container. During sample collection, maintain the pump rate at the same rate used during purging.

15. If after 2 hours stabilization cannot be achieved, the field teams should note observations on the purge form and inform the field team leader (FTL). The FTL will either approve sample collection, continue purging, or discontinue purging without the collection of samples, and record the full explanation for the decision in the field logbook.

16. Groundwater sampling (including the collection of all required quality assurance/quality control samples specified in the sampling plan) shall be performed immediately upon completion of purging. Generally, volatile (e.g., VOCs) and gas-sensitive (e.g., Fe^{2+}, CH_4, H_2S/HS^-, alkalinity) analytes should be sampled first. The sequence in which samples are collected for inorganic parameters in immaterial.

17. Place all samples in a cooler with ice or ice packs to comply with project, laboratory, and/or regulatory requirements.

18. After sampling activities have been completed, replace and secure the well plug and well cover. Clean up the work area and containerize purge water.

6.0 Restrictions/Limitations

Only grounded electrical devices should be used for low-flow sampling activities. If a gasoline-powered electrical source is used, place portable power sources (e.g., generators or field team vehicles) 50 feet (15 meters) or farther from the wellhead or turn off the source during sample collection to prevent potential contamination of samples. Additionally, it should be clearly noted in the field notes or on the Groundwater Sampling Log (Attachment 1) if water quality parameters did not achieve stabilization prior to sample collection.

7.0 References

ASTM D6452-99. 2012. *Standard Guide for Purging Methods for Wells Used for Groundwater Quality Investigations*. ASTM International, West Conshohocken, PA.

ASTM D4448-01. 2013. *Standard Guide for Sampling Ground-Water Monitoring Wells*. ASTM International, West Conshohocken, PA.

U.S. Environmental Protection Agency. 2017. *Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells*. Quality Assurance Unit.

U.S. Environmental Protection Agency. 2002. *Groundwater Sampling Guidelines for Superfund and RCRA Project Managers*. Ground Water Forum Issue Paper, U.S. Environmental Protection Agency Technology Innovative Office, Office of Solid Waste and Emergency Response, Publication EPA 542-S-02-001, Washington, DC.

U.S. Environmental Protection Agency. 1996. *Low-Flow (Minimal Drawdown) Ground Water Sampling Procedures*. Ground Water Issue, U.S. Environmental Protection Agency Publication EPA/540/S-95/504, Washington, DC.

Iron, Ferrous

DOC316.53.01049

1,10-Phenanthroline Method[1]

Method 8146

0.02 to 3.00 mg/L Fe^{2+}

Powder Pillows

Scope and application: For water, wastewater, seawater, brine solutions, produced waters and hydraulic fracturing waters.

[1] Adapted from Standard Methods for the Examination of Water and Wastewater, 15th ed. 201 (1980).

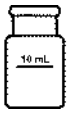
Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR6000 DR3800 DR2800 DR2700 DR1900	The fill line is to the right.	2495402 
DR5000 DR3900	The fill line is toward the user.	
DR900	The orientation mark is toward the user.	2401906 

Before starting

Samples must be analyzed immediately after collection and cannot be preserved for later analysis.
Install the instrument cap on the DR900 cell holder before ZERO or READ is pushed.
For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

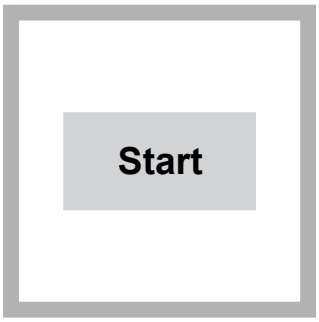
Description	Quantity
Ferrous Iron Reagent Powder Pillows, 25 mL	1
Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to Consumables and replacement items on page 4 for order information.

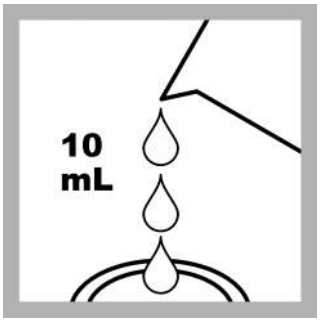
Sample collection

- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Collect samples in clean glass or plastic bottles with tight-fitting caps. Completely fill the bottle and immediately tighten the cap.
- Prevent agitation of the sample and exposure to air.

Test procedure



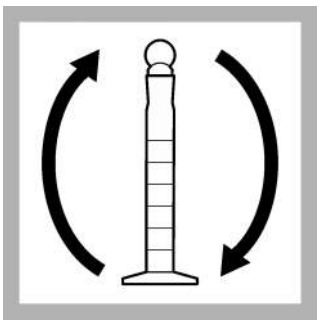
1. Start program **255 Iron, Ferrous**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



2. Prepare the blank: Fill the sample cell with 10 mL of sample.

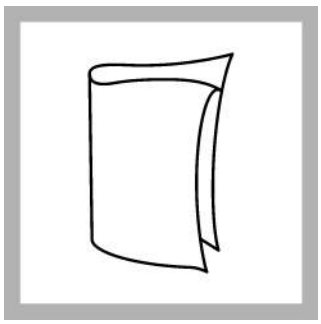
3. Prepare the sample: Fill a mixing cylinder to the 25-mL line with sample.

4. Add the contents of one Ferrous Iron Reagent Powder Pillow to the mixing cylinder.
An orange color shows if ferrous iron is present in the sample.



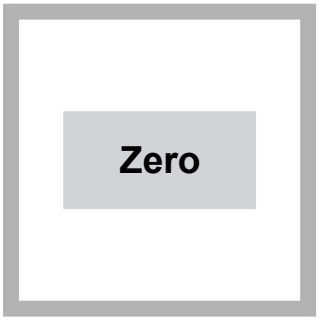
5. Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to mix.
Undissolved powder does not affect accuracy.

6. Start the instrument timer. A 3-minute reaction time starts.

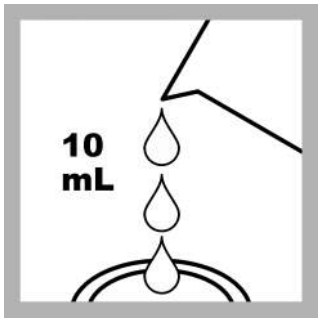


7. When the timer expires, clean the blank sample cell.

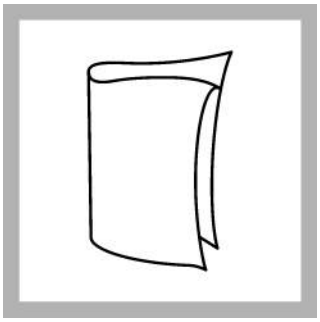
8. Insert the blank into the cell holder.



9. Push **ZERO**. The display shows 0.00 mg/L Fe^{2+}.



10. Fill a second sample cell with 10 mL of the reacted prepared sample.



11. Clean the prepared sample cell.

12. Insert the prepared sample into the cell holder.

13. Push **READ**. Results show in mg/L Fe^{2+}.

Accuracy check

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Ferrous Ammonium Sulfate, hexahydrate
- 1-L volumetric flask, Class A
- 100-mL volumetric flask, Class A
- 2-mL volumetric pipet, Class A and pipet filler
- Deionized water

1. Prepare a 100-mg/L Fe^{2+} ferrous iron stock solution as follows:
   a. Add 0.7022 g of ferrous ammonium sulfate, hexahydrate into a 1-L volumetric flask.
   b. Dilute to the mark with deionized water. Mix well.
2. Prepare a 2-mg/L ferrous iron standard solution as follows:
   a. Use a pipet to add 2.00 mL of the 100-mg/L Fe^{2+} ferrous iron stock solution into a 100-mL volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare the standard solution immediately before use.
3. Use the test procedure to measure the concentration of the prepared standard solution.
4. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard calibration adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
255	2.00 mg/L Fe^{2+}	1.99–2.01 mg/L Fe^{2+}	0.021 mg/L Fe^{2+}

Summary of method

The 1,10-phenanthroline indicator in the Ferrous Iron Reagent reacts with ferrous iron (Fe^{2+}) in the sample to form an orange color in proportion to the iron concentration. Ferric iron (Fe^{3+}) does not react. The ferric iron concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test. The measurement wavelength is 510 nm for spectrophotometers or 520 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Ferrous Iron Reagent Powder Pillow, 25 mL	1	100/pkg	103769

Recommended standards and apparatus

Description	Unit	Item no.
Balance, analytical, 80 g x 0.1 mg 100–240 VAC	each	2936701
Ferrous Ammonium Sulfate, hexahydrate, ACS	113 g	1125614
Flask, volumetric, Class A, 1000 mL glass	each	1457453
Pipet filler, safety bulb	each	1465100
Pipet, volumetric, Class A, 1.00 mL	each	1451535
Water, deionized	4 L	27256
Wipes, disposable	280/pkg	2097000

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

08/2019, Edition 11

how to use *radiello*

before sampling

assembling the supporting plate

Before using ***radiello***, you have to assemble the supporting plate with the clip, necessary to suspend it, and the adhesive label pocket.

insert the clip strip in the slot, with the peg facing upwards

ply the strip and insert the peg into the hole

peel off the transparent pocket

user tip

Assemble the supporting plate in your laboratory before the sampling campaign to save time in the field

and place it onto the plate in a central position; if you prefer, the pocket can be applied to the rear of the plate, but **BE CAREFUL**, always with the label insertion slot on the side (otherwise, if it starts raining the label can get wet)

on-field
to start the sampling

open the plastic bag, draw the cartridge out from the tube and put it in the diffusive body. ***Keep the glass or the plastic tube and stopper in the original plastic bag.***

The lower part of the diffusive body holds a seat for the central positioning of the cartridge. **A correctly centered cartridge should not stick out even by half a millimeter. If it does, the cartridge is not correctly positioned and out of axis.**
BE CAREFUL: do not hold the diffusive body horizontally when you screw it onto the plate, otherwise the cartridge could come out from its seat and stick out.
As a consequence, when the diffusive body is screwed onto the supporting plate the cartridge is bent, the geometry of the sampler is disturbed and the results obtained become unreliable. ***To place the cartridge centrally you need only to tap on the diffusive body.***

Insert a label in the pocket without peeling it off. Keep note of the date and time and expose ***radiello***. Sampling has started.

Keeping the diffusive body in a vertical position, to screw it onto the support plate

user tip

Do not touch the cartridge with your fingers if possible, particularly if it is impregnated with reactive

radiello is patented by
FONDAZIONE SALVATORE MAUGERI-IRCCS
Centro di Ricerche Ambientali - via Svizzera, 16 - 35127 PADOVA

595 North Harrison Road,
Bellefonte, PA 16823 USA
www.sigma-aldrich.com/radiello

SUPELCO

user tip

even if you can write date and time of the sampling start and end on the adhesive label, we suggest you to keep note of these parameters also separately: after a week exposure with bad weather conditions,

your writing might have become illegible!

DO NOT USE MARKER PENS to write on the label: they contain solvents that are sampled by ***radiello***!

after the sampling

Keep note of the date and time of the end of exposure.

Place the cartridge into the tube, peel off the label and stick it onto the tube **such that the barcode is parallel to the axis of the tube.**

If you have performed the sampling of different polluting compounds at the same time, ***BE CAREFUL NOT TO MIX UP THE TUBES***: place the exposed cartridge in its original tube, identified by the code printed on the plastic bag.

IMPORTANT

Always stick the label such that the barcode is parallel to the axis of the tube: any other position will compromise the barcode automated reading by the optic reading device.

radiello maintenance

When exposed outdoors or in a workplace environment, the diffusive body may get dirty from airborne dust. Fine particles (PM_{10}) are especially harmful to yellow diffusive bodies since they can obstruct the pores. When the diffusive bodies are dirty you can wash them as follows.

Immerse the diffusive bodies in a beaker with a soapy solution (e.g. dish detergent) and sonicate them for 20 minutes. As the diffusive bodies float, you may make them sink by putting a smaller beaker on them, with water inside enough to dip it a few centimeters. Rinse the diffusive bodies with plenty of water and then deionized water; let them finally dry in the air.

IMPORTANT: NEVER USE SOLVENTS TO CLEAN THE DIFFUSIVE BODIES!!!

After four or five washings, diffusive bodies need to be replaced: repeatedly adsorbed dust may have penetrated the so deeply that they cannot be removed by washing anymore.

The following table shows the advised washing schedule:

PM_{10} concentration ($\mu g \cdot m^{-3}$)	<30	40	>50
Washing after days of exposure	45	30	15

Method Procedures Manual

Field-Based Analysis of Chlorinated Volatile Organic Halocarbons

- **AQR Color-Tec combines sample purging with direct-read gas detector tubes to quickly detect low-levels of chlorinated compounds in liquid and solid samples.**
- **AQR Color-Tec detects concentrations of total chlorinated volatile organic halocarbons (CVOHs) below 3 µg/L in water and 3 µg/Kg in soil samples.**
- **AQR Color-Tec provides fast, low-level, economical, decision-quality data which maximizes sampling frequency and sampling coverage to locate source areas and delineate dissolved-phase contaminant plumes.**
- **Samples are analyzed by purging the volatile compounds from either liquid or solid samples through a colorimetric detector tube, which produces a distinct color change when exposed to any chlorinated compound.**

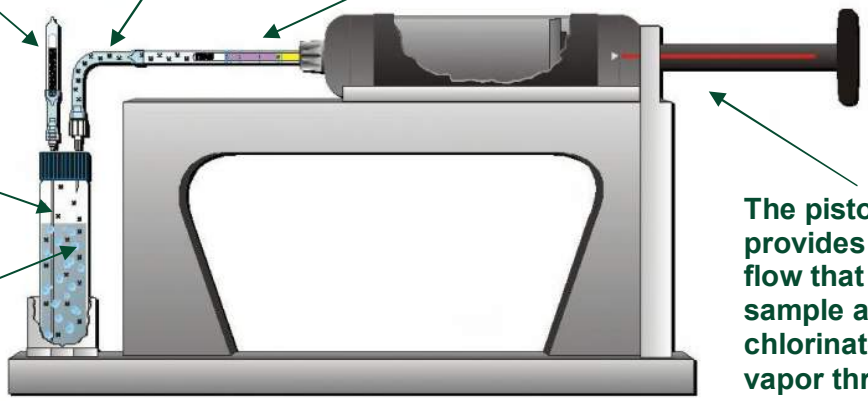
2 Ambient air enters the sample through a carbon filter

3 Filtered ambient air flows through the long purge needle to the bottom of the VOA vial

4 Ambient air purges and strips volatile compounds from the sample

5 The purged chlorinated volatile compounds are directed into the gas detector tube

6 A measured color change in the colorimetric tube is used to estimate the expected concentration of CVOHs in the sample



1 The piston-pump provides vacuum air flow that purges the sample and pulls the chlorinated volatile vapor through the tube

AQR Color-Tec® Contact and Ordering Information

- **For more information visit www.aqrcolor-tec.com**
- **For kit orders contact Phil Pecevich at 919-918-7191**

Table of Contents

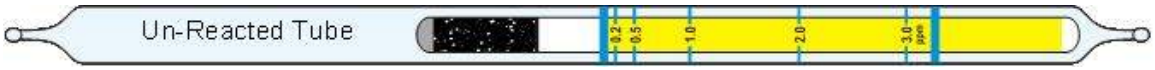
Section			Page
1.0	Method History and Principles		2
2.0	Color-Tec Test Kit Parts Description and Set-Up		3
	2.1	Materials Provided	3
		2.1.1 Hardware Kit	3
		2.1.2 Expendables Kit	3
		Figure 1 - Hardware Kit Parts	3
		Figure 2 - 20-Sample Expendables Pack Parts	4
	2.2	Accessories Supplied by User	4
	2.3	Storage & Stability of Colorimetric Tubes	4
	2.4	Heating Colorimetric Tubes and Samples	4-5
	2.5	Carbon Pre-Filter	5
	2.6	Color-Tec Workstation Set-Up	5-6
3.0	Sample Collection and Preparation		7
	3.1	Liquid Sample Media	7
	3.2	Solid Sample Media	7
	3.3	Purpose of the Second VOA Vial	7
4.0	Sample Analysis Procedure		8-9
		Troubleshooting Guide	10
5.0	Sample Purging and Detection Methodology		11
	5.1	50cc Purge	11
	5.2	100cc Purge	11
	5.3	200cc Purge	11
		Table 1 - Purge Volume Correction Factors	12
6.0	Reading the Tubes		12
	6.1	Very Low Concentrations	12
	6.2	Low to Medium Concentrations	12
	6.3	High Concentrations	13
	6.4	Recording Tube Readings	13
7.0	Estimating Sample Concentrations (Conversion Table)		13
		Table 2 – Conversion Table	14
8.0	Proposed QA/QC Procedures		15
	8.1	Analytical Confidence and Method Performance	15
	8.2	Chemical Inhibitors (False Negatives)	15
	8.3	Positive Interference (False Positives)	15-16
		Chlorinated Volatile Organic Halocarbons	16
		Water Vapor	16
		Hydrogen Chloride Vapor	16
		Free Chlorine	16
		Contaminant Carryover	16
	8.4	Ambient Air Interference	16
	8.5	Duplicate Sample Testing Procedure	17
	8.6	Collection of Split Samples for Laboratory Analysis	17
9.0	Safety Precautions and Disposal of Expendable Materials		17-18
	Product Warranty		18
EPA Guidance and Case Studies Featuring the Color-Tec Method			18
Recommended Field Applications for the Color-Tec Method			19

1.0 Method History and Principles

The Color-Tec method was developed during 1997 by the environmental professionals at Ecology and Environment, Inc. while assessing/remediating the earliest sites addressed under the Florida Department of Environmental Protection's (FDEP) Drycleaning Solvent Cleanup Program. Since its development, the method has been used extensively at EPA, DOD, and various state regulatory agency sites to provide real-time, decision quality data at thousands of chlorinated solvent sites.

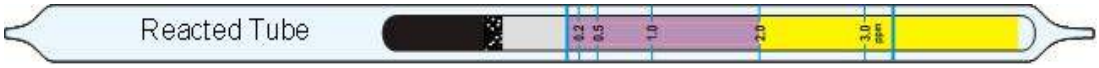
Color-Tec is a field-based analytical method which combines the use of colorimetric gas detector tubes (originally designed for occupational breathing-zone monitoring) with sample purging to detect very low (<3 µg/L or µg/Kg) concentrations of total chlorinated volatile organic halocarbons (CVOHs) in liquid and solid samples. Samples are analyzed by purging the volatile compounds from a groundwater or soil sample directly through the colorimetric tube, which is designed to produce a distinct color change when exposed to chlorinated compounds. Estimated sample concentrations are obtained by comparing the tube readings to a conversion table, which was developed based on comparison of Color-Tec readings to GC/MS analysis of split samples.

Each colorimetric tube contains an oxidizer (PbO_2) and a catalyst (H_2SO_4) which decomposes and converts the chlorinated compounds to hydrogen chloride, which discolors a reagent (4-phenylazodiphenylamine) in the tube from yellow to purple. The reaction formula provided by Gastec® for the PCE tube is as follows:



$Cl_2C{:}CCl_2 + PbO_2 + H_2SO_4 \rightarrow HCl$

HCl + (4-Phenolazo) Diphenylamine $\rightarrow$ Chloride



The colorimetric tubes react positively to all chlorinated volatile organic halocarbons, including saturated and unsaturated chlorinated alkenes and alkanes. The total response indicated by the detector tube (the distance that the color change travels through the tube) reflects the sum of the concentration of each individual chlorinated compound present in the sample. The method is primarily qualitative (detects the presence/absence of a compound or class of compounds).

The colorimetric gas detector tubes used in the method are designed to detect CVOHs in ambient air. **Color-Tec is an alternate use of these tubes**, which purges CVOHs from a water or soil sample and concentrates them into the colorimetric tube. When using colorimetric tubes for the Color-Tec method, the units (ppmV) printed on the tubes do not directly reflect the quantity of CVOHs present in the water or soil sample being analyzed. The Color-Tec tube reading (the distance that the color change travels through the tube) is a **relative response** to the amount of chlorinated-compound molecules that have been purged from the sample and directed into the tube. Therefore, the Color-Tec tube reading is a **unit-less** value used only to record the **relative response** for each analysis in order to facilitate comparison of that response to laboratory GC/MS analysis.

THE COLOR-TEC TUBE READING IS NOT THE SAMPLE CONCENTRATION!

The tube reading is a unit-less value which must be compared to laboratory results from split samples in order to yield an estimate of the actual concentration present in the sample.

This manual provides a conversion table, developed using comparison of Color-Tec tube responses to split-sample GC/MS analyses conducted on thousands of samples, which can be used to provide an estimate of the expected sample concentration based on the tube reading (See Table 2 on page 15).

2.0 AQR Color-Tec Test Kit Description and Set-up

The Color-Tec Chlorinated VOH Soil/Water Test Kit System consists of two primary components:

1. A hardware kit which contains all **reusable equipment** needed to conduct the method, plus a carrying case; and
2. Expendables kits containing all **disposable components** needed for analysis of 20 water or soil samples using the following ranges of Gastec® 133-series tubes:
   a. Ultra low range 133-LL tubes (expected detection range ~ 3 to 1200 µg/L or µg/Kg)
   b. Low range 133-L tubes (expected detection range ~ 75 to 25,000 µg/L or µg/Kg)
   c. Medium range 133-M tubes (expected detection range ~ 500 to 130,000 µg/L or µg/Kg)

2.1 Materials Provided

2.1.1 Color-Tec Hardware KIT (See Figure 1)

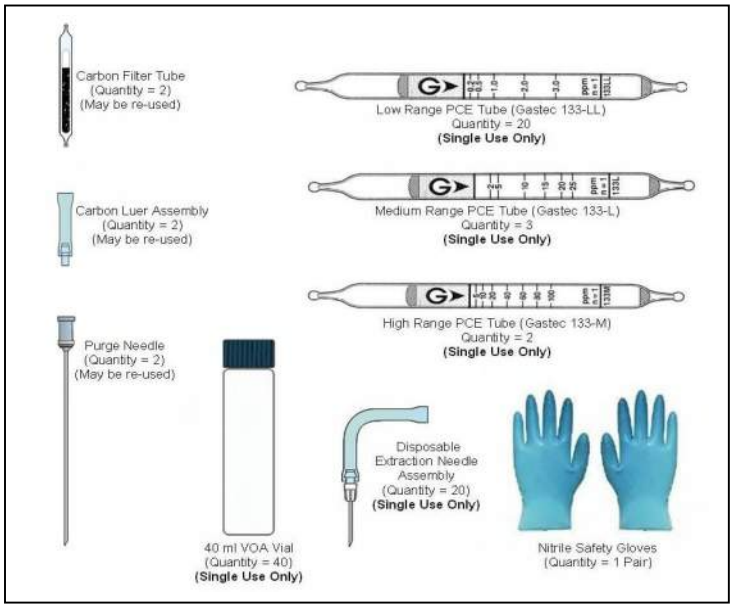
Item	Quantity
Piston pump	1
Color-Tec Pump Stand	1
Hot Plate	1
Stainless Steel Heating Pan	1
VOA Heating Rack	1
Thermometer	1
Decontamination Syringe	1
Pelican® hard case	1

2.1.2 Color-Tec 20-Sample Expendables Pack (See Figure 2) (Analyzes 20 samples)

Item	Quantity
Low-Range (133LL) Colorimetric Detector Tubes	20
Medium-Range (133L) Colorimetric Detector Tubes	3
High-Range (133M) Colorimetric Detector Tubes	2
Disposable Extraction Needle Assemblies **(single use only)**	25
40 Milliliter VOA Vials – empty (for samples)	40
Carbon Filter (may be re-used)	2
Carbon Filter Luer Assembly (may be re-used)	2
Purge Needle (may be re-used)	2
Nitrile Safety Gloves (pair)	1

Figure 1
Hardware Kit

Figure 2
Expendables Kit



2.2 Accessories Supplied by User

The following items (not provided in the AQR Color-Tec kit) are suggested for use with the Color-Tec method to perform the listed functions.

Item	Purpose
Organic-free water	for soil sample extraction and equipment decontamination
Safety gloves	personal protection
Safety glasses	personal protection
120V AC power source	for hot plate
Permanent marker	labeling sample bottles

Performance of the Color-Tec method requires the use of two standard, unpreserved VOA vials per sample. These VOA vials are not included in the standard expendables kits, but may be added as an option. The user may wish to collect a quantity of split samples for laboratory analysis to provide comparison data which may be used to determine site-specific method detection limits and/or to tentatively quantify Color-Tec results. Split sampling will likely require three pre-preserved VOA vials per sample. Pre-preserved VOA vials for split samples are not available in the Color-Tec expendables kits.

2.3 Storage & Stability of Colorimetric Tubes

The Gastec® colorimetric tubes have a shelf-life of two years with refrigeration. Tubes should be stored at or below a temperature of 10°C/50°F when not in use. Colorimetric detector tubes are single-use (one tube per analysis) and should be used immediately after the tips are broken. Tube readings should be recorded immediately following analysis because the intensity of the color-change fades over time. Each box of tubes has an expiration date printed in red ink on the top of each box. When heating the tubes for use with the Color-Tec method, it is recommended that the tube temperature does not exceed 40°C/104° F.

Other procedures and guidelines associated with the use of the tubes for their designed purpose (gas detection in ambient air) are included in the tube manufactures data sheets and tube instructions included in the tube packaging.

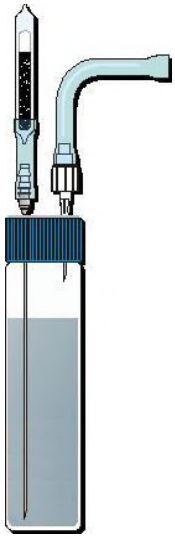
2.4 Heating Colorimetric Tubes and Samples

The colorimetric gas detector tubes used in the Color-Tec method were designed for the purpose of detecting volatile organic compounds (CVOHs) in ambient air. When using the tubes for analysis of ambient air, the calibrated operating temperature is 20°C/68°F. Using the tubes at temperatures above or below 20°C/68°F, for the purpose of testing ambient air, introduces error into the measurements requiring application of correction factors to correct that error. Because Color-Tec is an alternate use of the colorimetric tubes which concentrates CVOHs from water or soil samples into the tubes, the units (ppmV) printed on the tubes have no direct relationship to the quantity of CVOHs dissolved in the water/soil sample being analyzed and the temperature correction factors used for analysis of ambient air are not required when using the colorimetric tubes as part of the Color-Tec method. However, since the colorimetric tubes are more sensitive to the presence of chlorinated compounds at 40°C/104°F, and the purpose of the Color-Tec method is to detect the presence/absence of CVOHs in water at concentrations at the lowest concentrations possible, the tubes are heated to their optimum sensitivity (40°C/104°F) to maximize their detection capability.

The samples are also heated (in the VOA vials) to maximize contaminant volatilization and transfer of CVOHs from the water sample to the colorimetric tube. To heat the samples and colorimetric tubes, a hot plate is used to heat a water bath containing a test tube rack to hold the sample-filled VOA vials and unbroken colorimetric tubes. Special attention must be paid to the temperature of the water to avoid prolonged overheating the samples and tubes. The samples and colorimetric tubes should not be heated in excess of 40°C/104°F.

Given the size of the heating pan and VOA rack, generally only 3 sets of samples are heated at the same time. When a pair of VOAs is removed from the heating rack and placed on the pump stand, it can be replaced with a new pair for heating. After collection, samples should remain in a cool place until ready to be heated and analyzed. It is recommended to avoid heating the samples for more than about 2 minutes to avoid loss of CVOCs. Section 2.6 below, provides detailed water bath set-up and heating procedures.

2.5 Carbon Pre-Filter



Because ambient air is used to purge the samples, a carbon pre-filter is provided for attachment to the purge needle to prevent volatile airborne contaminants from passing through the sample and entering the detector tube during the purging process. To use the carbon pre-filter, break both tips of a carbon filter tube and insert the end of the tube onto the carbon lure assembly (make sure the air-flow arrows on the carbon tube point toward the carbon lure assembly), then tightly insert the male lure fitting on the carbon lure assembly into the female lure fitting on the purge needle (see Figure 2). At sites where little or no ambient air contamination is expected, a single pre-filter tube may be reused for several days. However, at sites where high concentrations of airborne chlorinated compounds are suspected or have been confirmed in the ambient air, the pre-filter tubes may need to be replaced more frequently. For most situations, one carbon filter per 10 samples is more than sufficient. Section 2.6 below, provides detailed carbon filter set-up and use procedures.

2.6 Color-Tec Work-Station Set-up

Pump Stand Set-up

1. Place the pump stand up-right on a flat stable surface.
2. Place the piston-pump into the curved tray on the top of the pump stand as shown.

Corning® Hot Plate Set-up

1. Connect the AC power cord to the back of the hot plate.
2. Connect the other end of the AC power cord to a USA 120VAC electric outlet.
3. Place the hot plate on a flat stable surface.
4. Set the hot plate thermostat control to between dial setting 4 and 5.

Hot Water Bath Set-up

1. Fill the stainless-steel water bath pan with tap water to approximately 1.5-inches from the rim.

2. Insert the VOA rack into the water-filled, stainless-steel, water bath pan.

3. Remove the cap from a 40ml VOA vial, fill the VOA vial with tap water and place it into the VOA Rack as shown. *Note: The bottom of the water-filled VOA vial should be slightly submersed in the water in the stainless-steel pan.*

4. Place the stainless-steel water bath pan onto the heating surface of the hot-plate.

5. Open a box of low-level (133LL) Gastec® tubes and place several tubes into the water-filled VOA vial. Insert the yellow reagent end of the tubes into the bottom of the VOA vial. Note: Do not place tubes with broken tips in the water bath – heating must be accomplished before breaking the tube tips.

6. Turn on the digital thermometer and place the steel probe into the water-filled VOA vial with the colorimetric tubes.

7. Once the water bath reaches a temperature of approximately 100ºF, the colorimetric tubes and VOA vials containing samples can be heated. *Note: The temperature of the water bath should not exceed 100ºF.*

Heating Samples

1. Place both VOA vials containing the sample into the hot water bath for approximately 1 to 2 minutes.

2. Be sure that the VOA vials are tightly sealed before heating. *Note: When properly heated, the VOA vials should feel warm in the hand – DO NOT OPEN VOA VIALS AFTER HEATING.*

Carbon Filter/Purge Needle Set-up

1. Break both ends of a carbon filter tube using the tip breaker on the piston pump.

2. Connect a carbon filter luer assembly to the carbon filter tube by sliding the open end of the vinyl tubing over the broken end of the carbon filter tube. Note: The carbon filter is re-used for multiple purge cycles.

3. Attach the carbon filter assembly to a purge needle by inserting the carbon filter assembly luer fitting into the purge needle luer fitting.

4. The purge needles are re-used after decontamination. Thoroughly clean and rinse the purge needle between each sample analysis to avoid contaminant carryover.

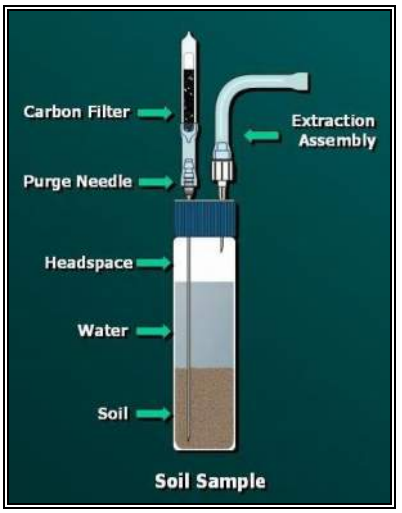
3.0 Sample Collection and Preparation

3.1 Liquid Sample Media

Collect the water or other liquid sample media directly from your sampling device into two 40 ml VOA vials by filling each vial to ~75% capacity (i.e. to about 1-inch below the shoulder of each vial). Tightly secure the caps onto the partially-filled VOA vials. The VOA vials containing the liquid sample to be tested must contain an air-filled headspace to accommodate purging. The caps must be tightened sufficiently to prevent loss of CVOHs during the time between sample collection and analysis (which includes the heating process) and to prevent air leakage during the purging process.

3.2 Solid Sample Media

Place about 1.5 inches of soil (or other solid sample media) into the bottom of each of two VOA vials (i.e. approximately 30 grams in each vial). Immediately after inserting the soil (or other solid sample media) into the two vials, add organic-free or other "clean" water to each VOA vial until they are both ~70 % full (i.e. to approximately 1-inch below the shoulder of each vial). Tightly secure the caps onto the partially-filled VOA vials. Once the caps are secure, shake the VOA vials vigorously for approximately for 5 to 10 seconds to thoroughly mix the soil and water. Additional mixing may be necessary for soil matrices comprised of clay-sized particles. The purpose of the mixing is to transfer any chlorinated compounds suspended in the soil matrix to the water to facilitate more effective purging.



IMPORTANT NOTE: The VOA vials containing the solid sample media and "clean" water must contain an air-filled headspace to accommodate purging. The caps must be tightened sufficiently to prevent loss of CVOHs during the time between sample collection and analysis (which includes the heating process) and to prevent air leakage during the purging process.

3.3 Purpose of the Second VOA Vial

The Color-Tec method is designed for use with two VOA vials (an original and a duplicate) for each sample collected. In certain situations, the duplicate sample may not be used in the performance of the method. However, the duplicate sample should always be collected in the event that it is needed to complete the analysis process. The duplicate sample may be used in either of the following situations:

- When the initial test does not induce a color change in the colorimetric tube, the second VOA vial containing the duplicate sample, may be purged (using the same colorimetric tube) to increase the probability of detecting very low (< 10 µg/L) concentrations.

- When the initial test induces a color change that exceeds the upper limit of the LL tube (a tube reading > 3), the extra VOA vial can be used to analyze the sample using higher range colorimetric tubes (133L or 133M) to tentatively quantify the higher concentration of chlorinated compounds in the sample.

4.0 Sample Analysis Procedure

1. Place both heated VOA vials (original & duplicate sample) into the two VOA holders on the pump-stand.

2. Remove a low-level tube from the hot water bath and wipe it dry.

3. Break both ends of the colorimetric tube using the tip breaker on the piston pump.

4. Insert the colorimetric tube into the pump inlet with the flow arrow (printed on the tube) toward the pump. ***Note:*** *Tube orientation is critical – the yellow reagent end of the tube is inserted in the pump.*

5. Connect a new extraction needle assembly to the colorimetric tube by sliding the open end of the vinyl tubing over the broken end of the colorimetric tube. This step must be completed before inserting the needle into the VOA (Prior to step 6).

6. Remove the protective cap from the extraction needle and insert the needle into the septa of the first VOA vial. ***Note:*** *Be sure that the tip of the extraction needle is positioned within the headspace of the VOA vial (above the water level). Do not insert the extraction needle as far as it will go into the headspace of the VOA vial, but rather only to a point slightly beneath the inside of the septa to reduce the possibility of sample water entering the extraction needle assembly and colorimetric tube during the purging process.*

7. Insert the purge needle (with carbon filter assembly) into the septa of the first VOA vial and push the tip of the needle to the bottom of the VOA vial.
IMPORTANT NOTE: Do not insert the purge needle before completing steps 5 and 6.

8. Align the 50ml label and red dot on the pump handle with the red dot on the pump shaft.

9. Pull the handle sharply until it locks in the 50ml (half pull) position.

10. Confirm that air is purging through the sample in the VOA vial.

11. Purge for approximately 30 seconds.

12. Check the yellow reagent in the tube for a color-change.

13. If no color-change reaction is visible or if the color reading is less than 1.5, rotate the pump handle ½ turn and pull the handle out to lock in the 100ml position.

14. Continue the 100ml purge until the flow cycle is complete. Note: Flow is complete when the end-of-flow indicator (located on the back of the pump handle) returns to its full brightness.

15. Check the yellow reagent phase in the tube for a color change.

16. If no color-change is visible, remove the extraction needle from the VOA with the vinyl tubing still attached to the low-level tube, rotate the pump handle ¼ turn and push the plunger back into the pump, remove the extraction needle from the first VOA vial and inject it into the septa of the second VOA (duplicate sample), then remove the purge needle from the first VOA vial and inject it into the septa of the second VOA (duplicate sample) - now re-pull the pump handle to lock into the 100ml position.

17. When the second 100ml purge cycle is complete, read and record the results.

For samples containing high concentrations (>150 µg/L) the resulting color-change may exceed the calibrated limit of the low-level tube, requiring the second VOA vial (duplicate sample) to be purged and analyzed by repeating steps 3 through 13 using a medium range (133L) or a high range (133M) tube.

For samples containing low (<5 µg/L) concentrations the color change does not usually begin until 100 CCs of air have purged through the sample. Furthermore, the color change induced at these low concentrations is very slight (below 0.5 on the tube scale) and appears as a slight darkening or light purple hue at the entrance of yellow reagent layer in the LL tube. When the sample contains higher concentrations (>10 µg/L) of chlorinated compounds, the resulting color change is an obvious light to dark purple, which propagates through the yellow reagent layer toward the pump end of the colorimetric tube. The tube reading (Color-Tec response) is obtained by matching the linear extent of the discolored reagent inside the tube to the calibration scale printed on the outside of the tube. Table 1 presents a troubleshooting matrix with causes and solutions potential problems.

Important Procedural Notes:

The disposable extraction needle assembly is intended for one use only. Decontamination and re-use of this part is highly discouraged because of the risk of contaminant carryover from the tubing and other plastic parts which can harbor contaminants from the previous analysis. Purge needles may be reused following decontamination using water and isopropanol.

Carbon filters should be discarded if they become wet from contact with sample water.

Never insert the purge needle into the VOA before the extraction needle assembly has first been connected to the colorimetric tube and inserted into the VOA headspace. If the purge needle is inserted first, the pressure inside the sealed VOA may force sample water up though the purge needle and into the carbon filter. Sample volatiles may be lost if the extraction needle assembly is inserted into the VOA headspace before connecting the tubing to the colorimetric tube.

To prevent clogging of the purge needle when inserting the purge needle into VOA vials containing soil samples, do not immediately push the bottom of the needle through the soil to the bottom of the vial; but rather temporarily position the base of the purge needle in the water above the soil until the pump handle has been pulled to begin air flow through the sample. Once air flow has been initiated, slowly extent the purge needle through the soil to the base of the vial. The air flow from the tip of the purge needle should reduce the potential for clogging as the needle moves through the soil. This procedure is especially helpful when working with clayey soils.

Troubleshooting Guide

Problem	Possible Cause	Solution
Sample does not appear to be purging (bubbling) after the pump handle has been pulled.	Clogged/blocked purge (long) needle.	Use the decontamination syringe to check the purge needle for clogs. If clogged, clean the needle or use a new purge needle.
	Clogged/blocked extraction (short) needle.	Use decontamination syringe to check the extraction needle for clogs. Use decontamination syringe to clean the needle or use a new extraction needle.
	Colorimetric tube is not securely connected to hand pump.	Remove and re-insert the colorimetric tube from the hand pump. If the fit seems loose, replace the hand pump inlet gasket.
	Colorimetric tube is not securely connected to extraction needle tubing.	Check the connection between the extraction needle tubing and the colorimetric tube. If loose, insert the colorimetric tube further into the extraction needle tubing.
	VOA cap is not tightly sealed.	Check the tightness of the VOA cap. Tighten if necessary.
	Colorimetric tube tips were not broken before connecting to hand pump and tubing.	Break both tips of the colorimetric tube before connecting to hand pump and tubing.
	Broken/bad plunger seal in hand pump.	Check the pump seal by holding your finger over the hand pump inlet while pulling the pump handle and lock into the 50cc position. If no vacuum is apparent, open the pump, remove the plunger, replace the plunger seal, and grease the new seal. Re-assemble the pump.
The colorimetric tube shows no reaction after purging a sample that contains chlorinated compounds. (False Negative)	Colorimetric tube is below the optimum operating temperature.	Heat the colorimetric tube to 40°C/104° F before using. It is also recommended to heat the sample. The recommended temperature for tubes and samples when using the Color-Tec Method is 40°C/104° F.
	Colorimetric tube was connected using reversed flow direction.	Use the flow direction arrows to properly align the tube. The purged air must pass through the black oxidizer phase and the white catalyst phase before entering the yellow reagent phase.
	The sample also contains a detectable concentration of xylenes or toluene.	Samples can be tested for the presence of xylenes and toluene using the **Gastec® 122L** colorimetric tube. The detection of chlorinated compounds may be diminished when xylenes or toluene are present in a sample.
The colorimetric tube indicates a reaction after purging a sample that contains no chlorinated compounds. (False Positive)	Chlorinated compounds are present at detectable concentrations the ambient air.	Test the ambient air using an LL tube to determine if chlorinated compounds are present at detectable concentrations. Attach the charcoal filter to the purge needle prior to purging samples.
	HCl vapor is present in the sample VOA or in the ambient air.	Avoid use of HCl in the area where Color-Tec is in use. Use only unpreserved VOAs for samples to be screened with Color-Tec.
	Water vapor has entered the yellow reagent phase of the tube indicating a positive reaction	Avoid purging more that 200 CCs through any sample. Stop purging before condensation inside the tube reaches the end of the black oxidizer phase. Avoid drawing any water from the sample VOA into the colorimetric tube.

5.0 Sample Purging and Detection Methodology

Samples may be purged using 50 cubic centimeters (cc), 100cc, or 200cc purge volumes. These various purge volumes are used in succession to maximize the low-level detection capability and detection range of each tube, thereby reducing the number of tubes needed to tentatively quantify the concentration of total chlorinated compounds in the sample. The pump stand is equipped with two VOA-vial holders to accommodate a second (duplicate) sample to be collected from each sampling location. This duplicate sample (collected and prepared in the same manner as the original sample) serves the following two potential purposes:

1. When purging the initial VOA vial does not induce a color change in the colorimetric tube, the second VOA vial containing the duplicate sample, may be purged (using the same colorimetric tube) to increase the probability of detecting very low (< 10 µg/L) concentrations.
2. When the initial test induces a color change that exceeds the upper limit of the LL tube (a tube reading > 3), the extra VOA vial can be used to analyze the sample using higher range colorimetric tubes (133L or 133M) to tentatively quantify the higher concentration of chlorinated compounds in the sample.

5.1 50cc Purge Volume

Initially, all samples are analyzed using a Gastec® 133-LL tube with a 50cc purge cycle. If the 50cc purge induces a color change reading of 1.5 to 3.0, read the calibration scale value aligned with the stained/unstained interface in the tube and use the pump stroke correction factors provided on Table 1 to determine the correct reading for a 50cc purge volume. If the concentration in the sample exceeds the upper detection limit of the tube (i.e. the color change moves beyond the upper limit of the calibration scale printed on the tube), repeat the analysis using duplicate samples and higher range tubes (133-L, 133-M, and 133-HA) until the color change reaction stops within the calibration scale on the tube. If the color change reaction exceeds the upper limit of the calibration scale of the HA tube, the sample contains a concentration of chlorinated compounds above the upper detection capability of the Color-Tec Method.

5.2 100cc Purge Volume

Following completion of the 50cc purge cycle, if the concentration in the sample has induced a color change in the tube which traveled less than half the distance of the calibrated portion of the reagent phase of the tube (less than a reading of approximately 1.5), pull the pump handle outward and lock it into the 100cc position to complete a full purge cycle. Record the value aligned with the stained/unstained interface on the tube. No correction factor is needed for a 100cc purge.

5.3 200cc Purge Volume

Following completion of the 100cc purge cycle, if the concentration in the sample has induced no color change reaction, remove the purge needle and extraction needle assembly from the VOA vial containing the original sample and insert them into the VOA vial containing the duplicate sample (which has also been pre-heating) and perform another 100cc purge cycle **using the same colorimetric tube**. To perform the transfer to the second vial, remove both needles from the original VOA vial and immediately insert both needles into the septa of the duplicate sample VOA vial. Before re-inserting the pump handle, temporarily remove the colorimetric tube from the tip of the hand pump and re-insert the pump handle completely into the pump while the tube is un-attached. Re-attach the colorimetric tube into the pump tip and pull the pump handle and lock it into the 100cc position. Following the complete second purge cycle, read the calibration scale value aligned with the stained/unstained interface in the tube and use the pump stroke correction factors provided on Table 1 to determine the correct reading for a 200cc purge volume.

Table 1
Purge Volume Correction Factors for 133-Series Tubes

Colorimetric Tube	Purge Volume	Quantity of Pump Pulls	Correction Factor
133-LL	50cc	Half Pull	Tube Reading x 3
133-LL	100cc	Full Pull	Tube Reading x 1
133-LL	200cc	Two Pulls	Tube Reading x 0.5
133-L	50cc	Half Pull	Tube Reading x 3
133-L	100cc	Full Pull	Tube Reading x 1
133-L	200cc	Two Pulls	Tube Reading x 0.5
133-M	50cc	Half Pull	Tube Reading x 2.5
133-M	100cc	Full Pull	Tube Reading x 1
133-M	200cc	Two Pulls	Tube Reading x 0.4
133-HA	50cc	Half Pull	Tube Reading x 3
133-HA	100cc	Full Pull	Tube Reading x 1
133-HA	200cc	Two Pulls	Tube Reading x 0.3∞

6.0 Reading the Tubes

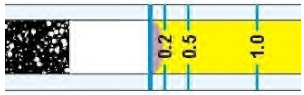
The basic Color-Tec method procedures are simple and intuitive; however, contaminant detection and semi-quantitative values are obtained through visual observation of the colorimetric reaction in the tubes, which is inherently subjective (especially in samples containing very low [<5 µg/L] total CVOHs). These low-level samples induce only a slight color change (i.e. slight darkening or light purple hue) prior to the 0.5ppm line on the tube scale at the entrance of yellow reagent layer in the LL tube. Samples containing concentrations of total chlorinated compounds above 5 µg/L usually induce a more apparent reaction within the LL tube.

6.1 Very Low Concentrations

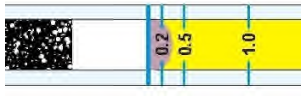
When a sample contains very low concentrations (<10µg/L) of chlorinated compounds, the resulting color change is not immediate or distinct. At these low concentrations the color change does not usually begin until between 100 and 200 CCs of air have purged through the sample into the tube. Furthermore, the color change induced at these low concentrations is very slight (below 0.5 on the tube scale) and appears as a slight darkening or light purple hue at the entrance of yellow reagent layer in the LL tube.

6.2 Low to Medium Concentrations

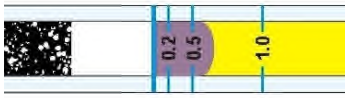
When the sample contains higher concentrations (>10 µg/L) of chlorinated compounds, the resulting color change is an obvious light to dark purple, which propagates through the yellow reagent layer toward the pump end of the colorimetric tube. The detected concentration level is obtained by matching the linear extent of the discolored reagent inside the tube to the calibration scale printed on the outside of the tube.



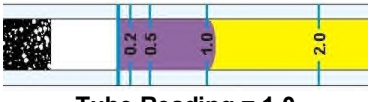
Tube Reading = 0.1
Estimated Concentration = 3 µg/L



Tube Reading = 0.2
Estimated Concentration = 15 µg/L



Tube Reading = 0.6
Estimated Concentration = 45 µg/L



Tube Reading = 1.0
Estimated Concentration = 70 µg/L

Tube Reading = 2.0
Estimated Concentration = 145 µg/L

6.3 High Concentrations

When the sample contains high concentrations (>100 µg/L) of chlorinated compounds, the color change reaction occurs quickly and usually exceeds the upper detection level of the Gastec® 133LL tube. The higher the concentration of chlorinated compounds in the sample, the faster the color change reaction occurs and the further it propagates through colorimetric tube. Samples containing very high concentrations (>1000 µg/L) of chlorinated compounds, often discolor the entire yellow reagent layer in the LL tube before the pump handle has been fully extended. In these cases, the purging can be discontinued to allow for the current sample bottle to be re-tested using a higher range detector tube. There is no need to continue purging the sample when the detection level of the tube is exceeded. Each subsequently higher range tube (133L, 133M, or 133HA) is used to purge each new duplicate sample in succession until the color change reaction does not exceed the calibration range of the tube being used.

6.4 Recording Tube Readings

It is recommended to record the observed concentration value (tube reading), the range of the colorimetric tube (LL, L, M, or HA) and the final purge volume when logging Color-Tec results. For example, a reading of 2.5 observed on an LL tube using a 100 ml purge should be recorded as **2.5/LL/100**. Purge volume correction factors must be applied for Color-Tec values which were obtained using any purge volume other than 100cc. For example, a reading of 0.2 observed on an LL tube using a 200 ml purge should be recorded as **0.1/LL/200**. A reading of 60 observed on an M tube using a 50 ml purge should be recorded as **150/M/50.**

7.0 Estimating Sample Concentrations (Conversion Table)

The Color-Tec reading (the distance that the color change travels through the tube) is a **relative response** to the amount of chlorinated-compound molecules that have been purged from the sample and directed into the tube. Therefore, the units printed on the tubes are used only to record the **relative response** for each analysis in order to facilitate comparison to laboratory GC/MS methods.

To provide a field-ready estimate of the total chlorinated solvent concentration in liquid and solid samples based on the colorimetric tube reading, The developer of the Color-Tec method created a conversion table (see Table 2) based on statistical comparison of water samples collected from chlorinated solvent sites in which the Color-Tec and GC/MS methods were used to analyze split samples. An estimated concentration may be obtained by matching the Color-Tec tube response to either the *median expected GC/MS concentration* or the *range of expected GC/MS concentrations* provided on the comparison table. The potential range of corresponding analytical values associated with each positive tube reading increases significantly as the sample concentration increases. The estimated concentrations presented on Table 2 represent the central tendency of the comparison data. The actual analytical values obtained by laboratory analysis of split samples may differ substantially from this estimate and may fall outside of the corresponding ranges provided on Table 2.

The expected GC/MS concentrations presented in Table 2 are based on comparison of water sample data only. These conversion values may also be used for soil data; however, the potential range in expected GC/MS concentrations may be increased as a result of the difference in soil volumes used in the two methods and in the inherent heterogeneity of most soil matrices. However, the potential deviation factors included in the *range of expected GC/MS concentrations* column should be sufficient to account for the intrinsic analytical variability of most soil sample results.

THE COLOR-TEC TUBE READING IS NOT THE SAMPLE CONCENTRATION!

The tube reading is a unit-less value which must be compared to laboratory results from split samples in order to yield an estimate of the actual concentration present in the sample. This conversion table provides a reasonable estimate of the expected sample concentration based on the tube reading.

Table 2
Conversion of AQR Color-Tec Readings (Relative Responses) to Expected GC/MS Total Chlorinated Volatile Organic Halocarbon Concentrations

Gastec® Colorimetric Tube	Color-Tec Tube Reading (relative response) (unit-less)	Median Expected GC/MS (Laboratory) Concentration (µg/L or µg/kg)	Range of Expected GC/MS Concentrations (µg/L or µg/kg)	
			Low	High
133-LL	0	3	>0	5
	0.1	7	5	10
	0.2	15	10	20
	0.5	35	25	45
	0.8	55	40	75
	1	70	50	95
	1.5	110	75	140
	2	145	105	190
	2.5	190	130	245
	3	230	160	290
	5	380	260	490
	9	900	630	1,160
133-L	25	2,500	1,250	3,750
	35	4,400	2,200	6,600
	45	7,700	3,850	11,550
	55	15,000	7,500	22,500
	75	17,200	8,600	25,800
133-M	100	21,100	10,500	31,600
	200	46,000	23,000	69,000
	300	85,000	42,500	127,500
133-HA	500	225,500	112,800	338,300
	700	598,300	299,200	897,500
	900	1,587,500	793,800	2,381,300

Notes:
The **Color-Tec Tube Reading** (Color-Tec units) is the value printed on the colorimetric tube at the interface between the reacted and un-reacted reagent (the extent of the color change in the tube for a positive result).

The **Median Expected GC/MS Concentration** is the estimated concentration in micrograms per liter (µg/L) of total chlorinated volatile organic halocarbons (CVOHs) present in the sample for the corresponding Color-Tec tube response.

The **Range of Expected GC/MS Concentrations** is an estimated range of potential concentrations (µg/L or µg/kg) of total chlorinated volatile organic halocarbons (CVOHs) for the for the corresponding Color-Tec tube response.

The **Median Expected GC/MS Concentration** was obtained using statistical comparison of Color-Tec Method data and GC/MS (EPA Method 8260B) data. Comparison data were obtained from 5348 water samples collected from 152 chlorinated solvent (primarily PCE) sites in which the Color-Tec Method was used to analyze the samples in the field and either a laboratory-based or mobile GC/MS was used to analyze split samples.

The **Range of Expected GC/MS Concentrations** reflects the potential deviation in the **Median Expected GC/MS Concentration** based on Color-Tec Method/EPA Method 8260B comparison results. The potential error increases as the concentration increases. The initial deviation factor used for a Color-Tec Reading of zero is +/- 30% and increases to +/- 400% at a Color-Tec Reading of 900 units.

The **Median Expected GC/MS Concentrations** presented in this table are based on comparison of water sample data only. These conversion values may also be used for soil data; however, the potential error or range in expected GC/MS concentrations may be increased as a result in the difference in soil volumes used in the two methods and in the inherent heterogeneity of many soil matrices. The potential deviation factors included in the **Range of Expected GC/MS Concentrations** data should be sufficient to account for the intrinsic analytical variability of most soil sample results.

The expected GC/MS concentrations in this table are provided only to give Color-Tec Method users an approximate concentration for the Color-Tec Tube Response. Actual GC/MS results on split samples may be outside of the stated range for a given Color-Tec Tube Response.

Refer to the **AQR Color-Tec Manual** for detailed information regarding general method principals and potential analytical variables.

8.0 Proposed QA/QC Procedures

As with any analytical method, standard sample preparation and quality assurance/quality control (QA/QC) procedures tailored to the specific project goals should be developed and followed precisely and consistently throughout the sampling and analysis program to insure consistent results and the lowest possible detection levels for all samples analyzed using the Color-Tec method. This section is intended to provide the Color-Tec user with a basic methodology for conducting QA/QC procedures which address various potential operational and procedural issues, such as analytical confidence, method performance, false positives/negatives, replicate accuracy, and contaminant carryover. Users of the Color-Tec method are encouraged to use the information provided in this section to develop project-specific QA/QC and sample handling procedures that insure the level of consistency and accuracy required for the user's sampling program.

8.1 Analytical Confidence and Method Performance

Using Color-Tec to analyze prepared sample spikes containing known concentrations of chlorinated compounds provides confidence that the method procedures are being performed properly performed and may provide a basis for estimating concentrations based on the low-range (133LL) colorimetric tube responses. Spiked sample concentrations should range between 10 µg/L and 200 µg/L to cover the detection range of the low-range (133LL) colorimetric tube. Most analytical laboratories will prepare spiked samples in VOA vials with specified compounds at specified concentrations. Conduct Color-Tec analyses on the spiked samples using the same procedures described in Sections 3 and 4 and record the results in your field log as described in Section 5.3. A 200cc purge using two VOA vials (as described in Section 4.3) may be required to produce a positive Color-Tec reading when testing spiked samples containing 10 µg/L or less of total CVOHs may require a 200cc purge to produce a positive Color-Tec reading.

Performance/confidence testing of the higher range tubes (133L, 133M, and 133HA) using high-concentration spiked samples is unnecessary because the high range tubes are usually not used unless the sample being tested has already exceeded the upper range of the low range tube, thus revealing that the sample being tested contains a sufficient quantity of chlorinated compounds to evoke a positive reaction from the next higher range tube. Given the inherent extreme variability of estimating high concentrations based on tube responses on the high range tubes (133L, 133M, and 133HA), comparison of high concentration (>500 µg/L) spiked samples generally

8.2 Chemical Inhibitors (False Negatives)

The presence of Toluene and Xylenes inhibits/diminishes the ability of the colorimetric tubes to detect CVOHs. At sites where the presence of these compounds is suspected to be present in the soil or water samples, QA procedures may include periodic testing of groundwater or soil samples and ambient air for the presence of toluene and xylenes using a Gastec® Toluene tube (the Toluene tube also detects xylenes). To conduct a test for the presence of compounds which could inhibit the detection of CVOHs use the Toluene (122L) tube to analyze a duplicate soil or water sample using the procedures described in Sections 2 through 4.

8.3 Positive Interference (False Positives)

Chlorinated Volatile Organic Halocarbons. The Gastec® 133-series colorimetric tubes used to perform the Color-Tec method detect all chlorinated volatile organic halocarbons (CVOHs) present in each sample. Thus, individual CVOH compounds cannot be identified/isolated using this method. But rather, each positive tube reading represents the sum total of all CVOH compounds present in the sample as "total CVOHs". This detection of the entire class of compounds is an inherent effect of the colorimetric tube design and thus may not be avoided by any alteration of method procedures.

Water Vapor. A build-up of water vapor in the colorimetric tube in the oxidizer stage (black portion of the tube) and through the catalyst stage (white portion of the tube) can induce a subtle color change similar to that of a low-level positive result if the moisture reaches the reagent stage (yellow portion of the tube). This problem is easily avoided by observing the build-up of condensation inside the tube in the oxidizer stage during purging, and stopping the airflow before the condensation reaches the white catalyst stage. This condition rarely occurs before the maximum required purge volume of 200 CCs is achieved and contaminant presence or absence has been determined.

Hydrogen Chloride Vapor. Hydrogen chloride vapor is the reactant that causes the color change in the yellow reagent used in the PCE colorimetric tubes. The HCl vapor is formed when chlorinated halocarbons pass through the oxidizer and catalyst stages of the tube. Free HCl vapor can also be formed when strong hydrochloric acid comes into contact with air or calcium carbonate. Any source of free hydrogen chloride vapor which enters the colorimetric tube will cause a strong positive reaction. To minimize the risk of false positives from hydrogen chloride vapor, avoid the use of pre-preserved VOAs when using the Color-Tec method. Natural sources of hydrogen chloride vapor are rare.

Free Chlorine. Very high (>20,000 ppm) concentrations of free chlorine can cause a low-level positive reaction in the 133LL colorimetric tube. The conditions necessary for this positive interference rarely occur in groundwater or soil samples.

Contaminant Carryover. It is highly recommended that VOA vials and extraction needle assemblies be discarded following each test. Re-use of these expendable items may cause sufficient carryover of contaminants to cause a false positive result in subsequent samples.

8.4 Ambient Air Interference

Because the Color-Tec method uses ambient air as the purge gas, airborne chlorinated compounds at low concentrations can enter the sample and cause a positive reaction in the detector tube. Conversely, low concentrations of either toluene or xylenes present in the ambient air may enter the colorimetric tube and inhibit/diminish the tube's ability to detect CVOHs. To prevent airborne contaminants from entering the sample and detector tube during sample purging and analysis, the method is used with a carbon pre-filter attached to the purge needle. To determine whether airborne chlorinated contaminants are present, a PCE (133LL) colorimetric tube may be used periodically to test the ambient air at the location where the field testing is being performed. If airborne contaminants are present and the carbon filter is being used, the carbon filters can also be tested periodically using a colorimetric tube to determine if breakthrough is occurring. The ambient air may be similarly tested for the presence of xylenes or toluene using the PCE (133LL) colorimetric tube.

To conduct a test for the presence of chlorinated VOHs in the ambient air, break the tips of a PCE (133LL) or PCE (133LL) colorimetric tube and properly insert it into the hand pump. Pull and lock the pump handle into the 100cc position allowing ambient air to enter the colorimetric tube. *Note: Do not attach an extraction needle assembly to the colorimetric tube while performing this test.* Once the 100cc flow cycle is completed, carefully read the tube and record the results. A positive result indicates the presence of CVOCs in the ambient air at concentrations detectable by Color-Tec which would affect sample results unless the carbon filter assembly is attached to the purge needle (see Section 9). A negative result indicates that CVOCs are not present in the ambient air at concentrations detectable by Color-Tec and therefore will not affect sample results. It is recommended that the carbon filter assembly is used regardless of the ambient air testing results.

8.5 Duplicate Sample Testing Procedure

Duplicate or replicate samples are collected from the same sampling location, at the same time, using the same collection methods, and analyzed using the same procedures as the original samples for the purpose of determining both sampling and analytical method variability. Since a second (duplicate) VOA vial is always collected for the Color-Tec method, a duplicate or replicate analysis may be performed on the second (duplicate) VOA vial any time that a positive result (color change) is evoked by the original sample (first VOA vial) without exceeding the upper limit of the low-level colorimetric tube. In those cases, the duplicate or replicate analysis is simply performed by using a new low-level colorimetric tube to analyze the duplicate sample in the second (unused) VOA vial. If sampling and method variability is low, the result of the duplicate test will be the same or similar to the results obtained from the original test. The relative percent difference (RPD) may be calculated to quantify any variability in the results.

8.6 Collection of Split Samples for Laboratory Analysis

It is recommended that sample splits be collected for laboratory comparison analysis from 5 to 20 percent of the total quantity of samples analyzed using the Color-Tec method. Given a sufficient quantity of split sample pairs and sufficient range of concentration values, the GC/MS-to-Color-Tec comparison data may be used to obtain estimated concentrations for samples in the data set which were analyzed only using the Color-Tec method. This can be achieved using linear regression analysis of the comparison data. Statistical analysis of the comparison data can also be performed to determine site-specific Color-Tec method performance data.

9.0 Safety Precautions

As with the use of any product, it is recommended that the user carefully review all product manuals and Material Safety Data Sheets (MSDS) provided with this product prior to use. Several components of the Color-Tec kit are products obtained from other manufacturers which have manuals including safety precautions. Users of the Color-Tec method should carefully review the manuals and safety precautions and should become familiar with the proper use of all components included in the Color-Tec kit. It is recommended that the procedures involved with the method be incorporated into the user's Site-specific Safety and Health Plan (SSHP). MSDSs for all chemicals provided as part of the Color-Tec kit are available upon request. The following precautions should be considered to reduce potential user safety risks associated with the performance of the Color-Tec method.

Activity	Potential Risk	Precaution
Breaking tube tips	eye injury, dermal puncture	safety glasses
Accidental tube breakage	dermal cuts, exposure to reagent	safety gloves
Use of purge/extraction needles	dermal puncture	use caution
Use of the hot plate	dermal burns, electric shock	limited setting
Use of PCE standards	dermal contact, dermal cuts	safety gloves

Additional Safety Notes:

- Use skin and eye protection while breaking colorimetric and carbon filter tubes;
- The thermostat dial setting of the Corning® Hot Plate should never be set above 5 for any heating purposes required by the Color-Tec method;
- Do not over-fill the water bath pan while heating the samples and tubes;
- Always conduct sample and tube heating activities on a flat, stabile, surface.
- Keep all flammable or combustible materials away from the Corning® Hot Plate during sample and tube heating activities.

- Always use the stainless-steel water-bath pan properly filled with water for heating the samples and tubes – do not heat samples or tubes directly on the surface of the Corning® Hot Plate;
- Do not use any heat source to heat the water-bath, tubes, or samples other than the Corning® Hot Plate provided in the hardware kit.

Disposal of Expendable Materials:

- Re-cap all needles before disposal;
- After re-capping each extraction needle, dispose of the extraction needle assembly while leaving the vinyl tubing attached to the colorimetric tube – Do not attempt to remove the extraction needle assembly from the tip of the colorimetric tube for disposal;
- Dispose of all sharps (needles and broken glassware) in accordance with any and all applicable local and/or federal rules or guidance.
- Dispose of all colorimetric tubes as specified in the Gastec® MSDS and/or in accordance with any and all applicable local and/or federal rules or guidance.
- Dispose of all VOA vials used to contain sample materials in accordance with any and all applicable local and/or federal rules or guidance.

Product Warranty

AQR warrants that the goods sold herein will be free from defects in material and workmanship. This warranty shall be limited to the replacement of defective parts. It is expressly agreed that this warranty shall be in lieu of all warranties of fitness and in lieu of the warrant of merchantability.

EPA Guidance Document References

Using Dynamic Field Activities for On-Site Decision Making
May 2003; OSWER No. 9200.1-40 EPA/540/R03/002; Chapter 5;
http://www.epa.gov/superfund/programs/dfa/download/guidance/40r03002.pdf

Site Characterization Technologies for DNAPL Investigations September 2004; EPA 542-R-04-017;
http://www.clu-in.org/download/char/542r04017.pdf

Understanding Procurement for Sampling and Analytical Services under a Triad Approach June 2005, EPA 542-R-05-022;
http://www.epa.gov/swertio1/download/char/procurement.pdf

Conducting Contamination Assessments at Drycleaning Sites EPA Technology Innovation Program; State Coalition for Remediation of Drycleaners; http://www.drycleancoalition.org/download/assessment.pdf

EPA Triad Implementation References

Using AQR Color-Tec for Source Identification and Delineation
Naval Construction Battalion Center Davisville North Kingstown, RI - 2008 Triad Conference;
http://www.umass.edu/tei/conferences/Triad_PDF/Anderson.pdf

Fast Track to Reducing Conceptual Site Model Uncertainty CH2MHill; Storage Tank Site ST-123 POL Fuel Yard;
http://www.Triadcentral.org/user/includes/dsp_profile.cfm?Project_ID=25

Best Practices in Triad Approach to Characterize TCE, National Laboratory Environmental Sciences Division Argonne, IL;
http://www.triadcentral.org/user/doc/TPP-Hurlburt-BestPractices.pdf

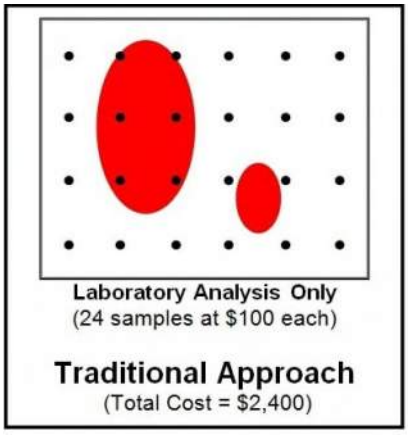
Adaptations to Triad as a Basis for Exit Strategy Development Decision Logic Flow Chart 2006 Triad Poster Session;
CH2MHill; http://www.triadcentral.org/user/doc/TPP-Hurlburt-TriadAdaptations.pdf

Successful Triad Implementations at Federal Sites AQR Color-Tec Method locates source areas at Calloway Drum Recycling Site, Auburndale, Florida http://www.triadcentral.org/user/doc/TPP-Callaway-Field_Based_Decision_Approach.pdf

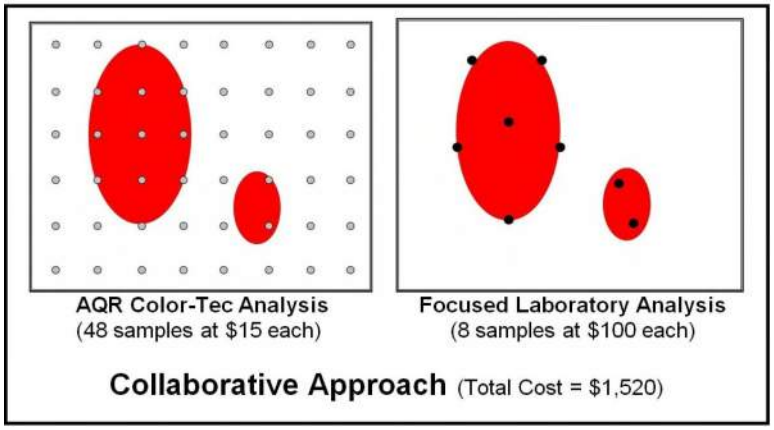
Color-Tec Method Applications

Source Area Identification at chlorinated solvent sites is highly complex given the low solubility of these compounds in water. Chlorinated solvent source zones often persist as suspended residual in unsaturated and saturated subsurface sediments for many decades. Surface water infiltration and groundwater flowing through the source zones slowly dissolves the suspended residual solvent leading to substantial aqueous phase contaminant plumes. Given the high volatility of most chlorinated compounds, residual solvents suspended in the unsaturated soil often leads to significant vapor phase contamination. The Color-Tec method is ideal for locating chlorinated solvent source areas by combining low level detection of all chlorinated compounds with low per sample cost to allow for significant expansion of sampling coverage compared to assessment approaches where only definitive analytical (laboratory) methods are employed to locate source areas. Definitive laboratory analysis provides high analytical accuracy, but sampling quantity is often limited to control costs, resulting in data gaps, sampling uncertainty, and low overall data quality. The low per-sample cost of Color-Tec method offers a 6:1 increase in analysis volume over laboratory methods, allowing for five times the sampling coverage for the same cost.

The illustrations below compare the traditional approach of source identification which uses only definitive laboratory analysis, to a collaborative approach which uses a high volume of Color-Tec data combined with a low quantity of definitive laboratory data. This collaborative approach combines high volume/low accuracy with low volume/high accuracy to achieve higher overall data quality than either method alone.



The diagram to the left shows the traditional site investigation scenario in which all samples collected are analyzed using only definitive analytical methods. The red areas represent previously unidentified source areas and black dots represent sampling locations intended to locate and delineate the contaminant plumes. Although this definitive-analysis only approach provides high analytical accuracy, the sampling quantity is often limited in order to control costs, resulting in data gaps, sampling uncertainty, and low overall data quality – and in this example the smaller source area remains undetected.



The two diagrams to the right show an investigation scenario in which a real-time measurement method, such as Color-Tec, is used to increase the overall sampling coverage, resulting in reduced sampling uncertainty and increased overall data quality. In this example, the smaller source area is identified and the Color-Tec data is verified and confirmed by focusing a reduced quantity of definitive, laboratory-based, analysis of split-samples onto the most critical areas of the site. Combining Color-Tec with focused laboratory analysis in this manner provides increased overall data quality and analytical accuracy at significantly lower costs than conventional approaches which rely only on definitive laboratory-based analysis.

Groundwater Profiling is the collection of discrete samples at multiple depths and locations working outward from known source areas to define the lateral and vertical extent of a dissolved groundwater contaminant plume. The technique is used in conjunction with the Color-Tec method at chlorinated solvent sites to allow for immediate decisions regarding subsequent vertical and lateral sampling locations.

Soil Matrix Profiling is similar to groundwater profiling, but uses sampling of the unsaturated soil to define the lateral and vertical extent of the vapor phase contamination.

Groundwater Matrix Profiling (Residual Zone Mapping) is similar to groundwater or soil profiling, but uses sampling of saturated unconsolidated aquifer matrix to define the lateral and vertical extent of suspended residual DNAPL.

Surface Water/Sediment/Pore Water Impact Evaluation is the collection and analysis of sediment, sediment pore water, and surface water to locate and characterize groundwater impacts on surface water.

AQR Color-Tec®

Contact and Ordering Information

- **For more information visit http://www.aqrcolor-tec.com/**
- **For kit orders contact Phil Pecevich at 919-918-7191**
  Email: pecevica@bellsouth.net

Equipment and Expendables

- **Hardware kit includes piston pump, pump stand, and heating equipment in a Pelican® hard case**
- **Expendables provided in 20-sample packs**
- **Expendables for QA/QC tests sold separately**
- **Cost per sample is $19.95**
- **Volume discounts available**
- **Professional technical support is included with every purchase**
- **Professional in-house or web-based training is available**

Hardware Kit

20-Sample Pack

KT-10 v2

Magnetic Susceptibility, Conductivity

and Combined Magnetic Susceptibility / Conductivity Meter

with GEOVIEW

User's Guide

Ver. 2.1

Terraplus
Geophysical Equipment Supplier

Phone: (905) 764-5505
Fax: (905) 764-8093
Web: http://www.terraplus.ca

KT-10 v2

Congratulations on your purchase of a KT-10 v2 (version 2) meter! Please read through this manual to familiarize yourself with your new instrument.

The KT-10 v2 (version 2) is available in a variety of different model configurations: KT-10 v2, KT-10 v2 Plus, KT-10 v2 C, KT-10 v2 Cx, KT-10 v2 S/C, KT-10 v2 Plus S/C, or KT-10 v2 Plus S/Cx. All of these models are available with either a circular or rectangular coil. The basic KT-10 v2 can also be upgraded to include extended magnetic susceptibility, conductivity and/or extended conductivity measurements. All upgrades can be performed via the internet. Please contact **Terraplus'** Sales Department at sales@terraplus.ca for further information.

This User's guide describes the operation of the meter as a combined magnetic susceptibility/conductivity meter, a magnetic susceptibility meter, or a conductivity meter.

The illustrations contained within this manual are of the KT-10 S/C – a combined Magnetic Susceptibility/Conductivity Meter.

Table of Contents

Chapter 1: Introduction

1.1 General Information Page 7
1.2 Operational Theory Page 7
1.2.1 Theory Page 7
1.2.2 Operating Principles Page 8

Chapter 2: The KT-10 v2

2.1 Specifications Page 9
2.2 Features Page 10
2.3 Layout Page 13
2.4 Controls Page 14
2.5 Menus Page 15
2.6 Icons Page 16

Chapter 3: Operating the KT-10 v2

3.1 Battery Installation Page 17
3.2 Power Page 18
3.2.1 Power On Page 18
3.2.2 Power Off Page 18
3.3 Setup Menu Page 19
3.4 Measure Page 25
3.4.1 Taking a reading Page 25
3.4.2 Storing a reading Page 27
3.4.3 Measurement Sub Menu Page 29
3.4.4 Measure Flow Chart Page 30
3.4.5 Measure Sequence Page 31

3.5 Scanner Page 32
3.5.1 Take a reading Page 32
3.5.2 Store a Reading Page 34
3.5.3 Measurement Sub Menu Page 35
3.5.4 Scanner Flow Chart Page 37
3.5.5 Scanner Sequence Page 38
3.6 Borehole Measure mode Page 39
3.6.1 Borehole Configuration Page 39
3.6.2 Scanner setup Wizard Page 41
3.6.3 Scanner measurement Page 43
3.6.4 Discrete mode setup Wizard Page 46
3.6.5 Discrete mode measurement Page 47
3.7 Voice Recorder Page 49
3.8 PIN Installation Page 50

Chapter 4: Software

4.1 GeoView Page 53
4.1.1 Installation Page 53
4.1.2 GeoView Calendar Interface Page 60
4.1.3 GeoView Data Interface Page 66
4.1.4 Data Download Page 67
4.1.5 Data Export Page 69
4.1.6 Borehole Mode data Display Page 75
4.1.7 Device Settings Page 76
4.1.8 Firmware Upgrade Page 78
4.2 Console Page 81

Chapter 5: Bluetooth Connections

5.1 PC Connections Page 87

5.2 GeoVision – Android app Page 91
5.2.1 GeoVision Installation Page 91
5.2.2 GeoVision Menu Page 93
5.2.2.1 GeoVision Pairing Page 94
5.2.2.2 Browse records Page 96
5.2.3 Graph / Scale Page 100
5.2.4 Zoom / Pan Page 102
5.2.5 Delete Data Page 102

Chapter 6: Troubleshooting

6.1 Note about switching off Page 103
6.2 Meter turns off during measurement Page 103
6.3 “Error” on screen Page 104
6.4 Maintenance Page 105
6.5 Contact Technical Support Page 105

Appendix A: **KT-10 v2 Plus feature** Page 106
Appendix B: Advice & Recommandations Page 114

This page is intentionally left blank.

Chapter 1

Introduction

1.1 General Information

The KT-10 v2 is an advanced hand-held magnetic susceptibility meter. The KT-10 v2 was developed as a joint venture between Terraplus Inc., a geophysical instrument supplier located in Richmond Hill, Ontario (Canada), and Georadis S.R.O., a Czech Republic based design and manufacturing company.

The KT-10 v2 is best utilized for obtaining accurate and precise measurements from outcrops, drill cores and rock samples. The KT-10 v2 is capable of measuring uneven rock surfaces and is well suited for automated drill-core logging with a digitally recorded scanning mode.

With its leading edge technology, the KT-10 v2 offers high sensitivity, higher operational comfort and excellent communication capabilities.

IMPORTANT: Your KT-10 v2 meter is calibrated at the factory and a periodic calibration is not required.

1.2 Operating Principle and Theory

1.2.1 Theory

Magnetic susceptibility is defined as the degree to which a substance can be magnetized. In mathematical terms, it is the ratio ***k*** of the intensity of the magnetization ***I*** to the magnetic field ***H*** that is responsible for the magnetization, i.e.

$$kH = I$$

From Ampere's law, it is known that a current (i.e. a moving electrical charge) generates a magnetic field. The inverse corollary to this is that a magnetic field can also influence a moving electrical charge. Thus, an oscillating EM field will be influenced to varying degrees by a magnetically susceptible material.

Conductivity is an intrinsic property of a microscopic volume of material. Apparent conductivity is a volume average of a heterogeneous half-space except that the averaging is not mathematical but dependent on each instrument. Only when the earth is a homogenous half-space is the apparent conductivity the same as the true conductivity. The main advantage of the electromagnetic conductivity method is that contact with the sample is not required. If the conductive material is moved near to the measurement coil, new elementary electric circuits are created. The sample will then behave like small secondary coils and influence the magnetic flux through the measurement coil caused by mutual inductance. Naturally, this leads to a change in the frequency. In general, the amplitude of the voltage signal decreases if the conductivity increases and vice versa. Our method is based on the analysis of this change.

1.2.2 Operating principle

The KT-10 v2 utilizes a 10 kHz LC oscillator with an inductive coil to measure the magnetic susceptibility and conductivity. Magnetic susceptibility is calculated from the frequency difference between the sample and free air measurements, while conductivity is calculated from the difference in amplitude between the two. It also takes into account geometric corrections to determine the true susceptibility. The frequency of the oscillator is extremely sensitive to temperature deviations. Any temperature instability is propagated in frequency deviations and has a direct impact on maximum sensitivity. To minimize these effects, the KT-10 v2 takes multiple measurements in free air before measuring the sample, and then multiple free air measurements are taken afterwards. Then using a sophisticated algorithm, the negative impact of temperature shift is minimized.

The sequence required to obtain a measurement is:

Step 1: The frequency and amplitude of the oscillator is determined in free air.

Step 2: The oscillator frequency and amplitude is then measured when the coil is placed on a rock sample, drill core, or outcrop.

Step 3: The frequency and amplitude of the oscillator is then measured again, in free air, and then the results are displayed.

Chapter 2

The KT-10 v2 S/C

2.1 Specifications

Susceptibility Sensitivity:	$1x10^{-6}$ SI Units
Conductivity Sensitivity (optional):	1 S/m
Susceptibility Range:	0.001×10^{-3} to 1999.99×10^{-3} SI
Conductivity Range (optional):	1 - 100,000 S/m Units Auto-Ranging
Operating frequency:	10 kHz
Measurement Frequency	20 times per second in Scanner Mode (5 readings are averaged together and 4 readings per second are stored)
Display:	High Contrast LCD Graphic Display with 104 x 88 pixels
Memory:	Up to 4000 measurements with voice notes.
Control:	One button with up and down functionality. A PIN mode is available for rough surfaces.
Communication:	USB, Bluetooth and GPS link via Bluetooth
Battery:	Two AA batteries - Rechargeable or Non-rechargeable
Battery life:	Up to 3000 readings without voice recorder, using alkaline batteries
Operating temperature:	-20 °C to 60 °C
Dimensions:	200mm x 57mm X 30mm
Coil Diameter:	65 mm
Weight:	0.30 kg

2.2 Features

The KT-10 v2 has many features that make it stand out in the crowd of available hand held magnetic susceptibility & conductivity meters, and they are as follows:

Multiple Configuration Capability (optional)*

The KT-10 v2 can be upgraded to include conductivity measurements. With this upgrade it can be configured as a simultaneous magnetic susceptibility/conductivity meter, a magnetic susceptibility meter, or a conductivity meter.

Android Application for Real Time Profiling (optional)*

The optional GeoVision software can be used to display real time scanner profile on Android platform operated smart phones. The large display of the smart phone will show real time graphical output while scanning and can be used as a data browser to display field measurements/records in the KT-10 v2 memory. The keypad of the smart phone can be used to enter additional text notes to the current data or any previously stored data on the KT-10 v2.

True Susceptibility and Conductivity

The KT-10 v2 uses automated correcting routines to display true susceptibility and volume conductivity.

Uneven Samples

The KT-10 v2 is offered with a **PIN** for rough surface measurements. When measuring field samples or outcrops with the **PIN**, and when the meter is kept parallel to the surface, it provides a reading with increased accuracy. It also automatically corrects and displays the true magnetic susceptibility and conductivity.

Fast Scanning

The KT-10 v2 scans up to 20 readings per second; increasing the amount of information that user can obtain per location or sample.

Please note: 4 readings per second are stored on the KT-10 v2; 5 readings are averaged together for each of the 4 readings per second.

Depth Correlation

Before starting the measurement routine, the user can now enter information such as borehole ID, box ID and the number of rows in a box, along with the start depth, end depth and depth interval. The user will have the option to choose between imperial or metric as their desired unit of measurement. In the Scanner mode, depth intervals can be recorded with the push of a button while scanning a core. All readings between depth intervals are interpolated.

Variable Audio

When used in the **Scanner Mode**, the KT-10 v2 meter's loudspeaker allows the operator to monitor the variation in the Magnetic Susceptibility and Conductivity measurements with a variable audio tone, which reflects the relative intensity of the reading. In the combined mode, Susceptibility & Conductivity, the variable audio is active for the primary reading. For example, in Cond+Sucs mode the audio will reflect the intensity of the Conductivity measurements. The voice recorder allows the recording and replaying of voice messages through the loudspeaker as well.

Data Storage

The KT-10 v2 can store up to 4000 measurements with two minutes of comments per reading, to its internal non-volatile memory. Average readings and standard deviation are also stored. The operator can record comments associated to specific readings through the KT-10 v2 digital voice recorder.

Data Averaging

Stored results of the measurement are automatically added in an averaging buffer. The average value is displayed on the LCD along with standard deviation. The operator can erase the buffer and control how many results will be averaged together.

USB Data Transfer

The KT-10 v2 uses USB communication standards as the default mode of communication. It allows fast data transfer of measured values and digital voice streams from the unit to any Windows PC. The USB connection can be also used for firmware upgrades and device parameter settings.

Bluetooth Connectivity

The KT-10 v2 comes standard with Bluetooth capabilities. When the KT-10 v2 is paired to an external Bluetooth enabled GPS, the user can store the GPS coordinates in the KT-10 v2's memory along with the readings. Bluetooth can also be used to download readings from the unit along with the voice streams to a computer. Alternately, one can also pair the KT-10 v2 to an Android running smart phone to obtain Real Time Scanner Profile.

Rain and Dust Proof

The KT-10 v2 meets IP65 standards; therefore it is protected against dust and provides additional protection in rainy or high humidity conditions.

Large LCD Display

A high contrast LCD is utilized for the display of both the magnetic susceptibility and conductivity readings and serves as the interface for operating the instrument. Together with two buttons and graphical menus, operators can interactively navigate between the different functions. On screen notification icons allows the operator to monitor the battery status, Bluetooth connectivity, GPS support and more.

Power

Power saving techniques and use of low consumption components allow for the use of high speed communication standards (USB, Bluetooth) and supports the large LCD display. The meter is powered by 2 AA type batteries. The user can use any brand of rechargeable or non-rechargeable battery. The unit does not contain an internal battery charger. Therefore, rechargeable batteries must be charged outside the KT-10 v2 in a suitable battery charger provided or similar.

Storage / Transportation

The KT-10 v2 is delivered in small pouch/case with a foam insert. The pouch can be mounted on a belt and comfortably carried on the waist. A set of spare batteries and **PINs** can be also placed in the pouch for storage. Even though the KT-10 v2 has been designed to be a rugged instrument, it still can be damaged by severe impacts. Also, please note that the KT-10 v2 is only water resistant, not water proof. Consequently, total immersion in water or long exposure in heavy rain is not advisable for this instrument.

2.3 KT-10 v2 Layout

Figure 1: KT-10 v2 Layout (KT-10 S/C shown)

2.4 Controls

To control the KT-10 v2, there is one button with associated UP/DOWN functions. There are five different options to use this button and they are as follows:

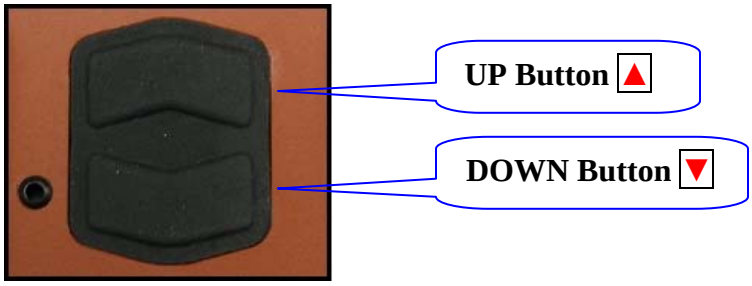


Figure 2: Control Button

SBP▲ Short Button Press UP – is a single short push of the button, pointing to screen, in its upper half. This symbol will be used throughout this document to represent this button press **SBP▲**

LBP▲ Long Button Press UP – hold the button in its upper half for more than one second or until a reaction appears on display. This symbol will be used throughout this document to represent this button press **LBP▲**

SBP▼ Short Button Press DOWN – is a single short push on the button, pointing away from screen, in its lower half. This symbol will be used throughout this document to represent this button press **SBP▼**

LBP▼ Long Button Press DOWN – hold the button in its lower half for more than one second or until a reaction appears on the display. This symbol will be used through out this document to represent this button press **LBP▼**

SBP▲ **SBP▼** Both buttons pressed together will turn the unit off at any time during operation. This symbol will be used throughout this document to represent this button press **SBP▼** **SBP▲**

2.5 Menus

The first screen you will see when you power on the meter will be a start up screen (shown in **Figure 3).**

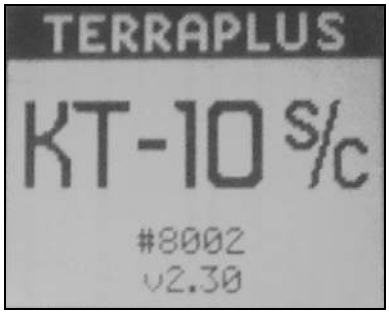


Figure 3: Start-up Screen

The information displayed on the start-up screen will be the unit's serial number and firmware revision, which is currently v2.70, at the time of this writing. The start-up screen will be displayed for about 2 seconds and then the main menu will appear (shown in **Figure 4**).

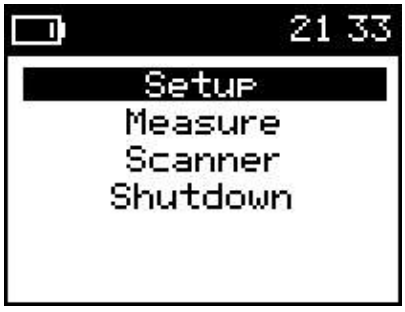


Figure 4: Main Menu

Measure and **Scanner** are the two modes of operation for the KT-10 v2. Selecting one of these options will activate the measurement routine for that mode. **Setup** is used to configure mode, date & time, core size selection, connection of Bluetooth GPS unit, and battery type selection along with calibration information. **Shutdown** will turn the unit off.

For detailed information on each measurement selection and the settings menu please refer to **Chapter 3** for Operation of the KT-10 v2 (starting on Page 17).

2.6 Notification Icons

There are several icons used on the KT-10 v2, which are displayed in the top bar of the instrument, known as the notification area. The time and battery indicator are displayed in this location and are displayed permanently during operation of the instrument. Below you will find a list of the other icons you will see used on the meter and the meaning for each of them.

Pin used for measurement

Readings have been saved

Button used for measurement

USB is connected

Core diameter selected

Bluetooth is connected

GPS is connecting

GPS is connected

Voice Note

Chapter 3

Operating the KT-10 v2

3.1 Battery Installation

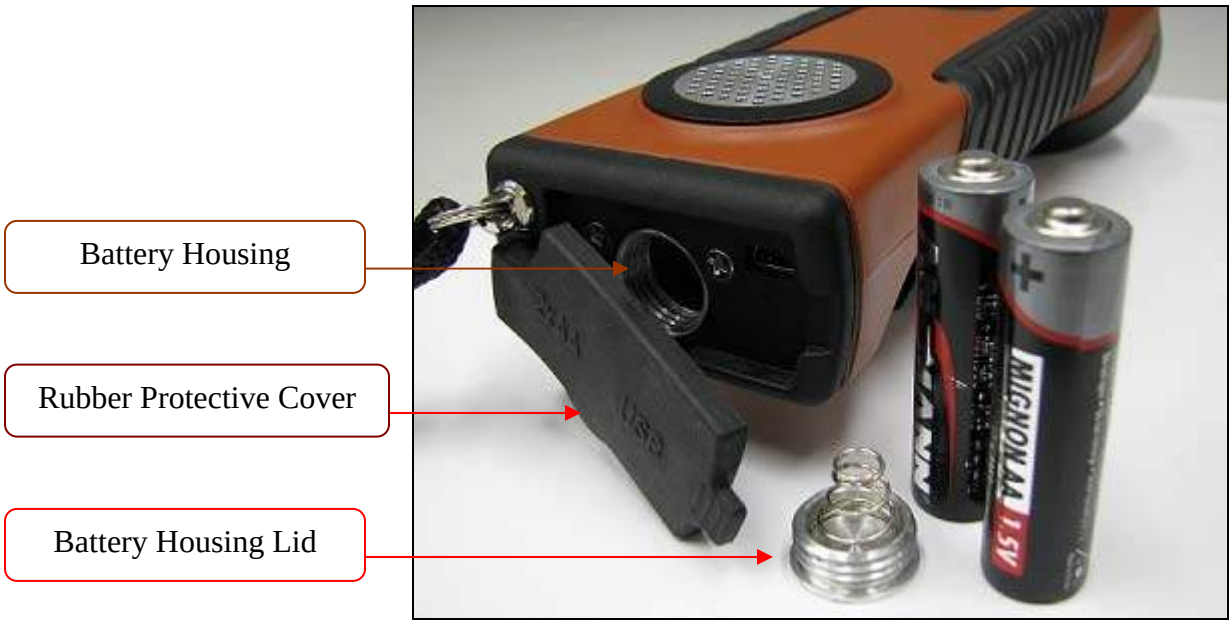


Figure 5: Battery Installation

To install batteries follow this procedure:

1. Bend the rubber protection cover on the rear of the instrument to gain access to the battery housing lid.
2. Use any flathead screw driver, or suitable coin, to open the lid.
3. Insert two rechargeable AA cell batteries provided. The positive side goes in first.
4. Close the battery housing by screwing the lid back onto it.
5. Attach the rubber protective cover back in position.

Note: If you are going to store your KT-10 v2 for long a period of time, please remove the batteries from the unit to prevent damage from electrolyte leakage. It is also recommended that you visually inspect the batteries after any long storage interval.

3.2 Power

3.2.1 Power ON

To power the unit ON, use **SBP▲.** An introductory screen (seen below in **Figure 6**) accompanied by a melody will be presented.

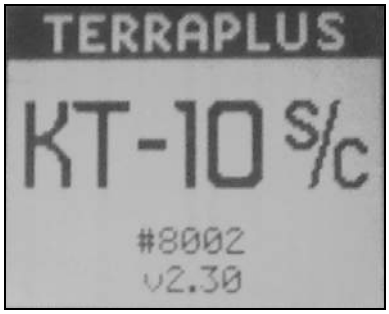


Figure 6: Power On

3.2.2 Power OFF

To power the unit off, use **LBP▲** or **LBP▼** on the Shutdown option from the main menu. You will be presented with the following screen…

Figure 7: Power Off

Alternatively

Press **SBP▲** and **SBP▼** together, to turn the meter off at any time during operation. The shut down screen will not appear. Instead the unit will immediately turn off

TIP: Place your finger or thumb tip between the two buttons to simplify this task.

3.3 Setup Menu

The Setup menu contains several different parameters to configure the KT-10 v2's operation. The selections in this menu are: **Mode, Core Diameter, Measure Units, Date/Time, Accessories, Advanced**, and **Main Menu**. To navigate to the desired parameter use the **SBP▼** or **SBP▲**, then when the parameter is highlighted use **LBP▼** or **LBP▲** to activate it.

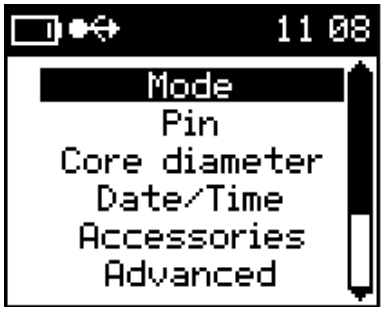


Figure 8: Setup Menu

Mode: (Optional for KT-10 v2)

KT-10 v2 can be operated in 3 different configurations: measure Magnetic Susceptibility and Conductivity simultaneously, measure only Magnetic Susceptibility, or measure Conductivity only.

For ease of operation, the user can select from 4 modes; Susceptibility, Conductivity, Susceptibility & Conductivity, or Conductivity & Susceptibility.

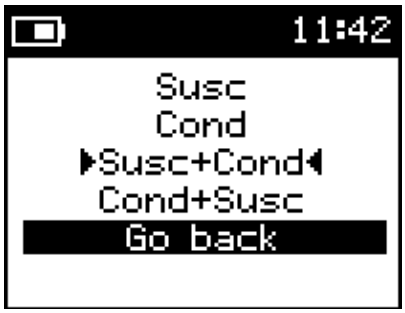


Figure 9: Mode menu

Sucs:

Enables the meter for susceptibility measurements. Along with susceptibility results, user can obtain data average and standard deviation values in the measure mode and data average and maximum values in the scanner mode
(Seen in **Figure 10**).

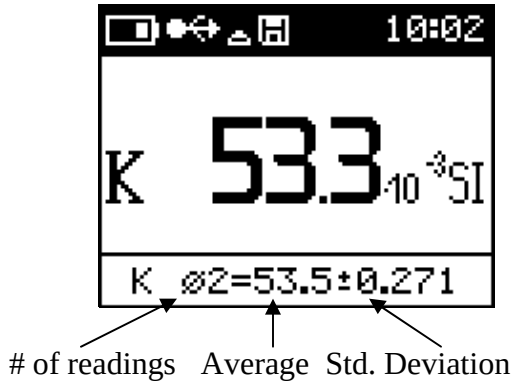


Figure 10 : (a) Measure Mode

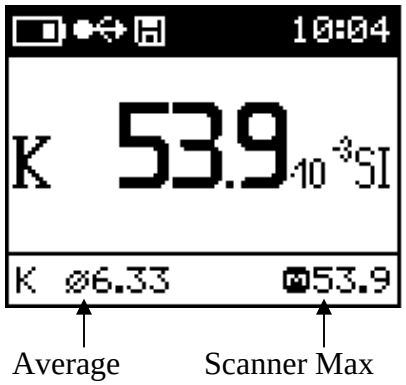


Figure 10 : (b) Scanner Mode

Cond:

Enables the meter for conductivity measurements. Along with conductivity results, user can obtain data average and standard deviation values in the measure mode and data average and maximum values in the scanner mode
(seen in **Figure 11**).

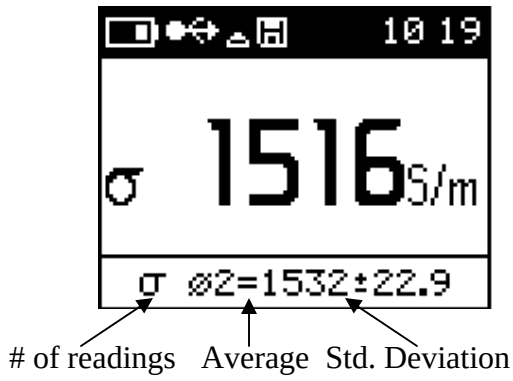


Figure 11 : (a) Measure Mode

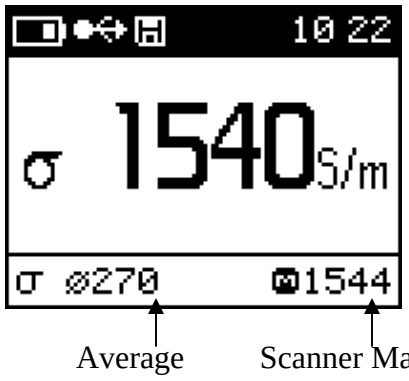
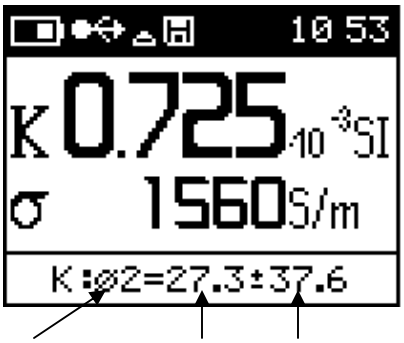


Figure 11 : (b) Scanner Mode

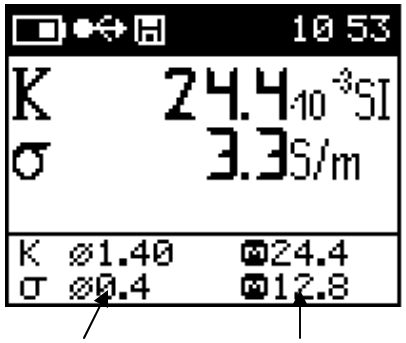
Susc+Cond:

This mode will enable the meter to read both susceptibility and conductivity simultaneously. In this mode, measurement of magnetic susceptibility is considered primary reading and the results of the primary readings are displayed in large text. User can obtain data average and standard deviation values in the measure mode and data average and maximum values in the scanner mode. (shown in **Figure 12**).



# of readings Average Std. Deviation

Figure 12 (a): Measure Mode

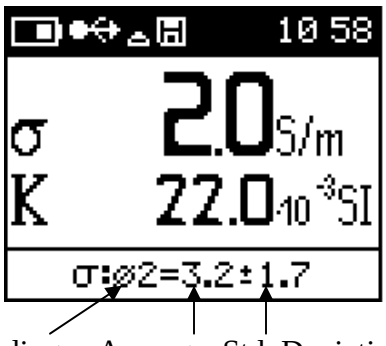


Average Scanner Max

Figure 12 (b): Scanner Mode

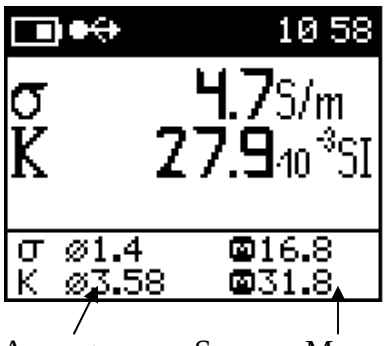
Cond+Susc:

This mode will enable the meter to read both conductivity and susceptibility simultaneously. In this mode, measurement of conductivity is considered primary and the results of the primary are displayed in large text. User can obtain data average and standard deviation values in the measure mode and data average and maximum values in the scanner mode. (shown in **Figure 13**).



# of readings Average Std. Deviation

Figure 13 (a) : Measure Mode



Average Scanner Max

Figure 13 (b) : Scanner Mode

Pin

The KT-10 v2 is equipped with a PIN for rough surface measurements. To enable the meter in the PIN mode, PIN must be selected.

Figure 14: PIN mode

Core Diameter

Contains a list of the different core diameter sizes that can be selected; both standard North American and non-standard diameters are available. When a diameter is selected, for either full cylindrical or split cores, the diameter correction is automatically applied to magnetic susceptibility and conductivity measurements.

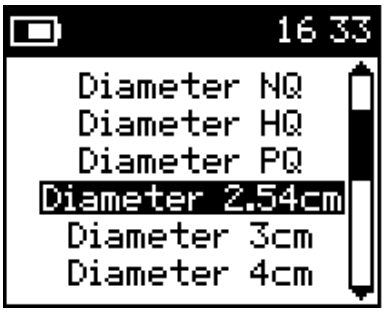


Figure 15: Core diameter

Concentration (Conc.) Tables (for Plus model only * see page 106)

The user can choose to obtain readings in either basic SI or CGS units, or as a grade estimate in a percentage (%) for any installed calibration table.

Figure 16: Measure units

Date/Time:

To set date and time on the KT-10 v2, in a 24 hour format.

Figure 17: Date/Time

Accessories

Allows for configuration of the GPS and Battery type.

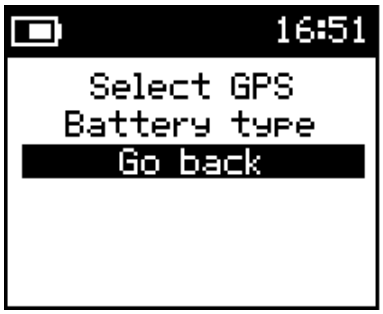


Figure 18: Accessories

Select GPS: Entering this option will start a search for Bluetooth capable GPS units. A search screen will be presented and when all the Bluetooth enabled devices have been detected, a list of all named Bluetooth devices will be displayed. Please consult the GPS user's manual for detailed information on settings for the Bluetooth GPS and any name it may use for its discovery.

Please note: Some Bluetooth GPS units require a "PIN" for secure pairing. To facilitate this, the KT-10 v2 allows entry of a "PIN" via GeoView software.

Battery Type: there are two choices for battery type selection, rechargeable and non-rechargeable.

Advanced

Provides access to the meter Calibration and QA parameters.

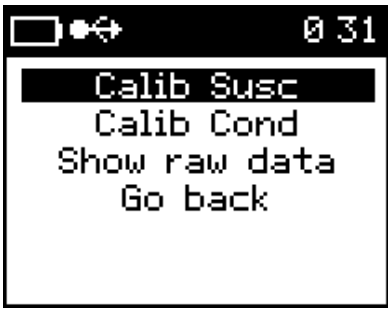
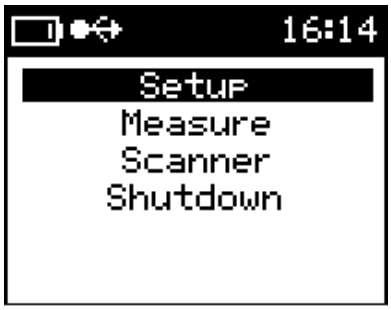


Figure 19: Advanced

Show raw data: A debug tool that monitors the frequency & amplitude of the measuring coil; this feature is primarily used at the factory.

Borehole mode

The Borehole mode allows user to integrate depth information to the data being collected in both the Measure and the Scanner mode. In the Setup menu, the Borehole mode option can be OFF or ON. When the Borehole mode is ON, the main menu will list Borehole as a method of measurement.



Borehole Mode OFF

Borehole Mode ON

Main Menu

Units

Users can obtain magnetic susceptibility measurements in either SI or CGS units; while conductivity measurements can be in either S/m or Ω.m units

* When Borehole Mode is enabled, unit of measure for distance can be selected

Main Menu:

This selection will take you back to the Main Menu.

3.4 Measure

Selecting Measure from the main menu will initialize the KT-10 v2 for a single measurement. In this mode, measurements can be obtained with (measurement of a core) or without geometric corrections to the readings. Measurements without geometric corrections to the readings are best utilized for quick recognisance of rock samples or outcrops with no specific geometry.

3.4.1 Take a reading

If **Measure** has not already been selected, use **SBP▼** or **SBP▲** to highlight this mode and select it with the use of **LBP▼** or **LBP▲**.

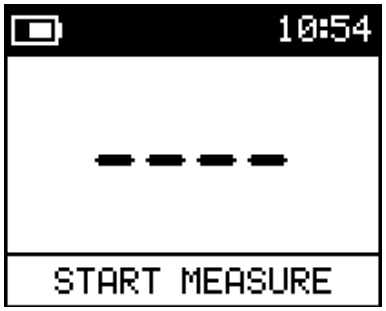


Figure 21: Start Measure

When you enter the measurement mode, the screen above will be displayed and indicate that the meter is ready to start the measurement process.

There are three steps involved in the measurement sequence: the first step is a free air measurement; the second is the sample measurement; and the final step is another free air measurement.

Note: The duration for the measurement sequence is 7 seconds long. Soon after the measurement is initiated, a new screen with 4 dashes and a progress bar will appear. It takes 7 seconds for the progress bar to complete. It is important that you do not wait for the bar to build up in between each of the 3 steps or else you will be presented with "Error" on the screen.

TIP: **Figure 26** on page 30 contains a flow chart for the measurement process. The chart shows all of the functions that are associated with the measurement routine and how to access them. **Figure 26** references **Figure 27** for the measurement sequence as shown on page 31.

To start the measurement process, follow the steps below:

1. Select Measurement Mode & Core Diameter Size

a. From the main menu (**Figure 4**), enter into the Setup menu.

b. Select Mode and select the preferred mode of measurement.

c. Select Core diameter then choose the core diameter size. Select "None" if you wish to measure rock samples.

d. Select "Back to menu" to go back to main menu

2. Select Measure

Ensure the meter is on the start measure screen that is displayed in **Figure 21.** Before proceeding with the measurements, ensure that the meter is position in a free air space void of all metallic objects.

Step 1: With the KT-10 v2 in free air, use **SBP▲** to start the measurement process. After about 1 second you will hear a short sound indicating the free air measurements are complete.

Step 2: Immediately place the KT-10 v2 on the sample's surface then use **SBP▲**. When the reading on the sample is complete you will hear a sound; this sound is different then the one heard during the free air measurements.

Step 3: Then immediately position the KT-10 v2 in free air once again for the final free air measurements. Wait for the final sound, which will be the same as the first tone heard in **Step 1**. This sound will indicate the final free air measurements are complete and the reading(s) will be displayed on screen.

Note: A button icon will appear in the notification area confirming a button press.

Tip: To repeat the measurement process without saving the results, position the unit in free air and go directly to **Step 1**.

With PIN

PIN Installation:

Remove the thread protection screw from head of the KT-10 v2. Install the PIN in the place of the thread protection screw. Ensure that the PIN is threaded all the way into the housing. **Remember to enable the PIN mode in the Setup menu.**

Step 1: With the KT-10 v2 in Free Air, use **SBP▲** to start the measurement process. After about 1 second you will hear a short sound indicating the free air measurements are complete.

Step 2: Immediately place the KT-10 v2 on the sample's surface keeping the coil parallel to the sample then use **SBP▲**. When the reading on the sample is complete you will hear a sound; this sound is different then the one heard during the free air measurements.

Step 3: Then immediately position the KT-10 v2 in free air once again for the final free air measurements. Wait for the final sound, which will be the same as the first tone heard in **Step 1**. This sound will indicate the final free air measurements are complete and the reading(s) will be displayed on screen.

3.4.2 Store a reading

To store the reading, there are two different options available; quick save and save with a voice note.

Quick Save: With the results displayed on screen, you can quickly store the reading by using **LBP▲**.

Save with voice note: To store the reading with an optional voice record use **LBP▼**. This will invoke the voice recorder and the screen shown in **Figure 22** will be displayed. Position the KT-10 v2 about 10 cm from your mouth and speak at a normal volume. You can end the voice record by using either **SBP▼** or **SBP▲** or, by allowing the time to elapse (45 seconds).

Figure 22: Voice Record

Once the recording has ended, it will be replayed if the KT-10 v2 has been enabled to do so; this will allow its contents to be confirmed. User can then store or discard the voice note.

Figure 23: Store voice record

When a reading is stored, by either method, a confirmation will be displayed. It will show the record number, date and time along with any GPS positions available (if the optional Bluetooth GPS is enabled).

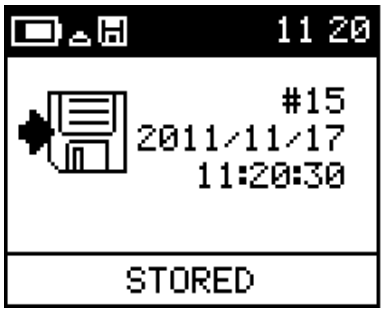


Figure 24: Record Saved

3.4.3 Measurement Sub Menu

The measurement menu can be accessed with **SBP▼** only when the results are displayed on the screen (shown in **Figure 25)**. This menu allows for storing of the reading, storing of the reading with a voice record, returning to the measure routine, disabling the GPS positions when GPS is enabled, clearing of the average buffer or returning to the main menu.

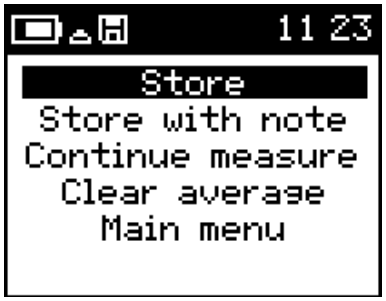


Figure 25: Measure Menu

To store the reading, navigate to the **Store** option with the use of **SBP▼** or **SBP▲** and select it by using **LBP▼** or **LBP▲** when it is highlighted.

To store the reading with a voice record, navigate to the **Store with note** option with the use of **SBP▼** or **SBP▲** and select it by using **LBP▼** or **LBP▲** when it is highlighted.

Selecting **Continue measure** will return the display to the results page. This is accomplished with the use of **SBP▼** or **SBP▲** to highlight the selection and use **LBP▼** or **LBP▲** to select it.

Disable GPS will remove the GPS positions from the data set. However, the meter will remain paired to the Bluetooth GPS. Highlight this option by moving the cursor with **SBP▼** or **SBP▲** and once highlighted select it with **LBP▼** or **LBP▲**. When you return to the measurement menu after you **Disable GPS,** the menu will show **Enable GPS.** Selecting Enable GPS will enable the GPS positions in the data again.

Clear average is selected to clear the averaging buffer. Each reading that has been saved is used to calculate the average and standard deviation of the stored readings. The averaged values and standard deviations are stored along with the readings for later retrieval. Clearing the average will enable the user to select which set of readings will be averaged together.

3.4.4 Measure Flow Chart

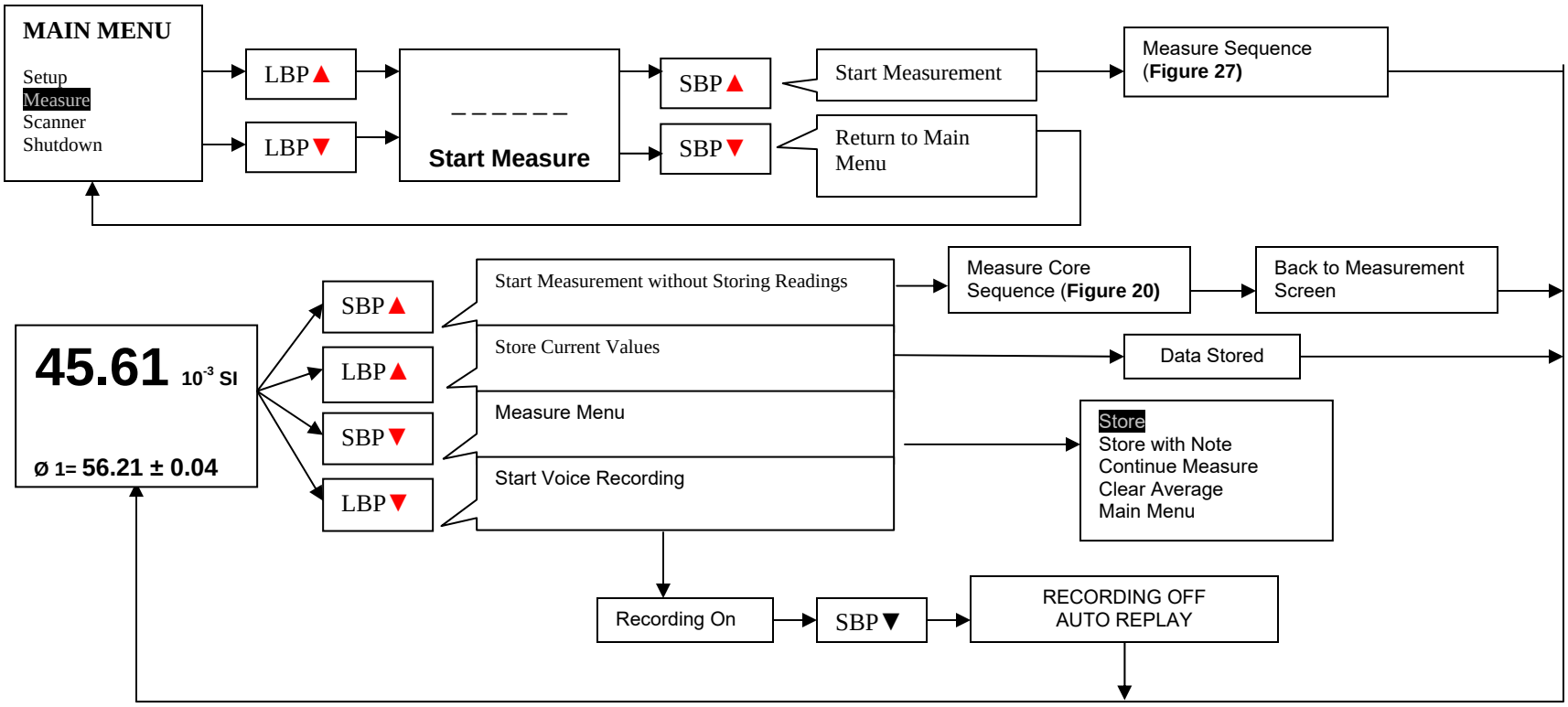
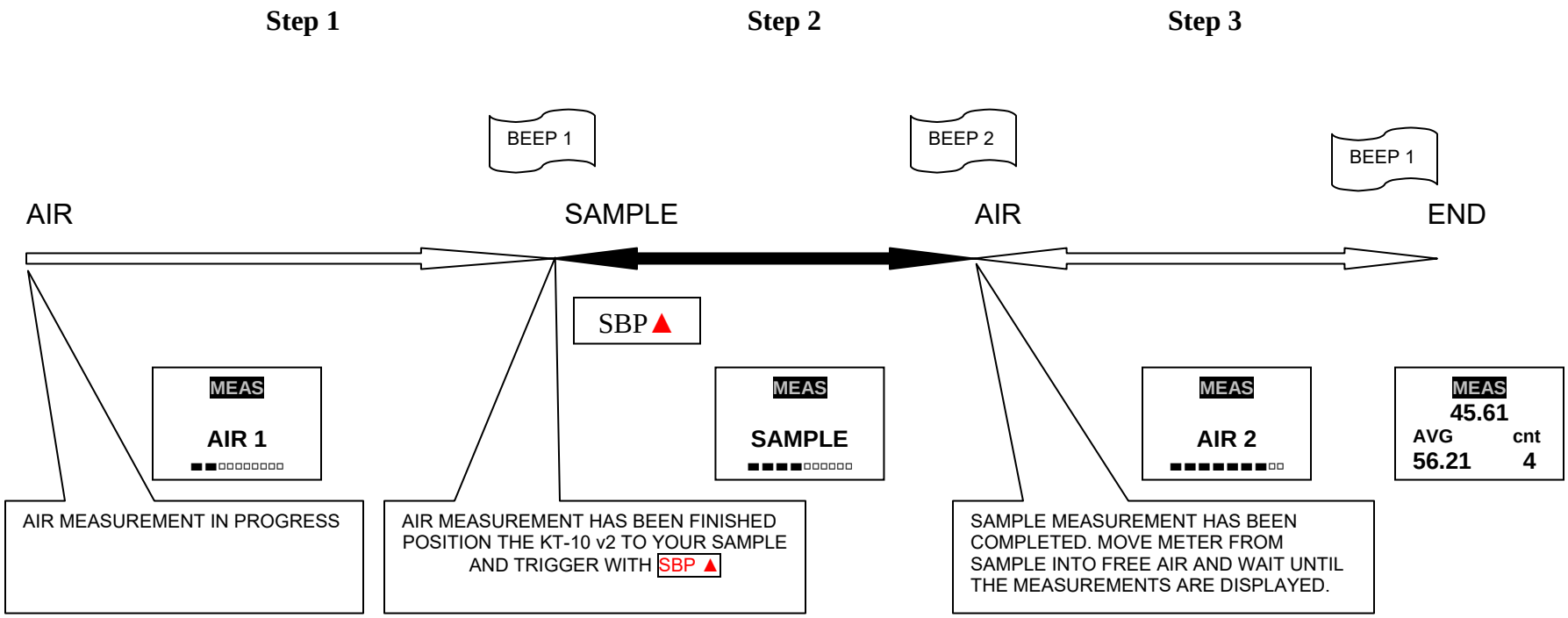


Figure 26: Measurement Flow

3.4.5 Measure Sequence



Please note: MAXIMUM OF 7 SECONDS BEFORE TIMEOUT ERROR WILL BE SEEN ON SCREEN.
Measurement sample must be started before this time has elapsed

Figure 27: Measurement Sequence

3.5 Scanner

The **Scanner** option will initialize the KT-10 v2 for continuous measurement. Geometric corrections can be applied to the magnetic susceptibility readings to display true susceptibility values. This mode is best utilized for logging drill cores or prospecting. In Scanner mode, the **SBP▲** is used to activate the **Scanner** measurement sequence and to add markers to the data set. **SBP▼** is used to end the scanner process.

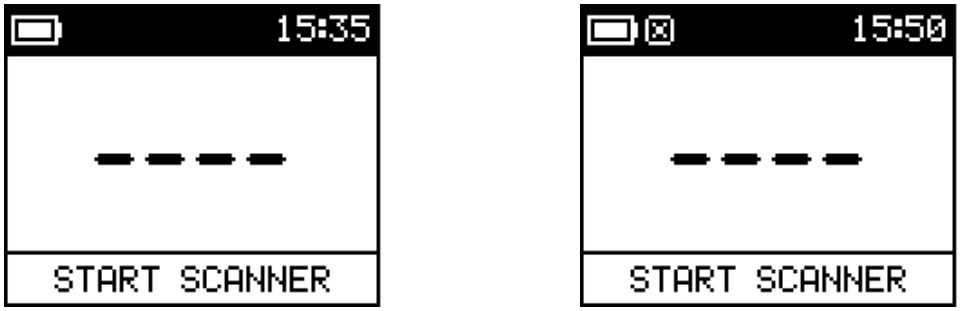
3.5.1 Take a reading in the Scanner mode

1. Set Measure Mode & Core Diameter Size

a. From the main menu (**Figure 4**), enter into the Setup menu.
b. Select Mode and select the preferred mode of measurement.
c. Select Core diameter then choose a core diameter size. Select "None" if you are measuring rock samples.
d. Select "Back to menu" to return to the main menu

2. Select Scanner

Ensure the meter is on the start scanner screen that is displayed in **Figure 28.** Before proceeding with the measurements, ensure that the meter is positioned in free air, void of all metallic objects.



No Core Correction with Core Correction

Figure 28: Start Scanner

This icon in the notification area indicates that a core diameter has been selected for susceptibility measurements. Conductivity measurements are not corrected for core size.

The KT-10 v2 is now ready to start the measurement process in **Scanner** mode.

TIP: **Figure 33** on page 36 contains a flow chart for the **Scanner** mode; it shows the functions associated with the **Scanner** routine and how to access them. **Figure 33** references **Figure 34** for the **Scanner** sequence and can be seen on the page 37.

There are two steps involved in the **Scanner** process. The first step is a free air measurement; the second is the sample measurement which will last for 120 seconds unless stopped with the use of **SBP▼**.

To start the measurement process follow the steps listed below. Ensure your KT-10 v2 is on the screen presented in **Figure 28** and is positioned in free space void of all metallic objects first.

Step 1: With the KT-10 v2 in free air, use **SBP▲** to start the **Scanner** process. Soon after you will hear a short sound indicating the free air measurements are complete and that the meter can be positioned on the sample.

Step 2: Begin to move the KT-10 v2 along the surface you wish to measure. The meter's loud speaker will indicate the relative intensity of the reading by the pitch of the audio. Place a marker in the data set with **SBP▲**. Use **SBP▼** at any time during the scanning process to end scanning.

Markers are special symbols that are added to the scanner data stream while scanned values are being stored to the memory. These can be used for correlation of the recorded samples which may help to synchronize measured values with important positions of anomalies in your samples. Markers can be used as often or as sparingly as needed.

To repeat the **Scanner** process without saving the results, position the unit in free air and go directly to **Step 1**.

3.5.2 Store a reading

There are two different options available to store the readings.

Quick save: With the results displayed on screen, you can quickly store the reading by using **LBP▲**.

Save with voice note: To store the reading with an optional voice record use **LBP▼**. This will invoke the voice recorder and the screen shown in **Figure 29** will be displayed. Position the KT-10 v2 about 10 cm from your mouth and speak at a normal volume. You can end the voice record by using either **SBP▼** or **SBP▲** or, by allowing the time to elapse (45 seconds).

Figure 29: Voice Record

Once the recording has ended, it will automatically be replayed; this will allow its contents to be confirmed. User can then store or discard the voice note.

Figure 30: Store voice record

When a reading is stored, by either method, a confirmation will be displayed. It will show the record number, date and time along with any GPS positions available (if the optional Bluetooth GPS is enabled).

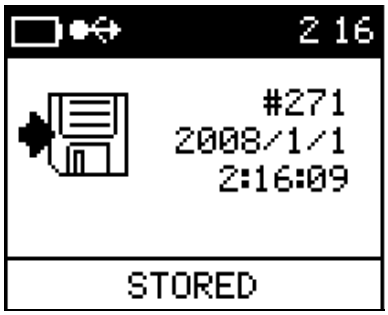


Figure 31: Saved Record

3.5.3 Measurement Sub Menu

The measurement menu can be accessed only when the results are displayed on the screen; this is accomplished by a **SBP▼** as shown in **Figure 32**. This menu allows for the storing of the reading, storing of the reading with a voice note, returning to the measure routine, disabling the GPS (if GPS connected), clearing the average or returning to the main menu.

Figure 32: Scanner Measure Menu

To store the reading, navigate to the **Store** option with the use of **SBP▼** or **SBP▲** and select it by using **LBP▼** or **LBP▲** once it is highlighted.

To store the reading with a voice record, navigate to the **Store with note** option with the use of **SBP▼** or **SBP▲** and select it by using **LBP▼** or **LBP▲** once it is highlighted.

Selecting **Continue scan** will return the display to the results page. This is accomplished with the use of **SBP▼** or **SBP▲** to highlight the selection and a **LBP▼** or **LBP▲** to select it.

Disable GPS will remove the GPS positions from the data set. However, the meter will remain paired to the Bluetooth GPS. Highlight this option by moving the cursor with **SBP▼** or **SBP▲** and once highlighted select it with **LBP▼** or **LBP▲**. When you return to the measurement menu after you **Disable GPS,** the menu will show **Enable GPS.** Selecting Enable GPS will enable the GPS positions in the data again.

3.5.4 Scanner Flow Chart

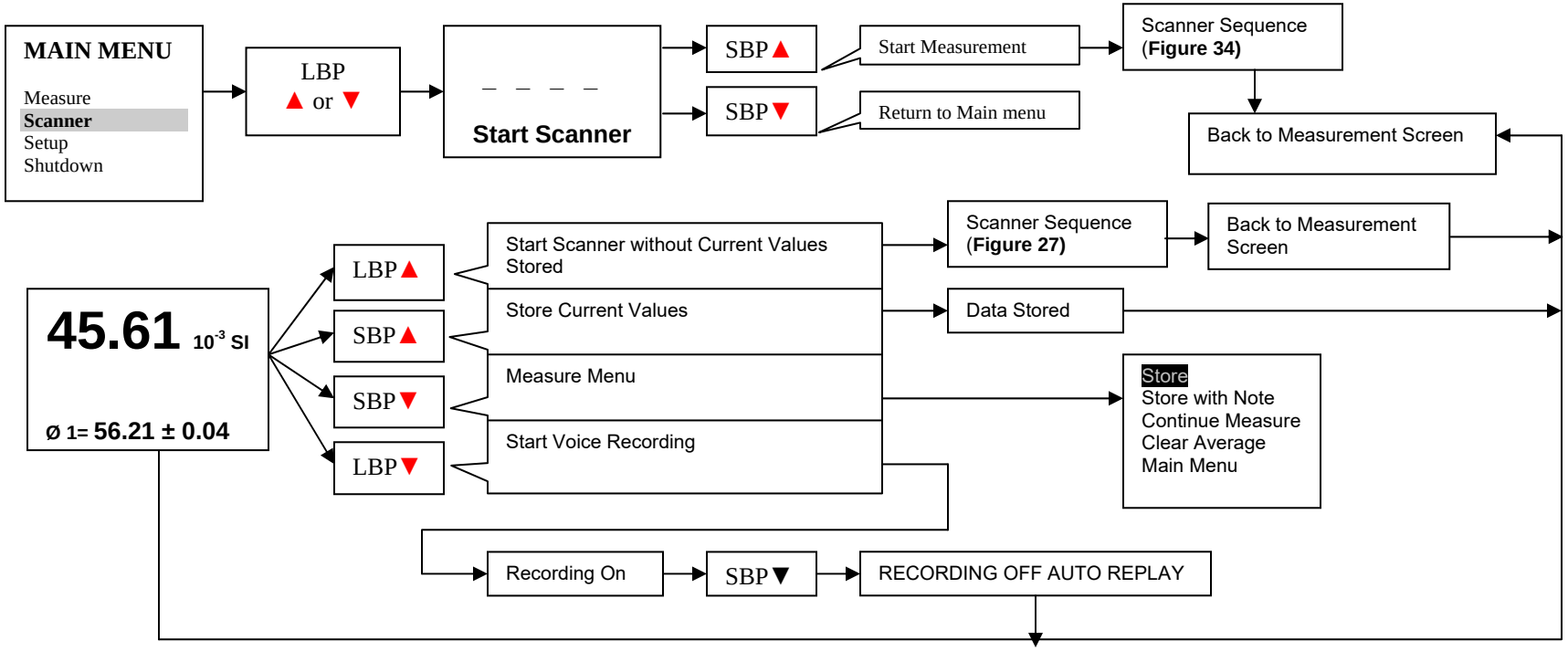


Figure 33: Scanner Measurement Flow

3.5.5 Scanner Sequence

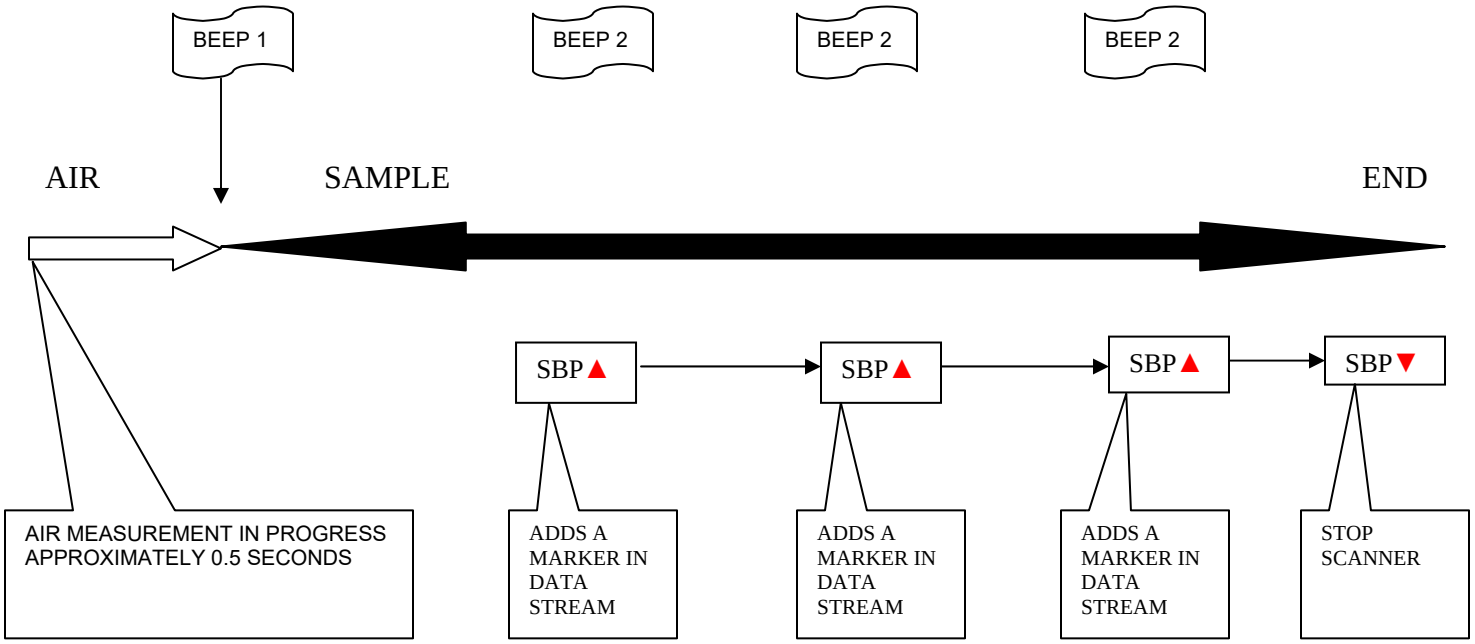


Figure 34: Scanner Measurement Sequence

3.6 Borehole mode

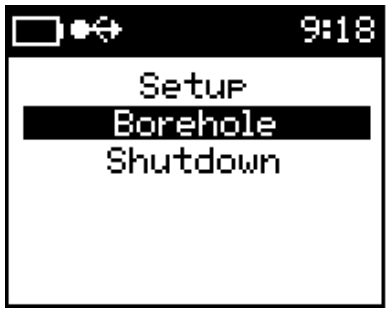
The **Borehole mode** option will initialize the KT-10 v2 for measurements on drill cores along with borehole parameter such as the Borehole ID, Start depth, End depth, Core length, # of Cores per box, and Depth interval. Geometric corrections applied to the magnetic susceptibility and conductivity readings to display true measurement values. In the Borehole mode, the **SBP▲** is used to activate the **Scanner** measurement sequence and to add depth interval to the data set. Depth intervals can be recorded with the push of a button while scanning cores. All readings between depth intervals are interpolated.

3.6.1 Borehole mode configuration

1) Enable Borehole mode

a. From the main menu, select Setup options.
b. Select Borehole mode and then enable it by selecting “On”.
c. Select “Back to menu” to return to the main menu

2) Select Borehole from the main menu



Borehole mode enabled

The options in the Borehole menu are as shown on the following page.

Borehole type

3) Borehole type

New borehole: - used to create a new borehole options

Last borehole: - uses last borehole options; this option is only active when a previously created borehole is not fully completed. For any event the new borehole is not finished, the last borehole is used to resume measurements.

4) New Borehole

Selecting new borehole will start the Borehole creation wizard. Up to six alphas-numerical characters can be used to enter the Borehole options. Use the SBP▼ or **SBP▲** to scroll through the characters and **LBP▼** or **LBP▲** to make selection a selection. The symbol "↵ " is used to proceeded to next in the setup wizard and the symbol " * " is used to exit the setup wizard. Use the " – " for a space or to make corrections.

5) Select measurement type

Borehole measurement type

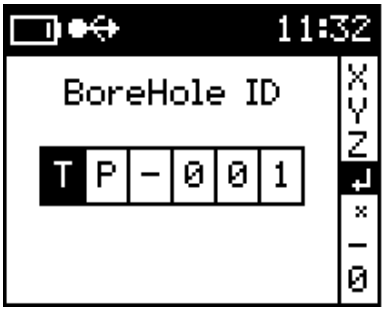
The two measurement modes are the Scanner and Discrete (Measure mode).

3.6.2 Scanner mode setup wizard

Use the **LBP▲** to select the Scanner mode. The Scanner setup wizard will be activated as shown below.

1) BoreHole ID

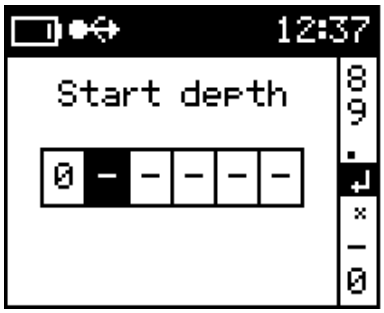
Use the alpha-numeric to enter identification for the borehole



BoreHole ID

2) Start Depth

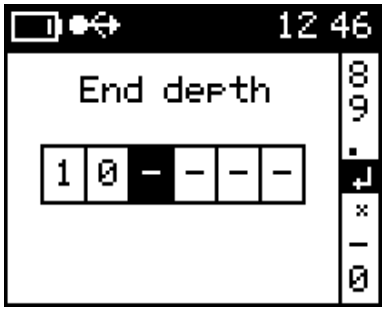
Enter the starting depth of the core



Start Depth

3) End Depth

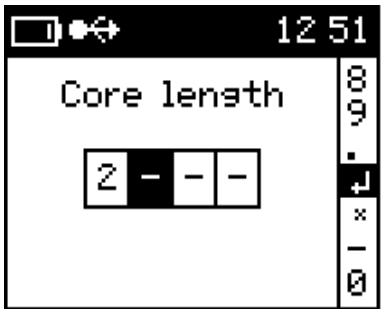
Enter the End depth of the core



End Depth

4) Core Length

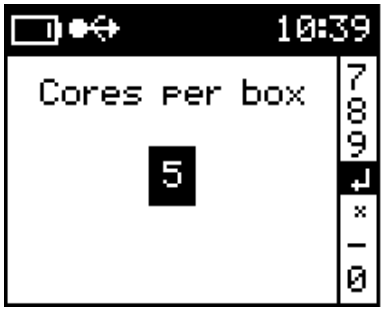
Enter the length of core



Core Length

5) Cores per Box

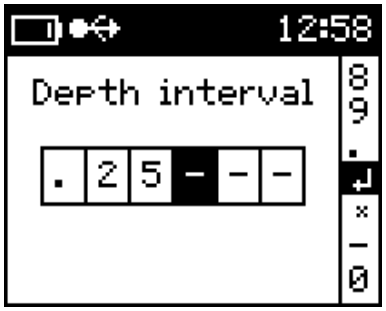
Enter the number of cores that are in a core box



# of Cores in a Box

6) Depth Interval

Enter depth interval for the core



Depth Interval

The Borehole setup wizard is now completed; there will be a brief message on the screen indicating that the new borehole is loaded and does not have any readings recorded for this borehole.

Borehole loaded

The meter is now ready for scanner measurements.

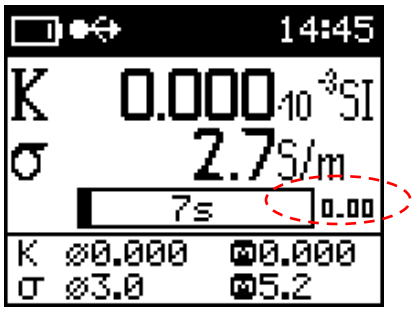
3.6.3 Taking a measurement in the Scanner mode

There are two steps involved in the **Scanner** process. The first step is a free air measurement. The second is the sample measurement which will last for 120 seconds unless stopped with the use of **SBP▼**.

To start the measurement process follow the steps listed below. Ensure your KT-10 v2 is positioned in free air space void of all metallic objects before proceeding with measurement.

Step 1: With the KT-10 v2 in free air, use **SBP▲** to start the **Scanner** process. Soon after you will hear a short sound indicating the free air measurements are complete and that the meter can be positioned on the sample.

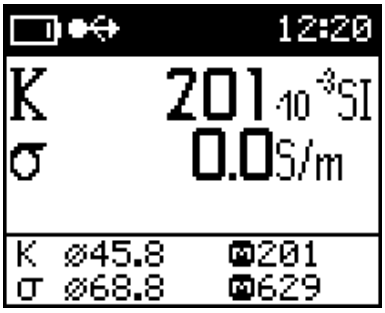
Step 2: When positioned on the sample, press the **SBP▲** to mark the beginning of the Start Depth on the core. This button press will trigger a new display, as shown below in red circle, showing the relative depth on the core. This number will increment with each depth intervals.



Relative Depth display

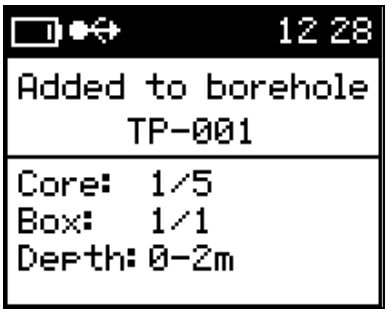
Note: **It is important to have the starting depth marked with the use of the SBP▲ or else the data will not correspond to the depth properly.**

Step 3: Begin to move the KT-10 v2 along the surface you wish to measure. The meter's loud speaker will indicate the relative intensity of the reading by the loudness of the audio. To insert a depth interval in the data set, use **SBP▲**. The scanner will automatically come to a stop when all depth intervals are accounted for given core length as seen below.



Scanner mode stopped

Step 4: Use the **LBP▲** to store the completed measurement for the 1st core. The meter will show the current core status briefly as shown below.



Borehole Status

The borehole status window provides the current status of the borehole measurement. As entered in the Borehole setup wizard, the Borehole ID is TP-00, Start Depth is 0m, End Depth is 10m, Core Length is 2m, # of cores in Box is 5 and Depth interval of 0.25. The status window above shows that 1 of 5 cores are logged for the depth of 0 to 2m as specified in the Borehole setup. This status window will be updated each time a new measurement is saved.

In case of more than one Core box, the after completion of the first core box, the meter will automatically will be configured for the next core box with the appropriate start depth without any intervention from the user.

Step 5: Use the **SBP▲** to continue scanning rest of the cores. When all of the cores have been scanned and saved, a message showing borehole complete will be displayed as shown on the next page.

Borehole completed

It is important to remember that it is not possible to store new reading for a borehole that is already completed and saved. The message shown below will appear when attempting to save new readings for a completed borehole.

Borehole completed – cannot save new record

3.6.4 Discrete mode setup wizard

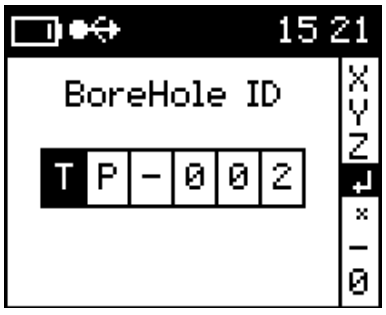
This mode is useful to those how only wishes to obtain single measurement at fixed depth on borehole cores. The sequence for the discrete mode remains the same as described in the **section 3.4.1** for the Measure mode. What is new for the discrete mode is the added benefit of collecting depth information to each stored readings. The operator will be able to associate the Borehole ID and the Depth interval to each new reading collected. In this mode, the depth interval is used as constant for incrementing depth for each successive reading.

If a **New borehole** is not already been selected, use **SBP▼** or **SBP▲** to highlight this mode in the borehole options and select it with the use of **LBP▼** or **LBP▲**.

Use **SBP▼** or **SBP▲** to highlight the **Discrete** mode and then use **LBP▼** or **LBP▲** to select it. A new borehole setup wizard will be activated as shown below.

1) Borehole ID

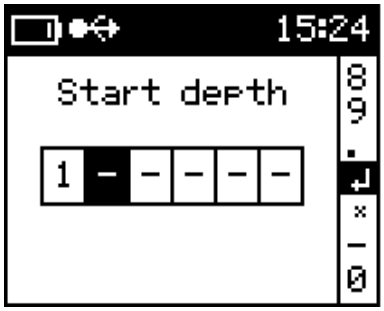
Enter the borehole identification



Borehole ID – Discrete mode

2) Start Depth

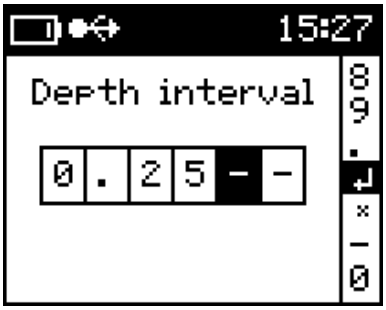
Enter the start depth of the core



Start measure

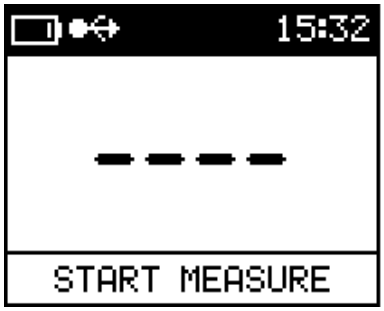
3) Depth Interval

Enter the depth interval as desired



Depth Interval

3.6.5 Taking a measurement in Discrete mode



Start Measure – Discrete mode

After completion of the borehole setup wizard, the screen above will be displayed indicating that the meter is ready to start the measurement process.

As described in the **Section 3.4.1,** there are three steps involved in the measurement sequence: the first step is a free air measurement; the second is the sample measurement; and the final step is another free air measurement.

Note: The duration for the measurement sequence is 7 seconds long. Soon after the measurement is initiated, a new screen with 4 dashes and a progress bar will appear. It takes 7 seconds for the progress bar to complete. It is important that you do not wait for the bar to build up in between each of the 3 steps or else you will be presented with "Error" on the screen.

To start the measurement process, follow the steps below:

Before proceeding with the measurements, ensure that the meter is positioned in a free air space void of all metallic objects.

Step 1: With the KT-10 v2 in free air, use **SBP▲** to start the measurement process. After about 1 second you will hear a short sound indicating the free air measurements are complete.

Step 2: Immediately place the KT-10 v2 on the sample's surface then use **SBP▲**. When the reading on the sample is complete you will hear a sound; this sound is different than the one heard during the free air measurements.

Step 3: Immediately position the KT-10 v2 in free air once again for the final free air measurements. Wait for the final sound that will be the same as the first tone heard in **Step 1**. This sound will indicate the final free air measurements are complete and the reading(s) will be displayed on screen.

Step 4: To quickly store the reading, use the **LBP▲**. Alternately press the **LBP▼** to bring up the measurement sub menu. The current measurement will be stored with the Start depth.

Note: The depth will increment automatically for the interval depth specified when the measurement is stored.

3.7 Voice Recorder

The voice recorder can be accessed after a measurement has been completed from any mode. To access this option, you must use the **SBP▼** which will bring you into the measurement menu (**Figure 25** on **page 29)**. Select **Store with note** and the voice recorder will begin immediately. For best recording, the KT-10 v2 should be positioned approximately 10 centimetres from the operator's mouth, and a normal speaking volume should be used. Speaking loudly will only cause distortion in the recording, which may make it difficult to understand when the file is played back. Using **SBP▼** will end the voice recording.

Note: voice notes are currently limited to 45 seconds per recording.

The voice recorder can add complimentary information to a set of core measurements or field measurements. For example, the voice recorder will allow an operator to indicate which borehole a reading came from as well as any other location information (if the optional GPS is not connected). Information such as the physical characteristics of a rock, sample interval on a drill core and the number of boxes to complete the borehole could also be added here.

All voice records are saved and transferred to a personal computer in wave format and can be replayed in the GeoView via audio program such as Windows Media Player.

3.8 PIN

The KT-10 v2 is equipped with a **PIN** for rough surface measurements.

There will be a single PIN located in the carrying case at the time of purchase, located near the back left as seen below (**Figure 35)**.

Figure 35: KT-10 v2 Pouch PIN Storage

Note: A spare PIN can be purchased and carried in the KT-10 v2 Pouch as shown in **Figure 35**.

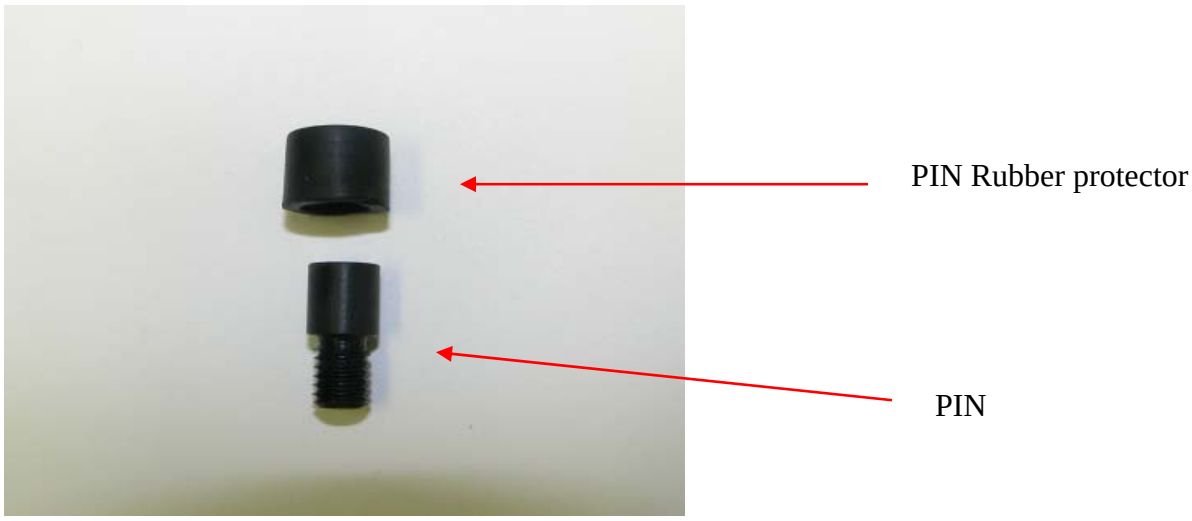


Figure 36: PIN

Remove the thread protector screw from the head of the KT-10 v2.

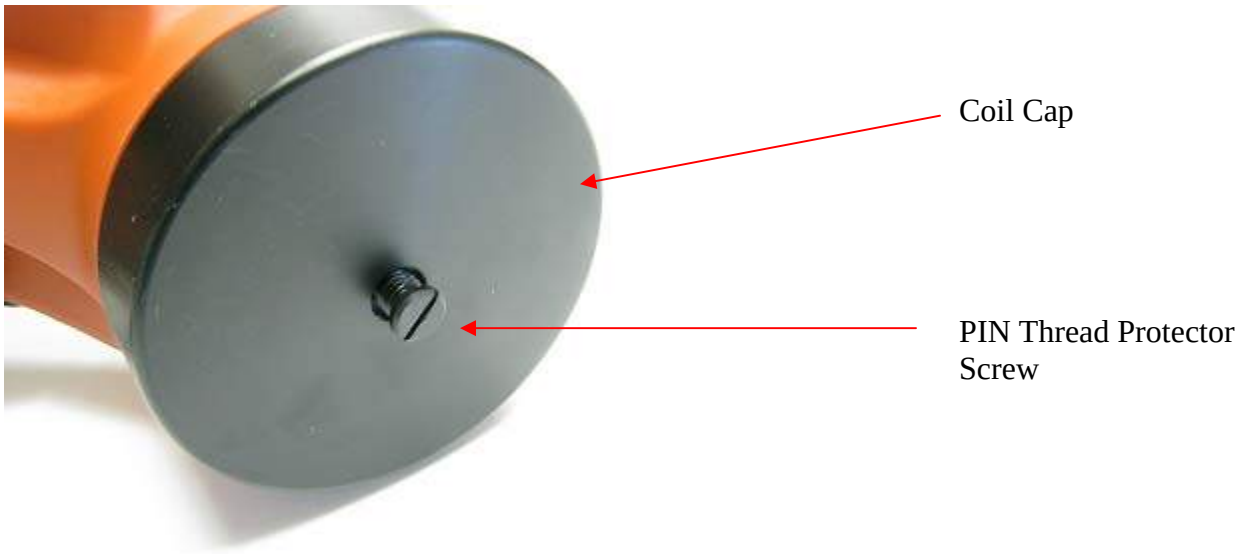


Figure 37: Coil Cap & Thread Protector

Coil Cap and PIN installed

When the thread protector have been removed, insert the KT-10 v2 PIN into the center hole where the thread protector came from, seen in the image on the left.

Chapter 4

Software

4.1 GeoView

GeoView is an easy to use Windows ™ based GUI (Graphical User Interface) which allows for data on a KT-10 v2 to be downloaded, stored and viewed on a Windows PC. It also supports data export functionality which allows the KT-10 v2 data to be converted into an ASCII file format that can then be easily imported into database or spreadsheet software. All data retrieved from the KT-10 v2 are stored in a firebird database, which is an integral part of the Geoview software. The Geoview database can be a local or a networked database, with the added possibility to have several databases existing for separating projects. Voice notes that are recorded and stored on the KT-10 v2's internal memory can be replayed from with-in the software interface. This allows for the voice notes and visual observations that have been recorded from the field to be added and stored along side the readings from the KT-10 v2 for a complete picture.

4.1.1 Installation

To install GeoView locate the install package provided to you on the KT-10 v2 software CD. Please locate GeoView_setup.exe file in the Software\Geoview folder. It will be approximately 21,613 Kilo Bytes in size as seen in **Figure 38**. Double click the icon to run the installation package.

Please note: **You must be an administrator to install the software and you will be prompted for this information if your Windows user account does not have administrator credentials.** **(Seen in Figure 39)**

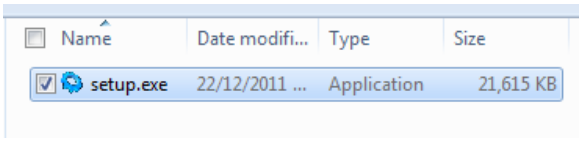


Figure 38: Setup.exe

Figure 39: Administrator credentials

The setup file contains installation packages for the GeoView Software, the firebird database, and the USB drivers for the KT-10 v2. If this is the first time connecting your KT-10 v2 to your PC it is recommended to install the drivers first, and then install GeoView software. To begin installation, select the appropriate option from the menu shown below (**Figure 40**).

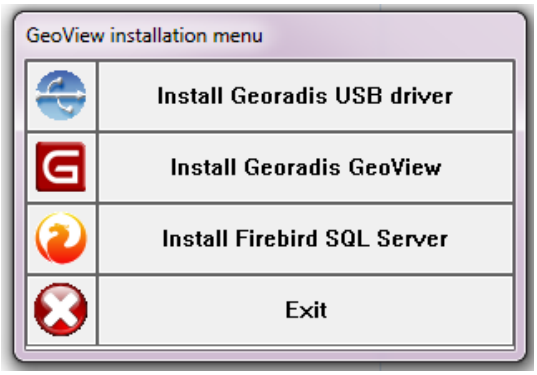


Figure 40: Install Splash Screen

USB Driver Installation

Please ensure that the meter is not connected to the PC via USB prior to driver installation.

When selecting ***Install Georadis USB driver,*** you will be presented with the following screen seen in **Figure 41**, select ***Next>*** to continue the installation or ***Cancel*** to exit.

Figure 41: Start Driver Installation

When the driver install has been completed, you will be presented with screen as seen in **Figure 42**.

Figure 42: Complete Driver Installation

At this time, plug the KT-10 v2's USB cable into the computer to have windows recognize the drivers and copy the files to the appropriate location. Windows will respond by showing you that a new USB device has been recognized. The new hardware wizard will start.

Note: You should be logged in as an administrator to complete this part of the install.

Figure 43: Windows New Hardware Wizard

You will not need to go to the internet, as the previous step has copied the files to your PC already, so select **No, not at this time** and then press **Next** button.

Figure 44: Install Automatically

Select **Install software automatically** and then press **Next>** button.

The drivers will start to be copied to the windows/system32/drivers folder but you may be warned that the drivers have not passed Windows Verification.

Figure 45: Start File Copy

Select **Continue Anyways**

Figure 46: Continue Anyways

The files will then be copied to your PC and you will be able to establish communication between your PC and the KT-10 v2.

Figure 47: Driver Files Copied

GeoView Installation

When selecting ***Install Georadis GeoView,*** you will be presented with the screen as seen in **Figure 48**, select ***Next>*** to continue the installation or ***Cancel*** to exit.

Figure 48: Begin GeoView Installation

You will then be prompted for the location of the installation directory. You may either leave this as default (C:\Program Files\Geoview) or use the browse button to find a new directory to install the GeoView software too. Select ***Next>*** when the correct path has been chosen.

Figure 49: Folder Location

You will then confirm the destination folder by selecting ***Install*** or if you wish to change this location use the ***<Back*** button to make the necessary changes.

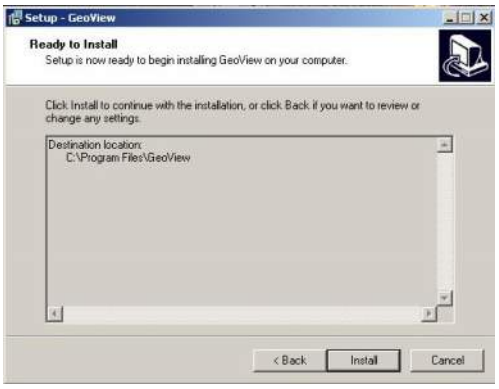


Figure 50: Folder and Installation Confirmation

An installation progress window, seen in **Figure 51**, will then be presented which is followed by a confirmation dialogue, seen in **Figure 52**. Selecting ***Finish*** completes the installation process.

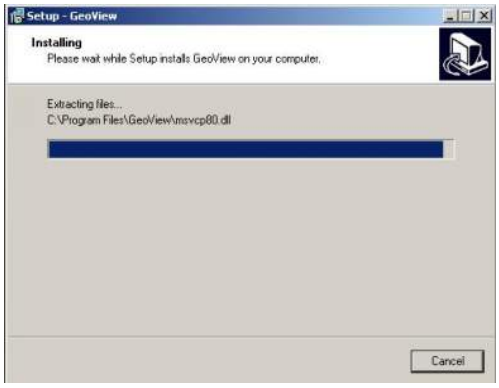


Figure 51: Install progress window

Figure 52: GeoView Installation Complete

4.1.2 GeoView Calendar Interface

To start using GeoView, double click on the software's icon which can be seen in **Figure 53**. When GeoView is first opened, a calendar is presented to the user, seen in **Figure 54**. This allows for the KT-10 v2 data to be organized by the date it was collected on, which allows for the quick retrieval of data from previous recordings.

Figure 53: GeoView Icon

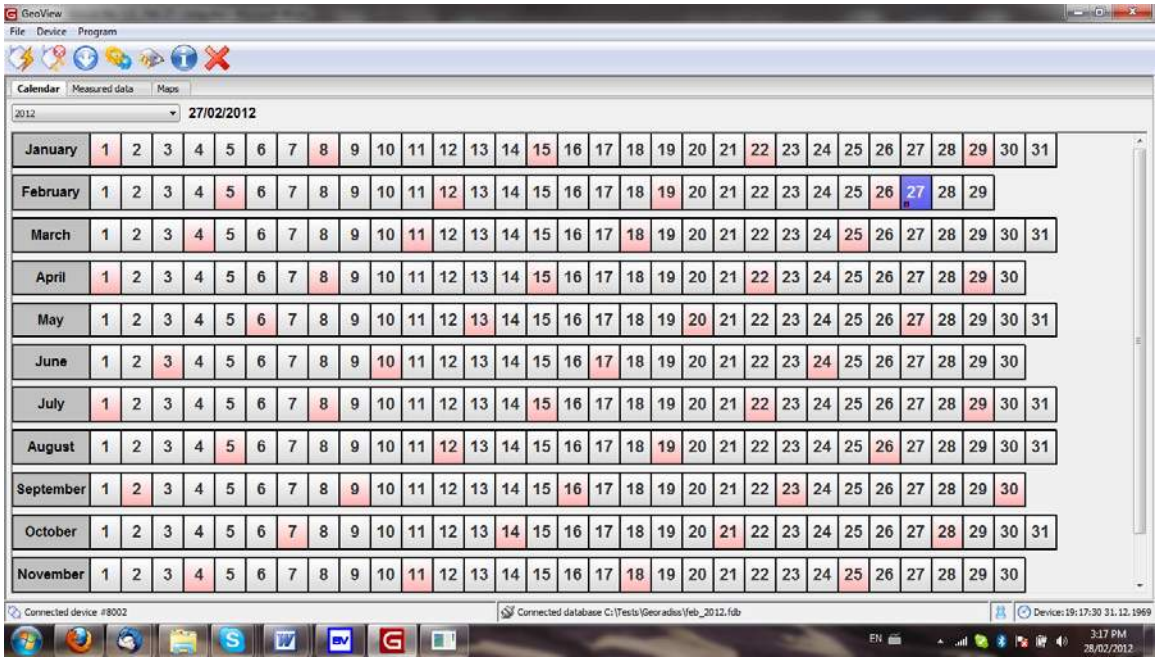


Figure 54: Calendar Interface

Let us get acquainted with the GeoView calendar interface. As is the case with most Windows software programs, there are menus located at the top row of the main window. On GeoView these are labelled ***File***, ***Device*** and ***Program***. Under each menu are several selections, which are outlined below in detail. Some of these selections that are used most often also have icons for quick access to the function. These are located directly below the menus and the associated icons are included below with the description of the selection.

Under the ***File menu*** you will find the following selections: ***Create local database, Open local database, Create remote database, Open remote database*** and ***Exit.*** The function of each selection is listed below.

Create local database: - This allows for the creation of a local database to store KT-10 v2 data into. The location and the name of the database are user definable but must be a location on your personal computer. One can use the default database name (.FBD) or choose a name for the database. Seen in **Figure 55** is the dialog for this task. Enter the desired name and then press the **Save** button when complete.

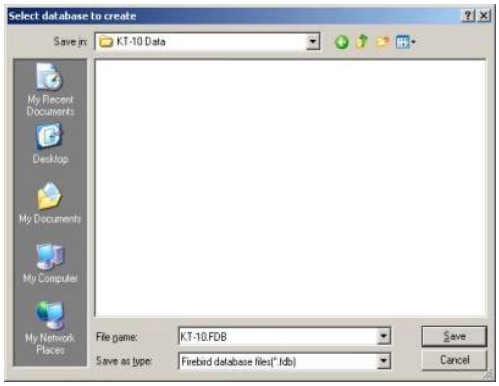


Figure 55: Create Local Database

Open local database: - This will open an already created local database which contains data from one or many KT-10 v2 units. Select the database file that you wish to work with and then select open.

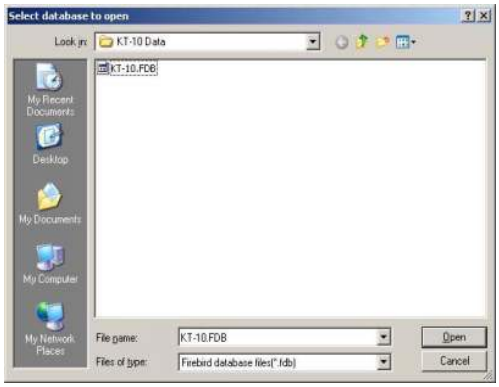


Figure 56: Open Local Database

Create remote database: - This allows for the creation of a remote database to store KT-10 v2 data into. The location and the name of the database are user definable and the

location can be on a server or networked PC. Enter the path and server name into the following box as seen in **Figure 57** and then press create when complete.

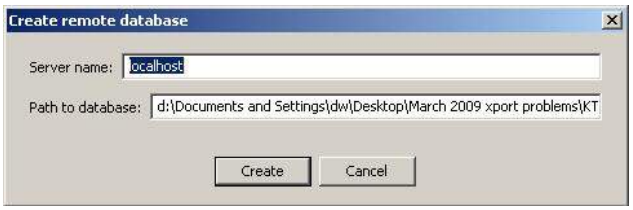


Figure 57: Create Remote database

Please note: The Firebird Super Server will have to be installed and setup correctly on the Server or networked PC which will hold the remote database. This must be completed for remote database option to work correctly, for further details please contact Terraplus Technical Support for assistance. Contact information can be found on the last page of this manual.

Open remote database: - This will open an already created remote database which contains data from one or many KT-10 v2 units. Fill in the server name, path to the database file and then press the open button to connect to the database.

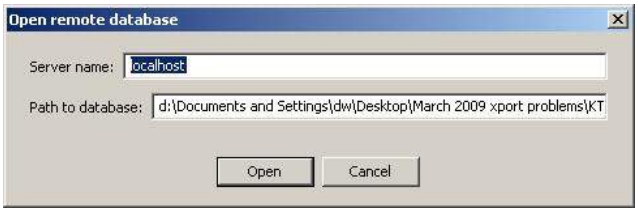


Figure 58: Open Remote Database

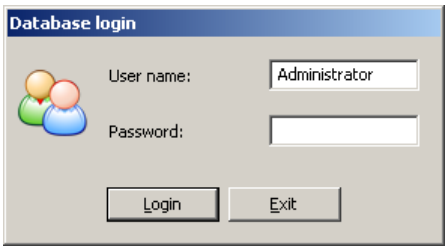


Figure 59: Remote Database Login

Exit: - Will **e**xit the program. You will be prompted to confirm this selection seen in **Figure 60.**

Figure 60: Exit Dialog

Under the **Device** menu you will find: ***Connect device***, ***Disconnect device***, ***Download data***, and ***Device settings***. Each of the functions is listed below.

Connect device: **-** Connect your Personal Computer to a KT-10 v2 with a USB cable or via Bluetooth connection. There is also an icon for quick access to this function and it can be seen below

Connect to Device Icon

Disconnect device: **-** Disconnect your Personal Computer from a KT-10 v2's USB or Bluetooth connection, icon seen below.

Disconnect from Device Icon

Download data: **-** Begin the process to download data from a connected KT-10 v2, icon seen below. Further details on this can be found in section **4.1.4 Data download** on **Page 67**.

Download data Icon

Device settings: - Open the device settings window to make changes to KT-10 v2 operations, icon seen below. More details on device settings can be found in **Section 4.1.7** on **Page 76**.

Device Settings Icon

Under the **Program** menu you will find ***Options*** and ***About***, details for each selection can be found below.

Options: - This selection allows for additional fields to be added to the database. These new fields will be stored along with your KT-10 v2 readings. More details on this option are further explained starting on **page 71**. There is an icon on the main window for quick access to this function and it can be seen below.

Program Options Icon

About: - This shows program version, database version and is the location for performing a GeoView software upgrade. There is also a quick link icon to this feature and it can be seen below.

About Geoview Icon

Directly below the quick link icons you will see a pull down menu with a date beside it. This can be seen below in **Figure 60**. This allows you to switch between different years in the database. You will also notice two tabs, one labelled **Calendar** and the other labelled **Measured data** which are directly above the year. This will change the interface between the calendar view and the data view.

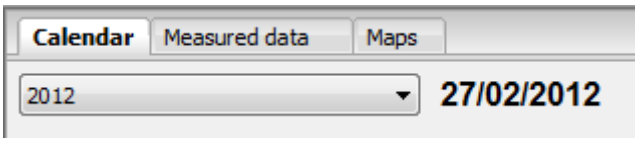


Figure 60: View Selection

Down at the bottom of the window you will also notice additional information being presented. This shows details on device and database connections along with system or device date and time and user credentials for database login. **Figure 61** shows an example of this portion of the screen.

Figure 61: Connections Status

To start adding data to the Geoview software, a database must be created for the storage of the KT-10 v2 data. This is accomplished by navigating to the file menu seen in **Figure 62** and then selecting either ***Create local database*** or ***Create remote database*****. (Most users will want to use the local database option)**

Figure 62: File Menu

A Windows file browser window will open, seen in **Figure 63**. Select the location and file name for the database file. Select **Save** when finished.

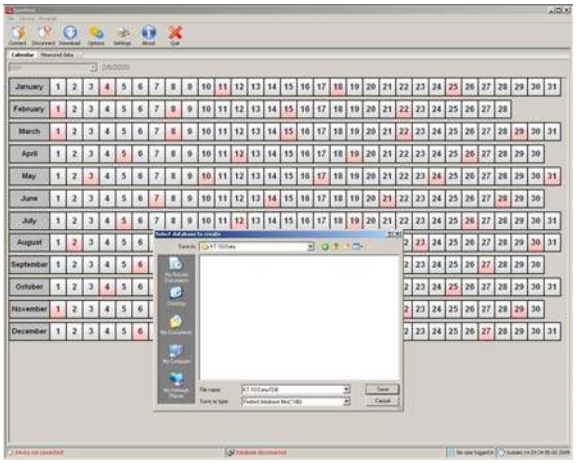


Figure 63: Database Creation Dialogue

You will notice at the bottom of the main window that a database connection is now shown.

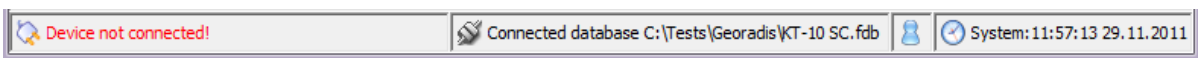


Figure 64: Database Connection Updated

4.1.3 GeoView Data Interface

Switching to data view changes the bottom three quarters of the screen only, with all icons and menus still available while in the data view or calendar view. To switch back to the calendar view, select the **calendar** tab directly above the displayed date. Below this you will see the data view window and we will now get familiar with this part of the interface.

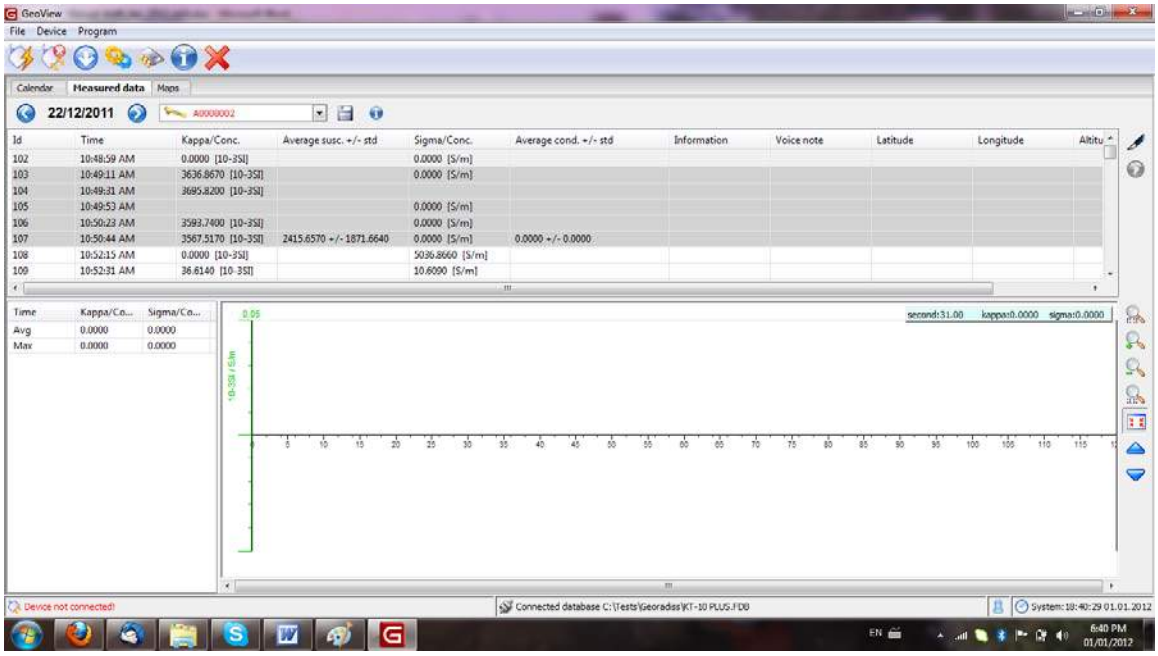


Figure 65: GeoView Data View

Directly below the **Measure data** and **Calendar** tabs is where you will start to notice the changes to the interface. The displayed date has two buttons on either side of it. These two buttons allow for you to move forwards and backwards through the calendar while remaining in the data view display. **Please note:** Only days that contain data for the serial number shown will be viewable. To the right of the date you will see a pull down menu which contains a serial number list of unit's with data being displayed. If you have data from other units on the same day this is where you would change between the different units. Pull the menu down with a left click of the mouse and all serial numbers that contain data for that day will be displayed. Select the serial number you wish to view the data for, with a left click of the mouse.

Following the unit serial numbers drop down box is an icon which is used to export data from GeoView to an ASCII format for use in another database system or spread sheet program.

4.1.4 Data Download

Once you have the database created and connected, you can then download data from your KT-10 v2 and store it on your PC. To accomplish this, connect your KT-10 v2 to your PC via the USB cable provided or by performing a Bluetooth pairing between your PC and KT-10 v2 unit. Use the Device menu and select **Connect device** or press the icon to connect the KT-10 v2 to the GeoView software. A window will open prompting you to select the method and serial number for the unit you are connecting to.

Figure 66: Connection dialog - USB & Bluetooth

The tabs at the top of the window are used to select either a USB connection or Bluetooth connection. The unit you have connected to your Personal Computer will show up in the list. In the case of a Bluetooth connection, you may have to use the discover button to bring the serial number up. Select a serial number from the list with a left click of the mouse, which will cause the listing to be highlighted, then press the connect button at the bottom of the window.

Once the KT-10 v2 is connected you will notice the bottom of the screen will be updated with the KT-10 v2 unit's serial number, as seen in **Figure 67**.

Figure 67: KT-10 v2 connection Updated

You may then proceed by pressing the download button icon  or use the **Device** menu and select **Download data**. When the download process starts the following window shown in **Figure 68** will be presented.

Figure 68: Synchronising with Device

GeoView will synchronize with the KT-10 v2 to determine how much data is on the device that is also not currently in the GeoView database. At this point the data will be downloaded and the dialogue box display will show the details of the operation. The display will change depending on which type of readings is being downloaded. The **record # / total # of records** will be presented when data is being downloaded and **Downloading note** # will be displayed when downloading a voice record. The Unique record ID will be used for referencing voice notes to the recorded readings.

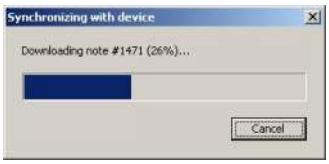


Figure 57: Downloading voice note

Figure 58: Downloading Records

When all the data has been downloaded from your unit you will be presented with a small window confirming data transfer is complete.

Figure 69: Download Complete

When data is loaded into the GeoView database, the day in which data is stored on will change its color to indicate that data is present for that day. When the cursor is over that day an icon will be shown for the instrument and the serial number will also be present. If multiple instruments are used on the same day these are also separated and a list of serial numbers will be presented on that day.

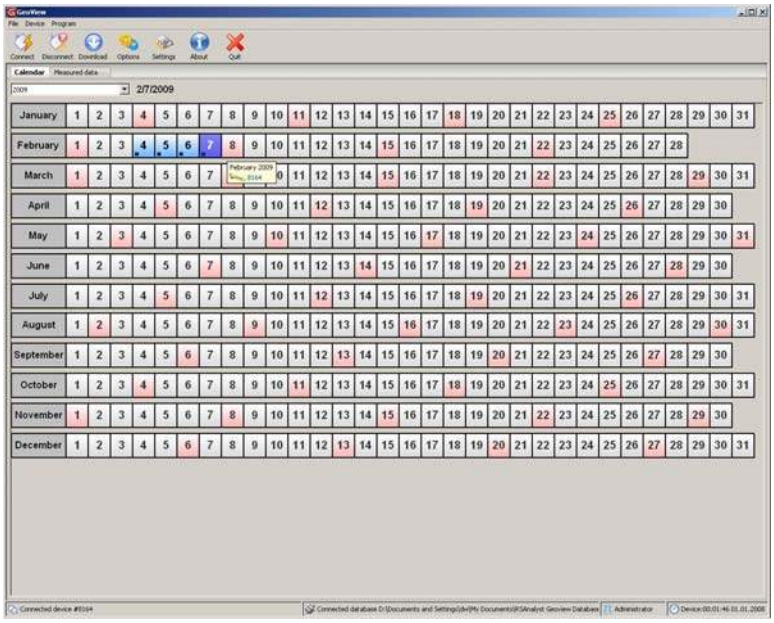


Figure 70: Calendar with Data Populated

To view the data on any of the days, double click on the day or click once on the day and select the M**easure data** tab.

4.1.5 Data Export

When the icon is pressed, the window of **Figure 71** will open. This dialogue is used to select the data that is to be exported over a definable period of time and to which location.

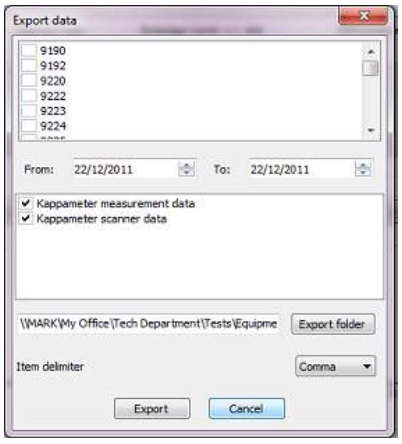


Figure 71: Data Export

A list of units in the database will be presented with a check box beside each unit serial number, populate the box for each unit the data is to be exported from. Then select a date range, if desired, and choose the folder location for the data to be exported too. Make sure to select your preferred delimiter. When selecting the export folder button a windows explorer window will open to choose the location, seen in **Figure 72**. Select a location and then press **OK** then the **Export** button to complete the task.

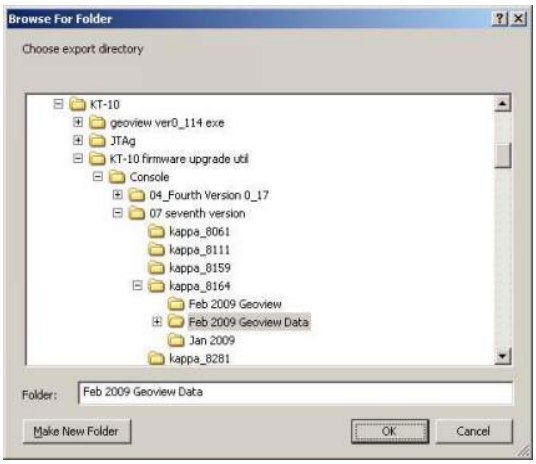


Figure 72: Data Export Location Window

The final icon on this row is used to add notes or information to the day. These will be displayed directly to the right of this icon when populated.

Over on the right hand side of the window you will notice two more icons. This icon is used to edit the custom fields in the database for the record that has been highlighted in the display. This icon is used to play the voice notes associated with a data record.

Please note: This icon my not appear until a record with a voice note is available in the database and will remain " greyed out " until voice records are available in the database.

The upper portion of the data view window shows the data in numerical format which contains column headers to define what each value represents. **Measure** and Scanner data will be displayed in this manner and will have the following fields by default.

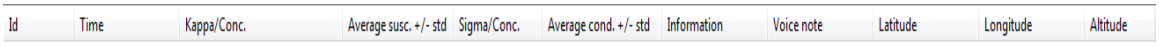


Figure 73: Default Database Field Names

Custom fields can be added to the database which will be appended to the end of each reading this information will also be exported along with the stored KT-10 v2 data. There are three types of data that can be added to the database and these are: Integer numbers, Real numbers and String data. Each data type has a name and a description associated with it and the user must define these when setting up the custom fields. With the numerical data you also have the ability to add a range of acceptable values, with values outside this range being rejected at entry. To add custom fields to the database select the **options** under program menu or select the icon. This will bring up a dialog box for adding fields to the database, which can be seen below.

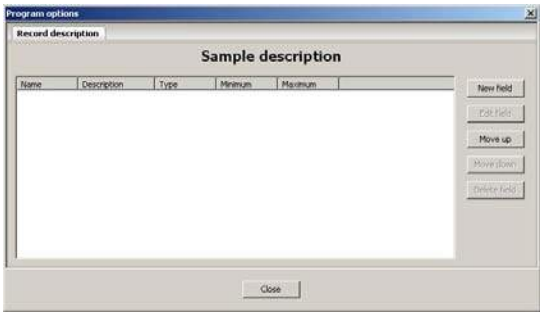


Figure 74: Enter New Field into Database

This dialog will list all fields that have been added to the database. To enter a new field select the New field button on the right of the window. This will open another window to select the type and particulars to the field, seen in **Figure 75.**

Figure 75: Field type Selection

Select the field type from the pull down list and select next. The next dialogue window will allow for you to enter the particulars for the field depending on the type selection. The different windows can be seen in **Figures 76, 77, and 78**.

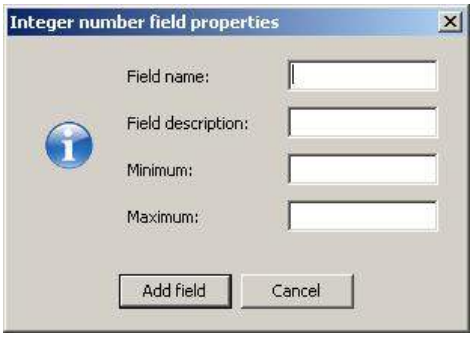


Figure 76: Integer field setup

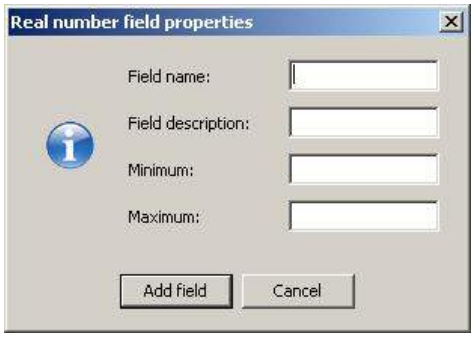


Figure 77: Real number field setup

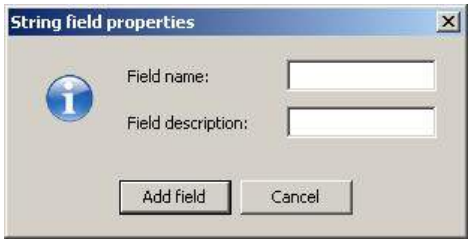


Figure 78: String Setup

Give the field a name and brief description, for numerical values provide the range of valid entries. When complete, select the **Add field** button at the bottom to complete the task. Repeat these steps for each new field that is to be added to the database.

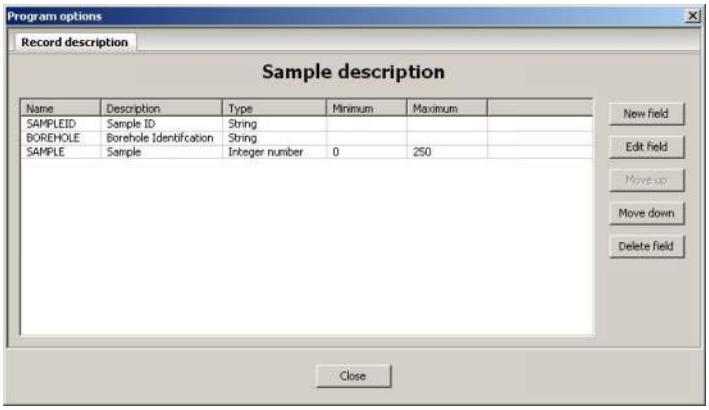


Figure 79: Custom Fields added to GeoView Databae

Once you have added the fields into the **Program options** window, press the close button at the bottom and the database will be updated with the new fields. The new fields will be appended to the end of the default columns. This can be seen in **Figure 80.**

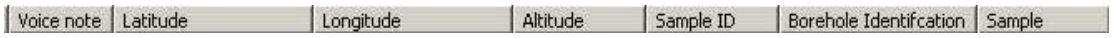


Figure 80: New fields added to the end of the columns

The custom fields can also have their location in the database listing moved but they will always be at the end of the default columns. Use the **Move up** and **Move down** to change the location of the fields in the database. **Edit field** will allow you to change the description and number range for a field. The field name cannot be changed when stored. To remove fields use **Delete field**, a confirmation window will be presented to confirm the deletion of the field.

Scanner records will also be listed in the upper numerical display and are blue in color. The values are given in the bottom left hand corner of the screen, only when a scanner record has been highlighted. When selected, the scanner record will also be plotted in the graph window directly to the right of the numerical display. This can be seen on the following page, **Figure 81**.

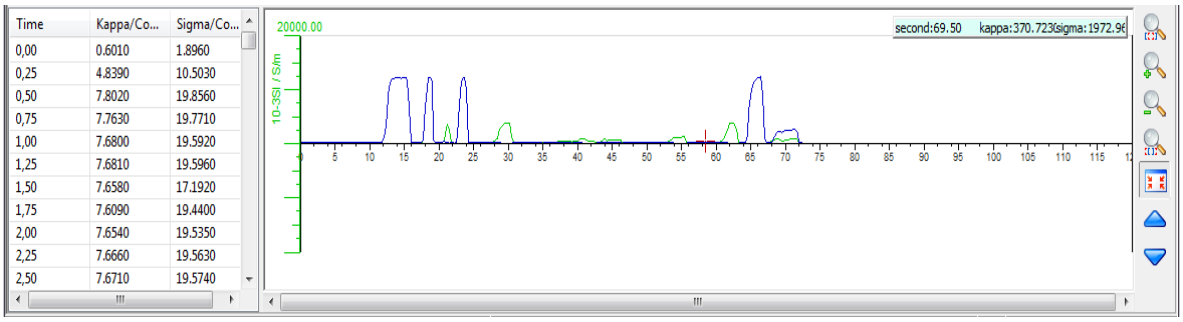


Figure 81: Scanner Data Plotted & Charted

To control the scanner graph use the icons to the right of the graph window, seen in **Figure 81** and **82**. There are four zoom function buttons, and three scale buttons.

The first of the zoom buttons, which is located at the top of the graph control pane, is used to zoom on a selected section. To use this feature click on the icon then navigate your mouse pointer to the section of the graph you wish to zoom in on, left click and hold the button down while dragging the mouse to the end of the section you wish to zoom to. When you release the mouse button the section highlighted will be seen as the entire graph plot. An example of this can be seen below in **Figure 82.**

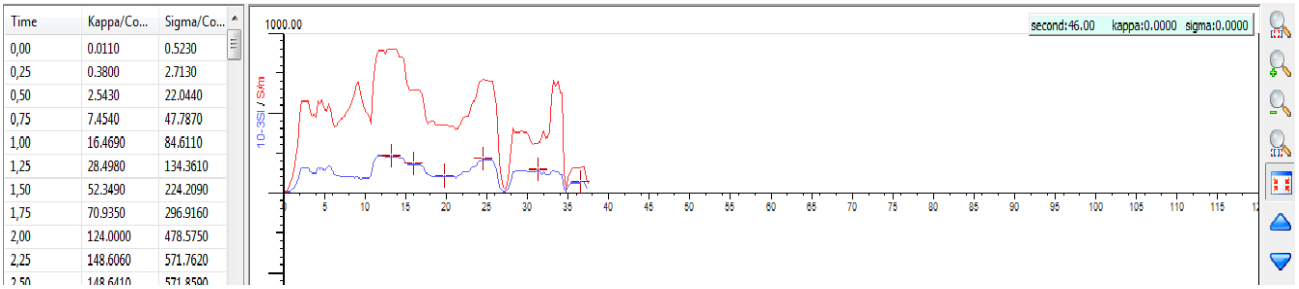


Figure 82: Zooming on Scanner Graph

The next two buttons are for zooming in and out. Left click on the button to increase the zoom level and left click on the button to decrease the zoom level.

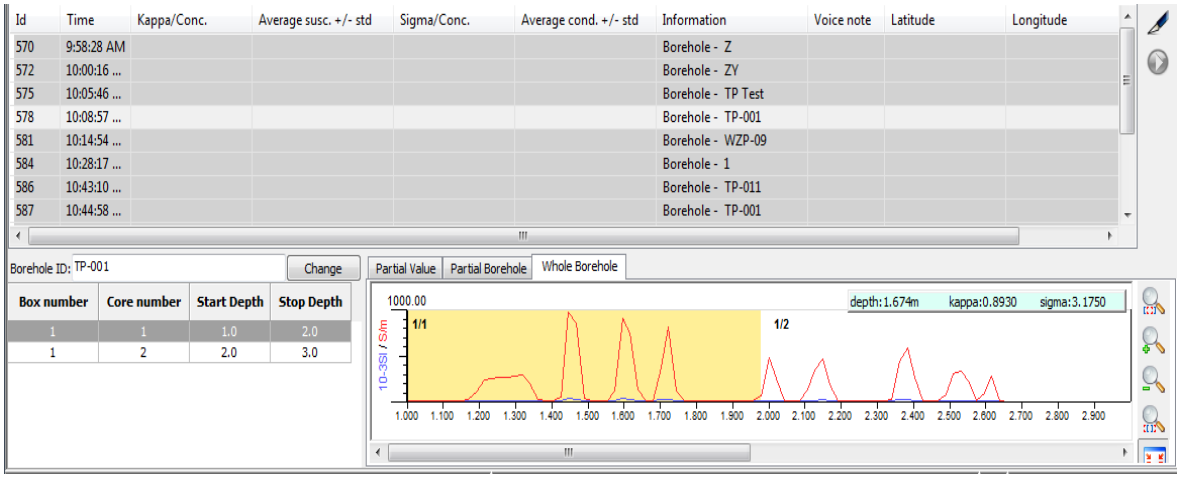
To reset the zoom level to default and look at the entire scan record, use the button.

To apply an auto-scale to the plot, which is the default view, use the button. To increase the scale use the button and to decrease the scale use the button.

Markers entered while scanning samples are presented in the plotted data and are visible in the numerical data as well. The numbers that are in red are denoted as a marker and in the plotted data the graphical representation of the marker is seen here as a cross +.

4.1.6 Borehole data display

Scanner Data: The screen capture below shows example data set collected using the Borehole mode.



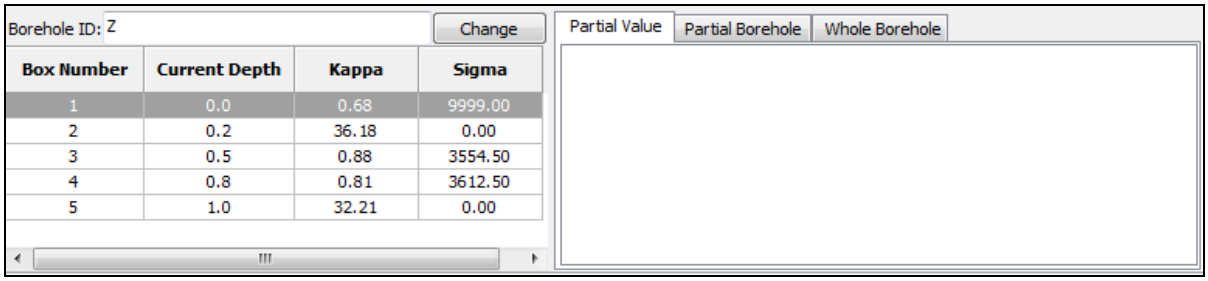
Along with the scanner graph, the GoeView shows the Borehole parameters such as the Borehole ID , Box Number, Core Number, Start & End depth of each scanned cores. The Borehole ID can be edited by clicking on the "Change" button.

Partial Value: Shows numerical value of a scanned core in time

Partial Borehole: Shows graphical view of a single scanned core in time

Whole Borehole: Shows graphical view of the entire cores or records collected for a given borehole. The graph is plotted with depth information.

Discrete mode (Measure mode)



For the discrete mode, the displayed data parameters are the core box number, depth, and the reading values for susceptibility and conductivity. The borehole ID and the data filed are editable. Clicking on the data field will unlock the field for editing. Please note that changes made into the GeoView database are not updated in the KT-10 v2.

4.1.7 Device Settings

Entering the Device options, allows for settings on your KT-10 v2 to be changed or saved. The KT-10 v2 will have to be connected to the PC for this menu to be accessible. Settings that can be changed with this menu are as follows: Sound, PC Authorization, GPS Pin, GPS, Shutoff timer, Automatic voice note replay, Battery selection and Time synchronization. Below you will see the window that is used to make these changes. This also allows for this information to be saved to a file for later use or loaded from a file to be sent to the KT-10 v2. With the exception of the GPS PIN setting each will only have two options available. The GPS PIN setting should be used when a Bluetooth GPS is to be used with the KT-10 v2 and has a documented PIN (Pairing Identification Number) or (Personal Identification Number). Consult with the documentation for your GPS to find this PIN and enter the number in the box provided. When all settings are changed to your preferences use the **Write to Device** at the bottom of the window to update the KT-10 v2.

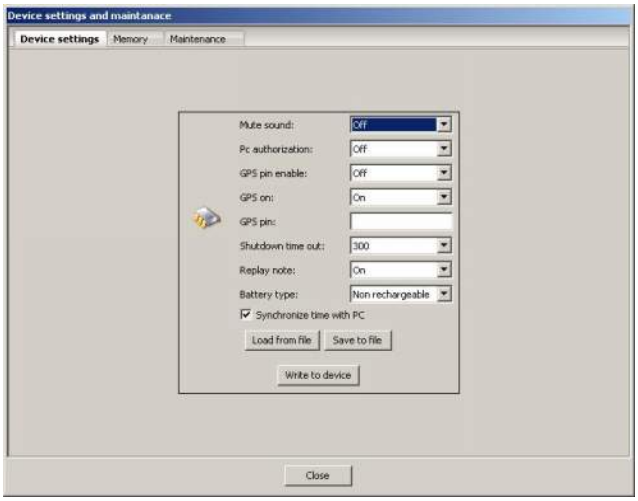


Figure 83: Device Settings

The Memory window will show the readings currently stored on your KT-10 v2 and allows for individual readings to be deleted.

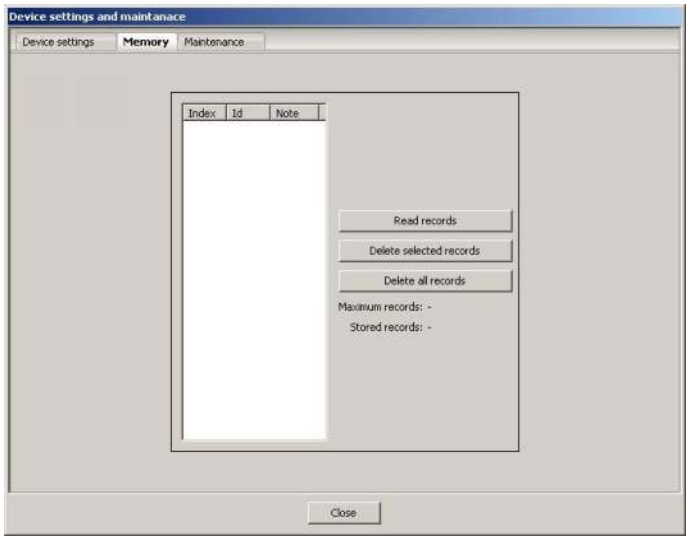


Figure 84: Memory Functions

Select the read records button to have GeoView list all readings currently stored on your KT-10 v2. Highlighting a record and pressing the delete record, will remove it from the meters's memory. Delete all records will format the complete memory on the KT-10 v2 and all data will be lost.

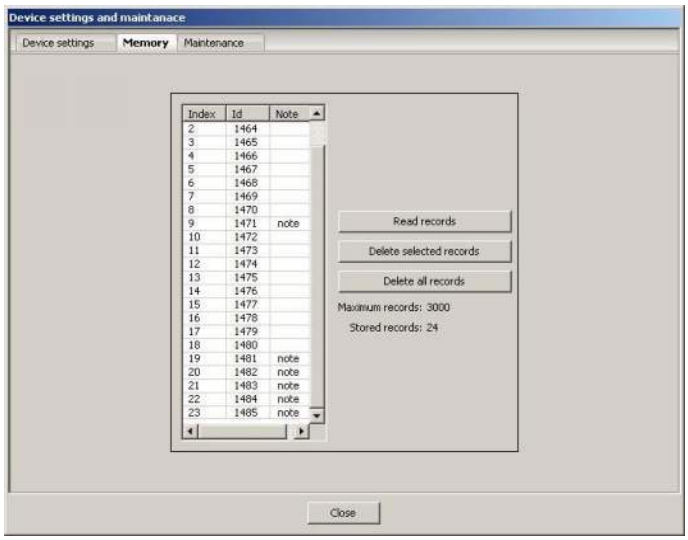


Figure 85: Memory Read

The Maintenance window has a firmware upgrade process and an additional assistance method which can be used to assist persons in the field to correct issues that may be un-foreseen. Session to maintainer is used for just this purpose. This will send the parameters file from the KT-10 v2 unit to the manufacturer for further analysis and possible

correction. This file can then be sent back to the end user to correct issues that maybe present.

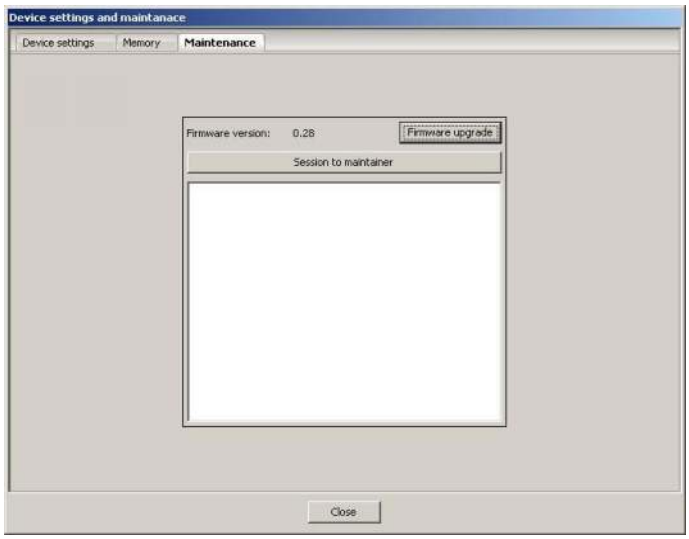


Figure 86: Maintenance

4.1.8 Firmware Upgrade

Firmware upgrade utility permits the user to install new firmware on their KT-10 v2 Firmware update allows the end user to enjoy new software features and/or correct any bug in the current firmware.

CAUTION: Failure to follow the upgrade instructions exactly could render the unit inoperative, requiring an RMA and a return to repair. Please follow the upgrade instructions explicitly.

When selecting ***Firmware Upgrade, as seen in figure 86,*** you will be presented with the following screen seen in **Figure 87**, select ***Next>*** to continue the installation or ***Cancel*** to exit.

Figure 87: Firmware Upgrade Wizard

You may select Download from the Internet, and then press **Next >.**

The wizard will automatically query the Georaids' web site. If it finds a firmware that is newer than what is currently on your KT-10 v2, it will be downloaded.

Figure 88: New Firmware Downloaded

Press **Next>** to continue

The utility will erase the current firmware and upload the new firmware on to the unit.

Figure 89: Firmware Uploaded to KT-10 v2

Press **Next >** to continue

You should see FW UPGRADE displayed on the KT-10 v2's display as seen in the Figure 90. It is very IMPORTANT not to disturb the unit during the upgrade process.

Figure 90: FW Upgrade screen

Upon successful upgrade, the KT-10 v2 will re-start.
You may then press the press **Finish** button to exit the firmware upgrade wizard.

Figure 91: Firmware Update Complete

4.2 KT-10 v2 Console – Geomon (not supplied)

The console program is called Geomon and is available for changing advanced parameter setting in the KT-10 v2. Some of the advanced parameter setting includes set or enable KT-10 v2 for re-calibration, read current calibration parameters, download measurement logs in text format, as well as it is used in advanced troubleshooting of the KT-10 v2.

Caution: Making changes without complete understanding or guidance from Terraplus Technical Support may result in incorrect operation of the KT-10 v2.

IMPORTANT: The KT-10 v2 console program cannot be executed directly from a CD-ROM. Please copy the console files(s) to local hard-drive of your laptop or desktop.

To install the console program run geomon.exe by double clicking on it. When the installation wizard starts the following security warning screen will be presented as seen in **Figure 92**.

Figure 92: Console Installation Wizard

Select, **Run** to continue…

Figure 93: Geomon Setup

Select, **Next >** to continue…

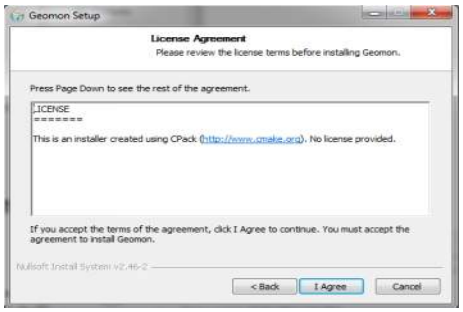


Figure 94: Licence Agreement

Select, **I Agree** to continue…

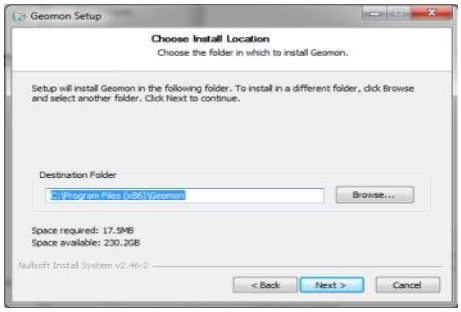


Figure 95: Destination folder

Select different destination folder if desired or click **Next >** to continue…

Figure 96: Start Manu folder

Select Start Menu Folder and then select "**Install**"…

To launch the Geomon, double click on the Geomon short-cut icon placed on the desktop or run it from the programs menu. **Figure 97** shows the Geomon software running.

Figure 97: Geomon

The buttons in the menu bar are as follow:

Figure 98: Connect device

Figure 99: Disconnect device

Figure 100: About

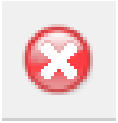


Figure 101: Quit application

To connect KT-10 v2 to Geomon;

1. Connect KT-10 v2 via the USB cable. Ensure that the GeoView software is not presently running.
2. Click on the Connect Device button as seen in **figure 98**.
3. Select device window will open up as seen in the figure below.

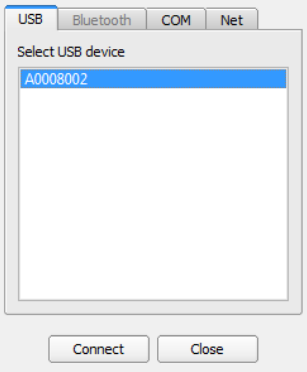


Figure 102: Select USB device

4. Highlight the serial number of your device in the box and then click on Connect. The following screen will appear as seen in the figure below. If the USB connection is successful, the status window, at the bottom of the window, will show device connected.

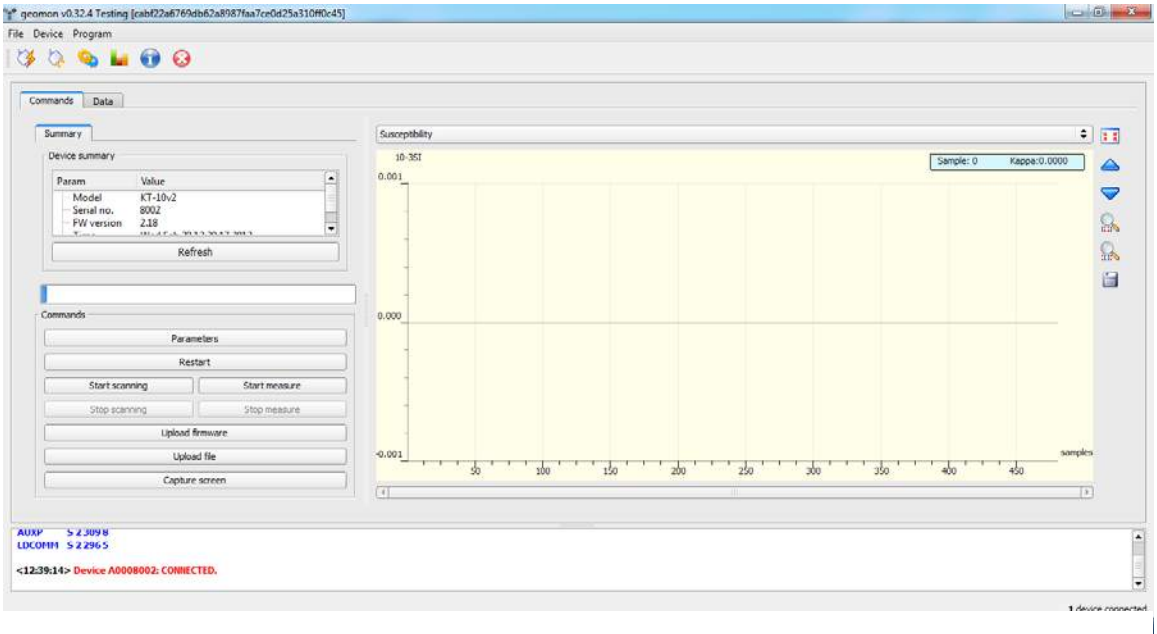


Figure 103: KT-10 v2 connected

Once the meter is connected, then the user can select between Commands or Data tab, as shown in figure below.

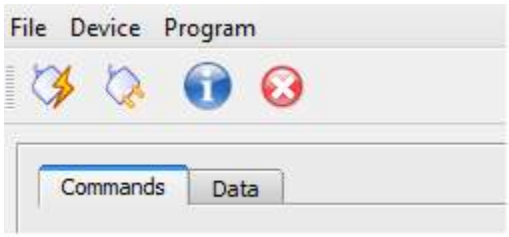
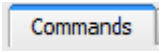
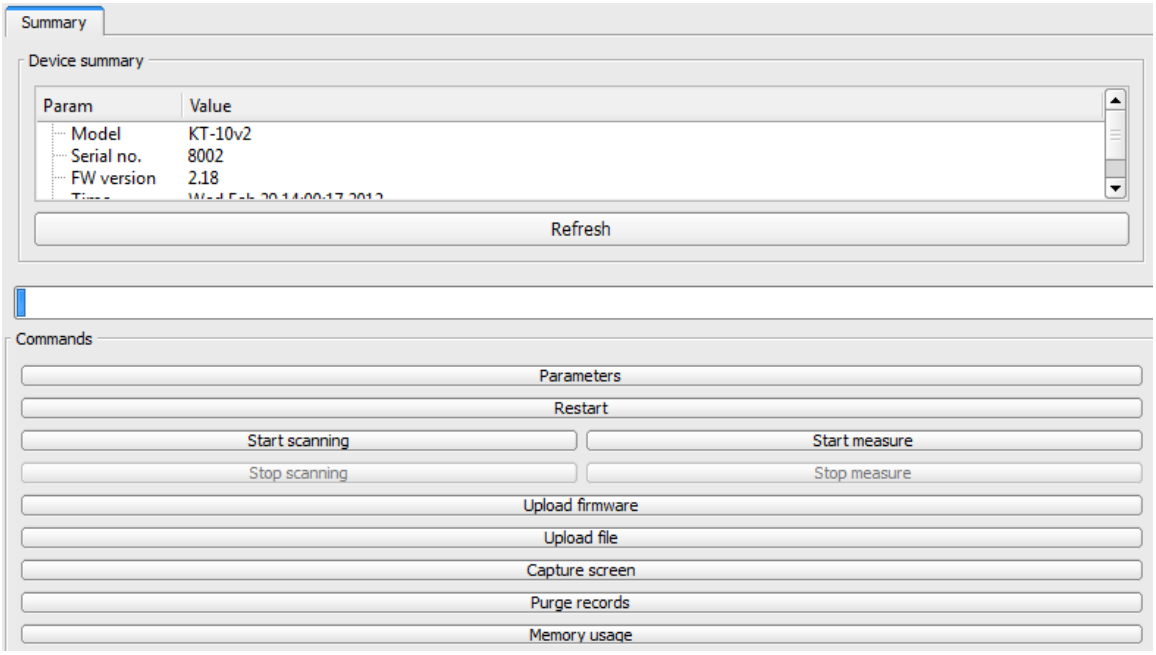


Figure 104: Geomon tab

The  tab offers the following:



Device Summary: lists information such as serial number, firmware version and model type of the meter that is connected.

Refresh: updates the summary window

Parameters: allows user to set device parameters, enter calibration value and so on…

Restart: re-starts the meter

Start scanning: starts scanner measurement

Stop scanning: stops scanner measurement

Start measure: activates measurement sequence

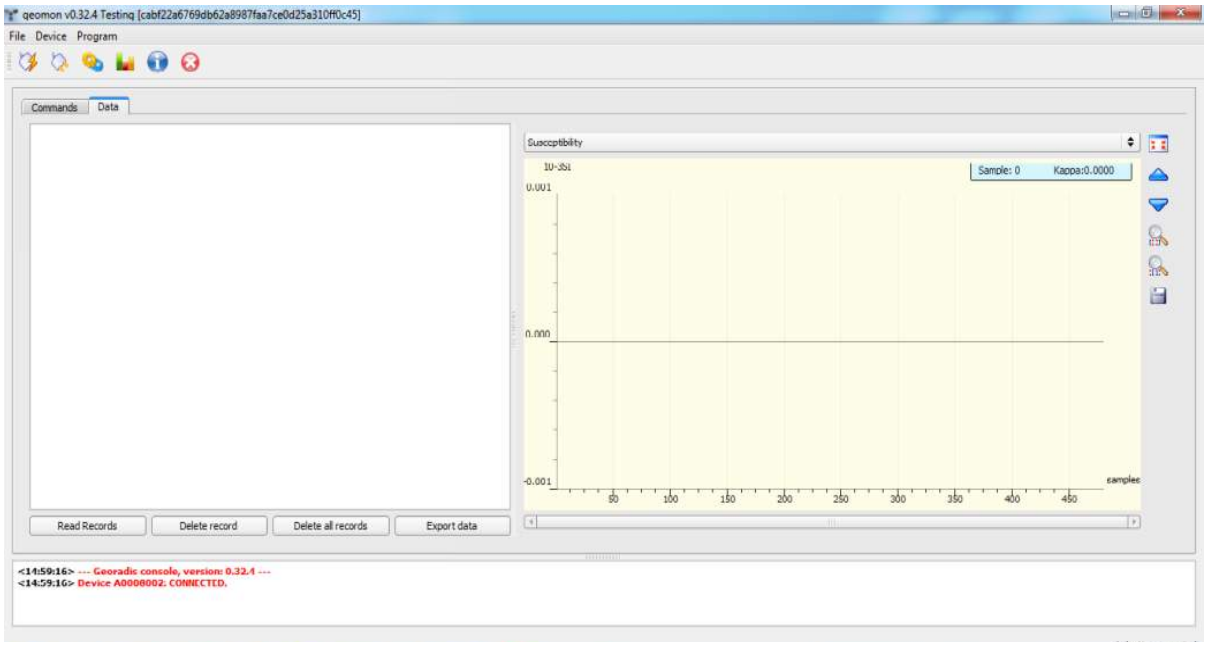
Stop measure: stops measurement sequence

Upload firmware: permits user to upload firmware on the KT-10 v2 as described in the section **4.1.8** on **page 78**.

Upload file: to upload .tar files on the internal memory of the KT-10 v2

Purge Records: permanently deletes records from the unit

The Data tab:



Read Records: reads all records on the device

Delete record: deletes individual record that is selected by the user

Delete all records: deletes all records on the device

Export data: Exports data set in text format

Chapter 5

KT-10 v2 – Bluetooth Connections

5.l PC Connections

The KT-10 v2 also has the ability to connect to the PC via Bluetooth, if your PC is Bluetooth capable. If you have the Bluetooth icon in the system tray of your PC you will have Bluetooth connectivity.

Figure 104: Bluetooth Icon

By right clicking on the icon you will receive the menu below

Figure 105. Add a Device

Select **Add Bluetooth Device** and the following window will be seen.

Figure 106. Bluetooth Wizard

Check the box **My device is set up and ready to be found**, and then select **Next**

Figure 107: Search for Device

Allow the Bluetooth search to finish, seen above. When it has completed the following screen will be presented. Select the KT-10 v2 with the correct serial number and choose N**ext>**

Figure 108: Select Device

No passkey is needed for the KT-10 v2 communications.

Bluetooth Passkey

The KT-10 v2 will then be connected to your PC.

Installing Bluetooth Device

When completed, you will be presented with a list of the ports associated with the KT-10 v2 Bluetooth connection.

Figure 109: Installation Complete

Finally, if you right click on the Bluetooth icon in the system tray and select show devices, the KT-10 v2 will now be listed.

Figure 110: Confirm Bluetooth Device

Further details on connecting the KT-10 v2 via Bluetooth to the GeoView software for Data Download is found in section 4.1.4 on Page 67.

5.2 GeoVision – Real Time Profile for Android Smartphone

(Standard with KT-10 S/C)

The GeoVision app can be paired with the KT-10 v2 to display real time scanner profiles on Android operated smart phones and tablets. Real time animated graphical outputs are displayed on the smart phone's screen when scanning. The GeoVision can also be used as a KT-10 v2 memory data browser to display field measurements/records, allowing the user to pan and zoom on the scanner graph. Additional text notes can be added to the current or previously stored data with an Android smart phone or tablet.

5.2.1 GeoVision - Installation

Important: The software requires Android OS 2.3.3 or higher

The GeoVision installation file, geovision.apk, is located on the KT-10 v2 Utility CD - inside of the Android folder.

Downloading the software onto your Smartphone;

1) Connect your phone to computer via the USB in Mass Storage mode

2) Once connected to computer, your phone's memory will appear as Removable Disk(s) - phone's internal memory or external SD card;

3) Copy the geovision.apk installation file into 1 of the 2 memory space available on your phone.

4) Once copied, disconnect the phone from the computer

Installing the software on your Smartphone;

1) Select Applications from the main menu 

2) Select My Files or any suitable file browser utility installed on your phone to browse to the geovision.apk file copied earlier.

Figure 111: GeoView Install file

3) Tap on the geovision.apk to being installation.

4) You will be then asked to confirm if you wish to install the application, confirm it by selecting the “Install” button.

Figure 112: Confirm Installation

5) After the successful installation of the application, you may either select Open or Done. Selecting “Open” will launch the GeoVision application on your device.

Figure 113: GeoVision installed

5.2.2 GeoVision – main menu

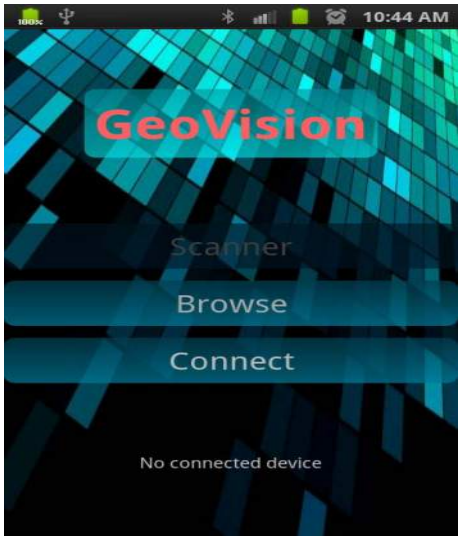


Figure 114: GeoVision menu

The main menu lists 3 selections and they are:

Scanner — Initiates real time scanner profile measurement on the KT-10 v2 if the meter is paired with GeoVision.

Browse — Allows an operator to review data stored in the KT-10 v2's memory. Operator can then view any records or attach text note using the keypad of the mobile phone.

Connect — Connects GeoVision to the KT-10 v2 via Bluetooth.

5.2.2.1 Connect / Disconnect

To connect GeoVision to the KT-10 v2:

1. Tap on the Connect button.
2. If your phone is not currently paired with any Bluetooth device, you will be provided with selection to Scan for Bluetooth device as seen in the figure below.

Figure 115: Scan for device

3. Tap on Scan for devices button.
4. When the scan is completed, a list of Bluetooth device will be populated as seen in **Figure 116**.

Figure 116: Device selection

5. Find and select your KT-10 v2 from the list by taping on the device's name.

6. When the GeoView is successfully connected to your KT-10 v2, the following screen can be seen momentarily – indicating that the device is connected.

Figure 117: Device connected

Please note that the Connect button is now replaced with Disconnect. Also, the Scanner button is now active.

To disconnect your KT-10 v2 from the GeoVision / mobile device;

Simply tap on the Disconnect button. When disconnected, the following screen will be displayed momentarily and the Disconnect button will be then replaced with Connect.

Figure 118: Device disconnected

5.2.2.2 Browse

To browse previously stored data on the KT-10 v2, tap on the Browse button. It takes a few seconds for the GeoVision to read all of the data from the KT-10 v2. When the reading of data is completed, a device log will be populated as seen in the figure below.

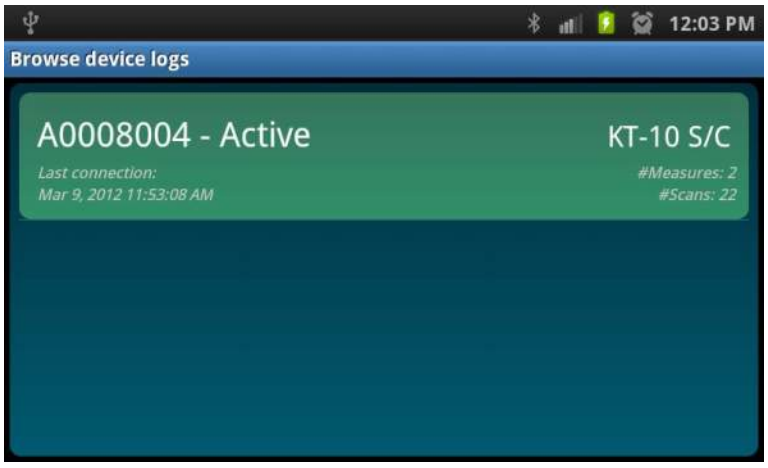


Figure 119: Device Log

Note: Phone

In the figure 119, the serial number of the paired KT-10 v2 as well the number of stored records for Measure and Scanner is displayed.

To browse stored data, tap on the serial number of the KT-10 v2. A new screen, as seen in Figure 120, will be displayed listing records under Scanner and Measure tab.

Figure 120: Data preview

To view Scanner records, select Scanner in the main header and then scroll up or down to browse different scanner record.

As seen in **Figure 121**, the scanner record is consist of Average and Max value of Susceptibility & Conductivity, Duration in # samples recorded, and the time when the measurement was saved on the meter.

Figure 121: Scanner record explained

Note: In the scanner mode 20 samples per seconds are collected. Of the 20 samples, 5 are averaged together, total of 4 samples are stored per second. The maximum number of samples for full scanner duration is 120 seconds x 4 = 480.

To preview a scanner graph, tap on the record of your choice. The scanner graph will be previewed as seen in the **Figure 122** below.

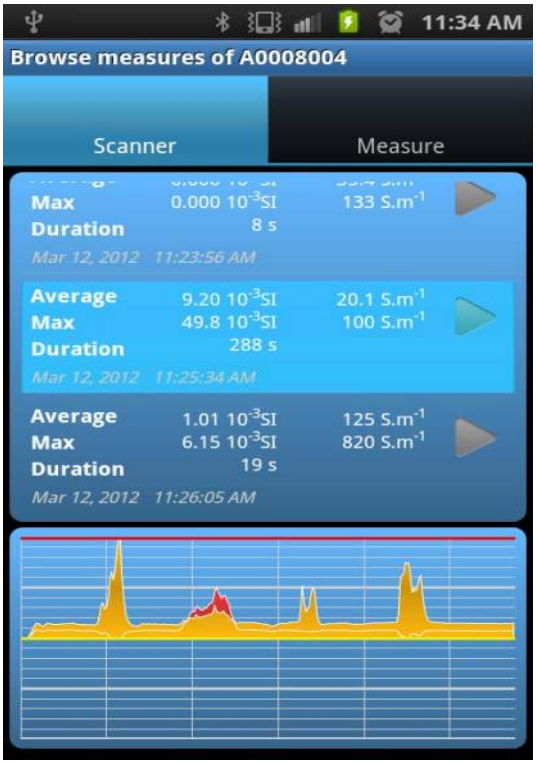


Figure 122: Scanner data preview

Tap on the scanner graph preview area to see the record in full size.

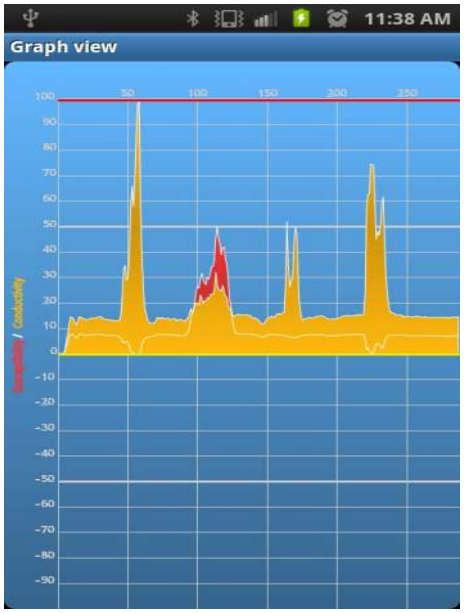


Figure 123: Scanner record full size (Portrait view)

Figure 124: Scanner record full size (Landscape view)

5.2.3 Graph Scale

To set/change graph scale, tap on the menu button  of your mobile phone. A new menu with scale options will be open up as seen in the figure 125 seen below.

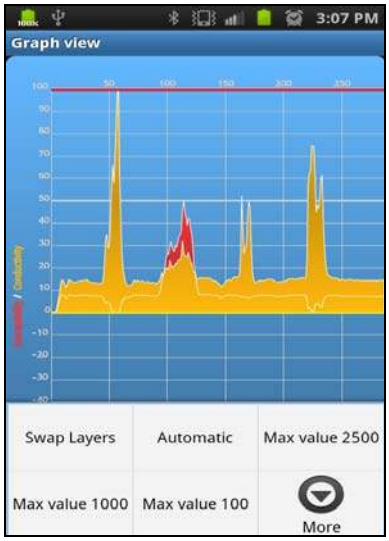


Figure 125: Adjust scale – pull menu

Swap Layers:

Changes the way the graph is displayed. Normally, the graph of the Conductivity is displayed in the foreground and susceptibility is displayed in the background. By swapping layers, the graph of susceptibility is now placed in the foreground and conductivity is moved to the background.

Automatic:

Sets automatic scale for the graph (Y axis) to ensure displayed graph is easier to read.

Max value 2500:

Sets the maximum scale value of the Y axis to 2500. Scanner record with readings over 2500 will be clipped.

Max value 1000:

Sets the maximum scale value of the Y axis to 1000. Scanner record with readings over 1000 will be clipped.

Max value 100:

Sets the maximum scale value of the Y axis to 100. Scanner record with readings over 100 will be clipped.

Tapping on the More button will show more options to set the Y axis scale.

Max value 10:

Sets the maximum scale value of the Y axis to 10. Scanner record with readings over 10 will be clipped.

Max value 1:

Sets the maximum scale value of the Y axis to 1. Scanner record with readings over 1 will be clipped.

Max value 0.1:

Sets the maximum scale value of the Y axis to 100. Scanner record with readings over 0.1 will be clipped.

In the Figure 126, the function “Swap Layers” is used on the display to show the same graph as shown in Figure 125.

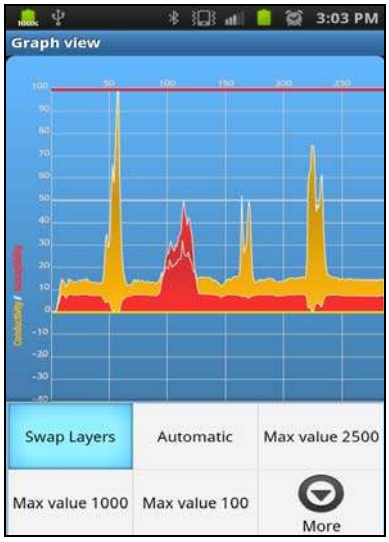


Figure 126: Swap Layers

5.2.4 Zoom / Pan

To **Zoom** into the graph, simply place your two fingers on the area you would like to zoom into and then separate the two fingers away on the screen. This pinching out of the two fingers for zoom can be done on both the Y and X axis.

To **Pan** into the graph, simply place your two fingers on the area you would like to Pan into and then bring the two fingers closer on the screen. This pinching in of the two fingers for Pan can be done on both the Y and X axis.

5.2.5 Delete data

If you highlight and hold on a measurement record (both scanner/measure) you will see a menu pop up for deleting record. By tapping on Delete record will delete the selected record from phone's memory as well the meter if there is an active Bluetooth connection between the meter and the GeoVision.

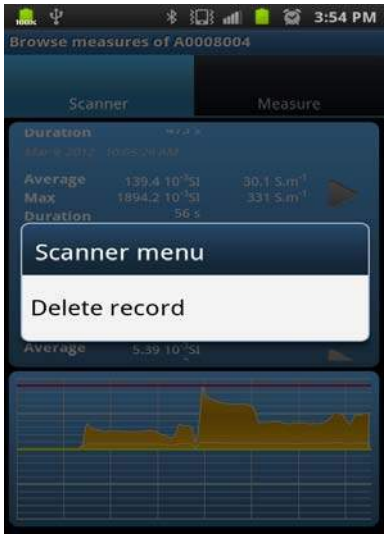


Figure 127: Delete record

Chapter 6

Troubleshooting

6.1 Notes about Switching OFF.

Like other instruments that are based on a microcomputer core, the KT-10 v2 meter may be sensitive, in some specific circumstances, on external distortion (strong electromagnetic field, discharge) and may show improper operational behaviour. The most common symptom is no reaction on any of the button presses. To bring the unit back in operational condition it is necessary to switch OFF and then switch ON the unit. To simplify the OFF/ON process, press and hold the Up & Down button together. This way, the user can switch OFF the unit in situation when there is no software switch accessible the way of contemporary push of button up and down; every time regardless of any working status of the unit.

6.2 KT-10 v2 Powers OFF during measurement

Battery:

Confirm that the 2 AA batteries are at proper voltage level. Replace if necessary.

Button:

It may be possible that both the UP and DOWN buttons are accidently being pressed at the same-time instead of a single UP or DOWN button. **Re-position your finger on the button so that only the intended button gets pressed.**

Battery Cap:

It may be possible that the spring tension of the battery cap has become weak, causing intermittent contact with the batteries when the KT-10 v2 is moved around during a measurement. A battery cap replacement will be required.

6.3 “Error” on the Display

An “Error” message is displayed on the KT-10 v2’s screen when it fails to compute a result as seen in the figure 128.

Figure 128: Measurement Error

The main reason for this error message may be that a measurement sequence is not being followed properly.

To ensure that a correct measurement sequence is being followed, please try do the following:

1. Set the KT-10 v2 in the Measure mode.
2. With the KT-10 v2 at arm's length, in the free air, press the UP button. Meter beeps, and the "progress bar" starts to run.
3. Immediately after the first beep, quickly move the KT-10 v2 to the sample’s surface and press the UP button again.
4. Hold the KT-10 v2 on the sample until it beeps a second time, with a different tone. After the second beep, quickly move the instrument back to arm's length. Do not push the button again.
5. The KT-10 v2 beeps a third time on it's own; displaying result on the screen.

TIP: Alternately, the Scanner mode can be used to confirm the operation of the meter.

6.4 Maintenance

Switch Cover: The rubber cover on the buttons can degrade over time with the regular usage. A worn-out rubber cover will allow dust particles to enter inside of the meter. Any dust particles inside of the meter could make the buttons unresponsive to the presses. It is important to replace a damaged switch cover to avoid any internal damages.

Spare rubber cover for the switch can be purchased through Terraplus Inc. and replaced in the field.

Coil Cap: The black coil cap on the head of the KT-10 v2 can wear and tear with regular usage. Spare Coil caps can be purchased through Terraplus Inc.

6.5 Contact Technical Support

If you are experiencing problem(s) with your KT-10 v2 or the application software, please contact Terraplus Technical Support for assistance. Our contact information is below; please try to contact Terraplus' Technical Support between the hours of 9 am and 5:30 pm E.S.T., otherwise please leave a voice message or send an email and someone will be in touch with you shortly.

Terraplus Inc.
52 West Beaver Creek Unit 12
Richmond Hill, Ontario
L4B 1L9 (Canada)
Phone: (905) 764-5505
Fax: (905) 764-8093
E-mail: support@terraplus.ca
Website: http://www.terraplus.ca/

Appendix A: KT-10 v2 Plus Feature

This document is in addition to the KT-10 v2 manual. It is intended to provide information specific to the operation of the Plus feature for the KT-10 v2. The procedures outlined in this section are also applicable for conductivity measurements and calibration. Please consult the KT-10 v2's operations manual for general operating instructions.

The KT-10 v2 Plus has an increased range of up to 10 SI units for magnetic susceptibility measurements. Direct iron ore concentration estimates can be obtained from the meter's display with the use of a magnetite calibration curve already loaded into the unit.

The KT-10 v2 Plus meter has been developed to allow users to calibrate the meter to specific ore types for quick recognition in the field. It can be used for sample selection, core analysis and grade control. With the ability to use three different calibration curves, one of which is the pre-installed magnetite curve, users have a flexible, user friendly instrument suitable for several different applications. If the samples or cores have a different composition or structure than those used to create the pre-installed calibration curve included in the KT-10 v2 Plus, the user can program up to two additional calibration curves that are specific to the samples and cores they are measuring.

At the end of this guide you will find recommendations for preparing samples for site specific calibration procedures.

The KT-10 v2 Plus has an increased range of operation (10 SI). Users can select different units (SI units or %) to receive measurement results in; this option is located in the Setup menu. From the main menu select the Setup option and the following menu will be presented.

Figure 129: Setup menu

When the option **Conc. tables** has been selected, the user will be presented with an option to select a list of calibration tables for susceptibility or conductivity **(Figure 130)**.

Figure 130: Conc. Tables Sub menu

Select the appropriate measurement mode (**Figure 130**) to reveal the installed calibration tables for that mode.

Figure 131: Susceptibility - Calibration tables

The first unit of measurement, Basic [SI], is used to obtain magnetic susceptibility measurements in SI units. Under this selection, measurements are displayed and stored in SI units.

Selecting Magnetite [%] will enable the unit to read iron ore concentration estimates as a percentage. This calibration table is based on magnetite with different concentrations.

The next two selections, User1 [%] & User2 [%], remain open to be populated by the end user. Please note that these two tables are currently populated with the same table used for magnetite.

The process to load a calibration curve is outlined on page 109. To begin using the preloaded calibration table, select it from the list and return to the main menu. The KT-10 v2 will remember the last table selection made upon restart.

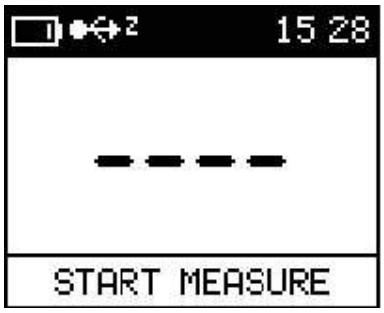


Figure 132: Table type notified

On the measurement screen (**Figure 132**), a number in the upper left hand corner of the display is used to indicate which calibration table is currently being used.

Legend used to notify current calibration table in use:

1 = Magnetite [%]

2 = User1 [%]

3 = User2 [%]

There will be no number shown in the notification area when the Basic SI is selected as the unit of measurement.

The procedure to take a reading is described in the in this user's manual and the quick user guide.

When a calibration table is used, the measurements are presented to the user as percentage with average and standard deviation displayed on the bottom line (**Figure 133**).

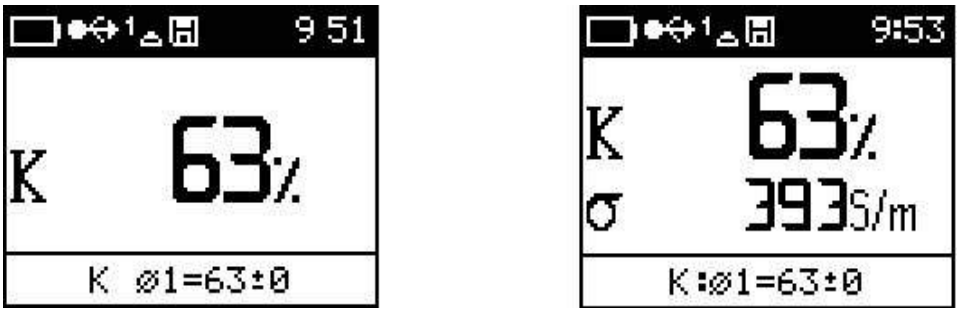


Figure 133: Calibrated Percentage Display

The data stored on the KT-10 v2 will be in a percentage with average and standard deviation values for each group of readings.

GeoView Software Interface

GeoView is used to access the data stored on the KT-10 meters. It also provides access to the settings and calibration tables used in the meter.

Connect your KT-10 Plus v2 to a PC and launch the GeoView software to load, view, delete, or save a calibration table. Click on the  button to access the Device Parameters settings. In this menu, you will see a "Concentration" tab. If there is no Concentration tab, then the instrument connected to Geoview is not a Plus meter.

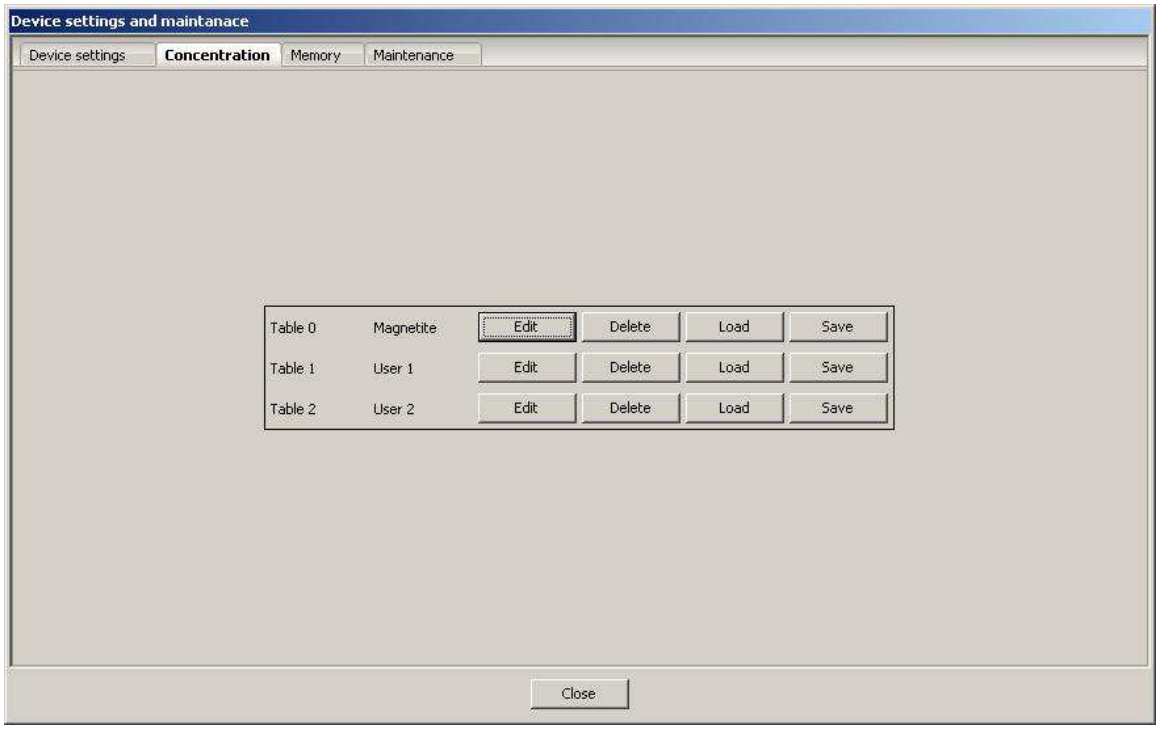


Figure 134: Concentration tab

In the Concentration tab, you will find the three tables that are loaded on the KT-10 Plus.

The ***Edit*** button will open the table for editing of the name and the calibration data points. The ***Delete*** button will erase the table from the meter's memory. The ***Load*** button will allow the user to load a new table in the meter's memory. The ***Save*** button will allow the user to save the table on the computer. The saved table then can be loaded onto another meter.

The name of the table can be edited so that it is easier to distinguish from other tables. The table's name cannot be longer than 11 characters. You can also edit the percentage and SI reading columns in the table. In the example below, ***Point 1*** is for 1 percent and has the assigned value of 26.0 X 10^3 SI units.

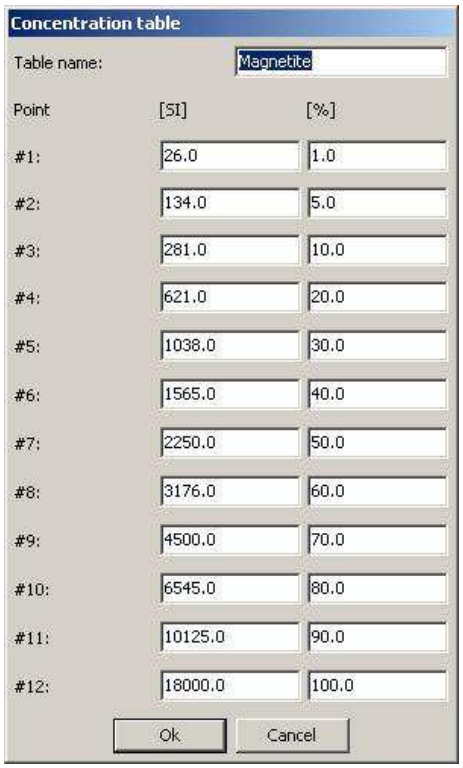


Figure 135: Concentration Table

When creating a table, you do not need to have all of the 12 data points entered for reference. Smaller tables, with a minimum of 4 data points, can be used, but they must follow a specific format.

In order to use a smaller table, the concentration table must be padded with zeroes where there are unpopulated data points. The absolute position of the table is not important, but it cannot contain zeroes between the start and end data points as this will result in the meter neglecting the higher concentration data points **(Figure 136)**.

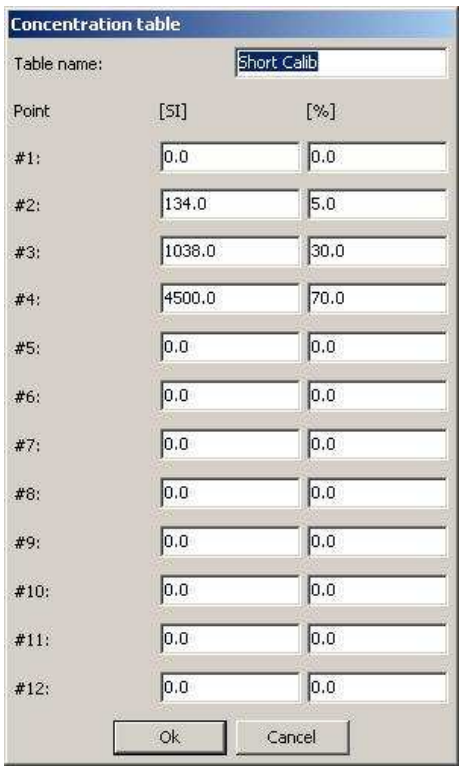


Figure 136: Short Calibration table

In **Figure 136**, the range from 5 % to 70 % has been included in only three data points. Please note that any values above and below this table will produce questionable a output. After the data has been collected and transferred into Geoview, you will see the additional information showing when the calibration curves have been used. In the information column, the calibration curve number is provided for each reading and is populated for any mode used with a calibration curve. **Figure 137** shows the results from scanner and measure modes where calibration curve 1 (magnetite) was used.

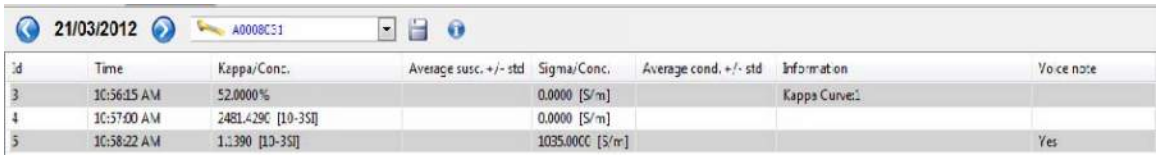


Figure 137: KT-10 Plus Data view

Recommendations for KT-10 Plus Calibrations

The calibration curves are based on the relation between the measured susceptibility and the real concentration of a specific type of mineral. The calibration consists of creating a numerical table or function that will cover all of the different concentrations found in an ideal sample. The KT-10 Plus comes with calibration for ideal magnetite.

Recommended Materials for the Calibration Process

1. KT-10 Plus v2
2. Five plastic bags approx size 8” x 8” or bigger
3. Five samples of a specific local mineral or rock at varying concentrations.
4. A PC with a text editor and the GeoView software.

Sample Preparation

The user must prepare the samples in a consistent geometry to eliminate the deviations caused by geometric factors or unsuitable shapes. The shape of the samples must conform to the expected or common shape of real samples that will be measured in future production. The closer the calibration sample compares to the real samples, the more precise the measurements will be.

If there is a scenario where the common samples are rocks and the user does want to crush them prior to taking measurements, then it is recommended to have samples with a minimum size of 4” x 4” and a thickness of 1.5”. In places where the most common mineral is in a fine gravel or sand, it is recommended that it be poured into a plastic 8” x 8” bag and formed into the shape of a pillow, with a thickness of approximately 2”.

Calibration Samples Selection

The calibration method expects to cover a wide range of local concentration profiles. For better interpolation, we recommend samples be taken from the mine or pit and contain both low and high concentrations. The user can define five points of conversion for the table and a suitable selection of calibration points can increase precision within a focused range. For example, if a site has an iron concentration of approximately 30% and this concentration is found in approximately three quarters of the reserves, while the remaining reserves have concentrations close to the local maximum (45%, in this case), the calibration can be configured as presented in the following table **(Figure 138)**.

Concentration	Sample Concentration
<5%	Below average
25%	
30%	Local average
40%	
45%	Local Maximum

Figure 138: Calibration Selection table

Calibration Table

To create the calibration table, the user will require calibration samples with known concentrations; the concentrations should be determined by another method, such as chemical assay, XRF, etc. The calibration samples must also be in a suitable form and geometry similar to the local samples found on site. Once the user obtains the appropriate calibration samples, they must perform five susceptibility measurements for calibration and the creation of the relation table. The following table is to be used only as an example.

Concentration	**Susceptibility**
<5	98.21 x 10^{-3} SI
25	405.66 x 10^{-3} SI
30	630.10 x 10^{-3} SI
40	867.72 x 10^{-3} SI
45	1139.23 x 10^{-3} SI

It is recommended that multiple measurements be taken on each sample; use the average value to populate the concentration table. Next, the user must transfer the concentration table to the KT-10 Plus v2; this can be done with Geoview following the steps beginning on page 109.

Format is:

Susceptibility in SI * 10^{-3}	&	Concentration in % (percent)
98.21		5
405.66		25
630.10		30
867.72		40
1139.23		45

Finally, the user must access the setup menu to select the output in concentration. Once this final step is complete, the measurements on production samples can begin.

Appendix B: Advice and Recommendations

Measurements of bulk susceptibility or conductivity using a new handheld meter can be used in a wide range of applications in various geological fields, making the instrument useful for both geologists and geophysicists. But there are some considerations that must be followed in order to obtain good results and protect the instrument against damage. These are as follows:

• Do not take measurements with weak batteries (exchange batteries after the low battery signal appears at your earliest convenience).

• Do not take measurements in the rain when the surfaces of rocks are very wet.

• The first and third steps in a measurement (measuring with coil in the air, otherwise known as free air measurements) should not be executed near earrings, necklaces or any other metallic objects. When taking free air measurements, maintain distance of at least 50 cm from anything metallic.

• When taking readings on drill cores, avoid measuring near the nails of the wooden core box. Never measure cores placed in metallic boxes. For best results, it is recommended that cores be removed from the boxes during measurements.

• When measuring on outcrops, care must be taken to find convenient surfaces in order to eliminate the influence of weathering. Weathering results in a decrease of susceptibility, which is intensified the more the sample is weathered. It is important to remember that the coil is most strongly influenced by the rock nearest to the coil's surface, even if one measures using the pin. Magnetic anisotropy exists and measure parallel and perpendicular to the foliation in metamorphic rocks. Make corrections for the unevenness of the rock surfaces. Susceptibility distribution in any outcrop is found relatively reliably if more than 12 measurements are made; one, two or three measurements are insufficient. Take care not to measure near the geological hammer you may have with you.

• When placing the meter on a rock's surface, do so gently. Beware that shocks and rough handling of the meter on rocks can damage the measuring coil.

• To verify magnetic anomalies, it is recommended that users measure all kinds of rocks available in the region of interest, even small rocks and soil debris. They may not cover the entire surface of the coil and may be very thin; but, it is important to remember that the value you obtain is informative only. From a collection you gathered you can take the characteristic pieces for the lab measurement, sufficiently big and suitable for cutting the lab specimens. Susceptibility measurements help you to take representative samples for lab measurement of anisotropy and/or remnant magnetization measurement.

Warranty:

All KT-10 v2 models come with a one-year warranty from Terraplus Inc from the date of shipment. Warranty for the KT-10 v2 models covers defective components and workmanship for the repair at Terraplus' office in Richmond Hill, Ontario, Canada. Shipment costs to and from Terraplus' facility are not covered under warranty and are the responsibility of the customer. Malfunctions or damages due to negligence or improper use are not covered under this warranty. Users who need to send their meter to Terraplus for repair should complete the RMA request form on our website. The RMA request form can also be found at: http://www.terraplus.ca/misc/support.aspx

Terraplus Inc.
52 West Beaver Creek Unit 12
Richmond Hill, Ontario
L4B 1L9 (Canada)
Phone: (905) 764-5505
Fax: (905) 764-8093
E-mail: sales@terraplus.ca
E-mail :support@terraplus.ca
Website: http://www.terraplus.ca

Appendix B
Project Data Quality Objectives

1 - Problem Description: The U.S. Department of Veterans Affairs (VA) operated a part-time dry cleaning operation that used tetrachloroethene (PCE) over a 6-year period in the late 1970s and early 1980s. During this period, dry cleaning residuals were disposed of in the sanitary sewer. PCE-contaminated groundwater is present beneath the VA Medical Center (VAMC) property and the Sunnyside Park sewer line, as well as in areas hydraulically downgradient, extending to the East Side Springs (ESS) neighborhood (**Figure 2-1**). The site was placed on the EPA Superfund National Priority List in 2013 and is now referred to as the 700 South 1600 East PCE Plume Superfund Site (Site). The University of Utah, Mount Olivet Cemetery, and East High School, in addition to residential neighborhoods, are in the vicinity of the Site. The current conceptual site model is presented in the Conceptual Site Model Update (EA 2017), illustrating sources of contaminant release, fate and transport, potential exposure pathways, and receptors. Human exposure to PCE-contaminated groundwater is possible via existing or new water supply wells (including SLC-18 if it were brought back online). Additionally, humans may also be exposed due to vapor intrusion (VI) and direct-contact pathways associated with shallow groundwater and springs in the ESS area. Ecological receptors may encounter contaminated surface water and shallow groundwater. Although the Phase 1 Remedial Investigation (RI) and past investigations have collected measured concentrations of PCE and other volatile organic chemicals (VOCs) in various media, additional data are needed to characterize the hydrogeology and nature and extent of VOC contamination, to assess potential transport and exposure pathways and risks, and to inform the development and detailed analysis of remedial alternatives during the subsequent feasibility study (FS).

2 - Principal Study Question	3 - Information Inputs (1)	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
E1 (Hydrogeologic Features) What hydrogeologic features control VOC fate and transport? ***Estimation Statement:*** There are several hydrogeological features to be estimated that may control VOC fate and transport that vary across the site, including: location of faults, lithology, hydrostratigraphy, hillside discharge in the ESS area, hydraulic connection between the source area and production wells (i.e., SLC-18, University of Utah wells, and Mount Olivet well), hydraulic influence of Red Butte Creek, and groundwater flow direction and discharge. A 3-dimensional (3D) numerical groundwater model will be constructed and used in tandem with a 3D visualization model to incorporate these data into the Conceptual Site Model (CSM) and estimate their impact on the fate and transport of VOCs in groundwater.	The following information is needed to determine the hydrogeologic conditions, including: ▪ Lithology and hydrostratigraphy data (lithologic logs, piezometric head data, and extent and thickness of perching layers in the source area) ▪ Structural geology (fault trace and orientation) ▪ Borehole geophysical data (hydraulic conductivity and porosity provided by nuclear magnetic response, natural gamma/neutron gamma) ▪ Water levels and horizontal and vertical hydraulic gradients ▪ Recharge and pumping history ▪ Aquifer test results and estimated hydrogeological properties including transmissivity, hydraulic conductivity, and storativity ▪ Information on surface water discharges, seeps, and springs ▪ Influence of Red Butte Creek on groundwater flow direction and discharge The 3D numerical groundwater model should incorporate measured data collected during all phases of the RI (i.e. AOU1 RI, Phase 1 OU2 RI, and Phase 2 OU1 RI).	The lateral study boundary is shown on **Figure 2-1** and is defined by areas where soil, groundwater, surface water, seep and spring water are known to have been impacted by PCE and degradation products from historic operations on the VAMC property. The known impacted area includes VOC-contaminated groundwater beneath the VAMC property and in areas hydraulically downgradient, extending to the ESS neighborhood. The vertical interval of VOC impacts will be evaluated at borings during this investigation. Because the lateral and vertical extent of the groundwater PCE plume has not been fully defined the spatial bounds of the study area could expand or be reduced as more information about the Site is obtained. Temporal boundaries will vary by information input. Geologic and hydrogeologic properties of the subsurface, including but not limited to lithology/hydrostratigraphy, structural geology, and geophysical properties are not expected to vary over time. Information inputs necessary to understand the hydrogeological controls on the plume, including water levels/hydraulic gradients, pumping history/rates at municipal and irrigation wells, vary over time. Water level and hydraulic gradient measurements will be completed over the course of at least one calendar year to account for seasonal variation. Historical and current well pumping data will be obtained to the extent practical, with monthly extraction data desired for at least one calendar year. There is limited space for installation of additional monitoring wells in the vicinity of Buildings 6 and 7 on the VAMC campus, thus	The study will estimate the transport characteristics (rate and direction) of VOC-impacted groundwater within the plume. The statistical parameter of interest when estimating hydrogeologic properties is the mean value of a hydrogeologic parameter measured at a specific well or borehole location. The information inputs will be integrated into a sitewide CSM, using 3D visualization tools and groundwater modeling software. If professional judgment shows too much uncertainty the hydrogeologic parameters of interest in specific locations or regarding specific factors, then additional data collection will be considered.	Site lithology and hydrostratigraphy varies across the Site and several hydrogeologic features have been identified that may control VOC fate and transport, including faults, hillside discharge in the ESS area, hydraulic connection between the source area and production wells (i.e., SLC-18, University of Utah wells, and Mount Olivet well), and the hydraulic influence of Red Butte Creek. Thus, measured hydrogeologic data should be spatially representative (i.e. laterally and vertically) of these hydrogeologic features. The hydrogeologic data inputs will be incorporated into the CSM using a 3D numerical groundwater model to estimate the impact of specific hydrogeological features on the fate and transport of VOCs in groundwater at the site. Data to support this study question will be collected in accordance with standard operating procedures presented in the QAPP to reduce the potential for sampling and measurement error and reduce uncertainty in the values.	Phase 2 investigation activities and proposed investigation locations are presented in Section 4.2 and Section 5 of this RI work plan. The specific hydrogeological data collection activities during Phase 2 include: ▪ Collect lithology, hydrostratigraphy, and borehole geophysical data from new and existing monitoring wells and assimilate information into a 3D visualization. ▪ Collect multiple rounds of water level data from new and existing wells, including synoptic water level measurement events at all wells, and transducer measurements at select wells. ▪ Perform aquifer tests on selected wells to characterize hydraulic properties in areas impacted by the PCE plume.

2 - Principal Study Question	3 - Information Inputs [1]	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
		collection of hydrogeologic data of desired density may be physically constrained by the structures in this area. Collection of borehole geophysical measurements is limited to wells which are constructed in a manner that is compatible with the logging tools used. The smallest estimation unit for estimating hydrogeologic parameters is a single hydrogeologic unit or feature (i.e. zones that have similar hydrogeologic characteristics and behavior). The 3D numerical groundwater model will be used as a tool to incorporate hydrogeologic data into the CSM to identify areas of low confidence where additional data may be required to refine the model output, thus spacing of boreholes/monitoring wells should be sufficient to keep model uncertainty within acceptable limits.		
E2 (Plume Characterization) What is the lateral and vertical extent of PCE and degradation products in groundwater downgradient from the source area? ***Estimation Statement:*** The extent of PCE and degradation products in groundwater at the Site is to be estimated during development of the CSM, using 3D visualization techniques to evaluate the lateral and vertical extent of groundwater containing VOCs. In combination with the evaluation of hydrogeological conditions, the transport of PCE and degradation products in groundwater will be evaluated to estimate flow paths and transport times. These data will be used to evaluate areas of the site where additional investigations may be necessary to evaluate risks to human receptors.	The following information is needed to determine the 3D extent of VOCs in the groundwater plume at the site: ▪ VOC concentrations in groundwater at monitoring wells, including temporal trends ▪ Well depth, screen interval, lithology, and ground surface and top of casing elevations ▪ Water levels and horizontal and vertical hydraulic gradients ▪ Maximum contaminant levels (MCLs) or applicable screening levels The analytical method should be selected such that the reporting limit (RL) for each VOC is below its respective MCL or applicable screening level for decision-making. The lateral and vertical extent of PCE and degradation products in groundwater should be derived using measured hydrogeologic and groundwater data collected during all phases of the RI (i.e. AOU1 RI, Phase 1 OU2 RI, and Phase 2 OU1 RI).	The lateral study boundary is shown on **Figure 2-1**. The vertical interval of VOC impacts will be evaluated during this investigation. There is limited space for installation of additional monitoring wells in the vicinity of Buildings 6 and 7 on the VAMC campus (i.e. source area), thus installation of additional groundwater wells in this area may be physically constrained by the structures in this area. Additionally, the Site is located in a highly developed, urban/residential area where structures may restrict access to investigation in other areas. Ideally, monitoring well density should be sufficient to delineate the plume to within one city block. However, due to the size of the plume (approximately 300 acres) and the physical constraints on investigation in some areas, a 3-D groundwater transport model will be used to estimate groundwater concentrations between monitoring wells and in areas of low well density. Thus, spacing of boreholes/monitoring wells should be sufficient to keep model uncertainty within acceptable limits.	The statistical parameter of interest when estimating VOC concentrations in groundwater is the maximum detected concentration at a single well over a specified time period (e.g. one year). VOC concentration trends over a longer time period (i.e. multiple years) will also be evaluated to understand plume behavior and to monitor plume stability. A 3D visualization and a numerical groundwater model will be utilized to incorporate groundwater VOC concentration data into the CSM and to estimate the extent of PCE and degradation products in groundwater. If professional judgment shows too much uncertainty the interpretation of the lateral and vertical extent of PCE and degradation products in specific locations, additional data collection will be considered.	VOC concentrations in groundwater vary across the Site due to hydrogeologic conditions/features that influence groundwater flow paths and transport of contaminants from the source area to the downgradient groundwater plume. Thus, measured groundwater data should be spatially representative (i.e. laterally and vertically) of the varied hydrogeologic units at the Site. Data should also be spatially representative within the plume extent (i.e. source area, plume centerline, perimeter, toe of plume) to define the distribution of PCE and degradation products. An adequately spaced monitoring network is required to delineate the plume horizontally and vertically, and to monitor plume behavior and stability. Maximum concentrations of PCE and degradation products in groundwater at individual monitoring wells will be used in conjunction with a numerical groundwater flow and solute transport model to delineate the	Prior to initiation of the Phase 2 investigation, an evaluation of data from Phase 1 will be completed to identify data gaps in the estimation of the lateral and vertical extent of PCE and degradation products in groundwater. This evaluation will utilize a 3D groundwater transport model to incorporate Phase 1 groundwater VOC concentration data into the CSM. This information will be used to finalize or adjust proposed Phase 2 investigation locations, if necessary. Phase 2 investigation activities and proposed investigation locations are presented in Section 4.2 and Section 5 of this RI work plan. The specific data collection activities during Phase 2 include: ▪ Installation of new monitoring wells to define the northern and western extent of the plume. ▪ Installation of step-out wells where Phase 1 perimeter wells indicate VOC concentrations exceeding MCLs or screening levels.

2 - Principal Study Question	3 - Information Inputs (1)	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
		VOC concentrations in groundwater vary over time due to migration of contaminants in the subsurface, and seasonal fluctuations in groundwater levels (e.g. due to infiltration from rainfall and irrigation and/or pumping from agricultural wells) that can alter groundwater flow paths and flow rates. Thus, measured VOC concentrations should be adequately representative of the full range of expected concentrations both within and between years. At a minimum, collection of data over the course of a calendar year is desired.		lateral and vertical extent of groundwater containing VOCs and to predict concentrations in areas of low well density. Professional judgment and model uncertainty will be used to determine if the interpolated distribution of PCE and degradation products in groundwater shows data gaps and too much uncertainty. Data to support this study question will be collected in accordance with standard operating procedures presented in the QAPP to reduce the potential for sampling and measurement error and reduce uncertainty in the values.	▪ Installation of monitoring wells in the ESS area to define the lateral and vertical extent of PCE and degradation products and the depth to impacted groundwater in areas susceptible to VI. ▪ Installation of additional borings and wells to delineate VOC impacts around Buildings 6/7 and Sunnyside Park. ▪ Collection of multiple rounds of groundwater VOC concentration data from all new and existing monitoring wells.
E3 (Plume Mass Discharge) What is the mass discharge (2) of PCE in groundwater at the source area and in the downgradient groundwater plume (i.e., mid plume and toe of plume)? ***Estimation Statement:*** The mass discharge of PCE that is occurring in the source area and in the downgradient groundwater plume is to be estimated during development of the CSM using PCE concentration and groundwater velocity data in conjunction with groundwater modeling techniques. Mass discharge estimates quantify source or plume strength at a given time and location and will be used to improve evaluation of natural attenuation and active remedial alternatives. These data will also improve assessment of risks posed by contamination to downgradient receptors, such as wells or surface water bodies.	The following information is needed to determine the mass discharge of PCE in the source area and downgradient groundwater plume: ▪ PCE concentrations in groundwater at monitoring wells in the source area and downgradient plume ▪ Well depth and screen interval ▪ Lithology and hydrostratigraphy data ▪ Groundwater (seepage) velocity estimates ▪ Water levels and horizontal and vertical hydraulic gradients ▪ Aquifer test results and estimated hydrogeological properties including transmissivity, hydraulic conductivity, and storativity The mass discharge of PCE in groundwater should be derived using measured hydrogeologic and groundwater data collected during all phases of the RI (i.e. AOU1 RI, Phase 1 OU2 RI, and Phase 2 OU1 RI).	The lateral study boundary is shown on **Figure 2-1**. The vertical interval of VOC impacts will be evaluated during this investigation. VOC concentrations in groundwater vary across the Site due to hydrogeologic conditions/features that influence groundwater flow paths and transport of contaminants. Thus, data should be spatially representative within the plume extent (i.e. source area, mid plume, toe of plume) to define the discharge of PCE at different locations within the plume. There is limited space for installation of additional monitoring wells in the vicinity of Buildings 6 and 7 on the VAMC campus (i.e. source area), thus installation of additional groundwater wells in this area may be physically constrained by the structures in this area. Additionally, the Site is located in a highly-developed, urban/residential area where structures may restrict access to investigation in other areas. The smallest estimation unit for estimating the discharge of PCE in groundwater is a monitoring well transect comprised of at least 3 monitoring wells.	The statistical parameter of interest when estimating concentrations of PCE in groundwater is the maximum detected concentration at a single well over a specified time period. When estimating groundwater velocity, the statistical parameter of interest is the mean at a single monitoring well or monitoring location over a specified time period. Vertical transects will be created at some well locations by installing wells screened in multiple hydrogeologic units. The mass discharge of PCE will be estimated at the source area, mid plume, and the toe of the plume using measured and estimated groundwater velocity and PCE concentration data in conjunction with a numerical groundwater model and professional judgement.	Adequately located monitoring well transects (i.e. source area, mid plume, and toe of plume), oriented perpendicular to the direction of groundwater flow, are required to estimate the discharge of PCE in groundwater at the source area and in the downgradient groundwater plume. The transect method will be used to estimate mass discharge, in which individual monitoring points are used to integrate concentration and flow data. Mass discharge will be estimated at various locations in the plume by analyzing new and existing flow rate and VOC concentration data along transects oriented perpendicular to isocontours (or along transects using existing monitoring wells), as wells as by using a solute transport model requiring flow and concentration data as input parameters. Data to support this study question will be collected in accordance with standard operating procedures presented in the QAPP to reduce the potential for sampling and	Prior to initiation of the Phase 2 investigation, an evaluation of data from Phase 1 will be completed to identify data gaps in the estimation of the lateral and vertical extent of PCE in groundwater. This evaluation will utilize a 3D groundwater transport model to incorporate Phase 1 groundwater VOC concentration data into the CSM. If the 3D model or professional judgment indicates too much uncertainty in specific locations, additional data collection will be considered in Phase 2. Collection of additional data may include: ▪ Installation of one or more new monitoring wells in the source area (i.e. VAMC and Sunnyside Park) to complete a well transect for evaluation of PCE mass discharge. ▪ Completion of a monitoring well transect upgradient of the ESS area to evaluate PCE mass discharge at the toe of the plume. ▪ Collection of groundwater VOC concentration data from new and existing monitoring wells.

2 - Principal Study Question	3 - Information Inputs [1]	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
				measurement error and reduce uncertainty in the values.	▪ Collection of lithology, hydrostratigraphy, and borehole geophysical data from new monitoring wells. ▪ Measurement of hydraulic conductivity at transect well locations ▪ Collection of water level data from new and existing wells and calculation of hydraulic gradients
E4 (Natural Attenuation) How does natural attenuation change the concentrations of PCE and degradation products in the source area vadose zone and downgradient groundwater plume? ***Estimation Statement:*** The extent of natural attenuation that is occurring in the source area vadose zone and/or downgradient groundwater plume is to be estimated using both direct and indirect measurements. Direct measurements provide an estimation of the reduction in PCE and degradation products, while indirect measurements provide an estimation of the potential of attenuation to occur and how complete the attenuation process may be. Degradation mechanisms will also be compared with VOC data to evaluate if decreasing concentrations of total VOCs or individual VOC constituents are observed alongside evidence of biotic/abiotic attenuation mechanisms.	The following information is needed to determine whether natural attenuation is occurring in the source area vadose zone and/or downgradient groundwater plume: ▪ Vadose and saturated zone mineral properties (specifically ferrous iron minerals, magnetic susceptibility, and fraction of organic carbon) ▪ Stable isotope composition of source mass and dissolved PCE (and degradation products) ▪ Reduction/oxidation geochemical data and dissolved oxygen ▪ Biological data supporting the assessment of reductive dechlorination ▪ Groundwater (seepage) velocity estimates incorporating aquifer hydraulic properties, horizontal and vertical gradients ▪ Temporal and spatial concentrations of VOCs in groundwater and estimates of plume mass to determine if the plume is stable, expanding, or retracting The analytical method for VOCs should be selected such that the RL for each VOC is below its respective MCL or applicable screening level for decision-making. The evaluation of natural attenuation potential of PCE in groundwater should be derived using measured data collected during all phases of the RI (i.e. AOU1 RI, Phase 1 OU2 RI, and Phase 2 OU1 RI).	The lateral study boundary is shown on **Figure 2-1**. The vertical interval of VOC impacts will be evaluated during this investigation. Aquifer conditions and VOC concentrations in groundwater vary across the Site due to various factors that influence geochemical parameters and transport of contaminants from the source area to the downgradient groundwater plume. Thus, measured geochemical data should be spatially representative (i.e. laterally and vertically) within the plume boundary. Aquifer conditions also vary over time due to seasonal fluctuations in groundwater levels (e.g. due to rainfall and irrigation) that can alter geochemical conditions in groundwater. Thus, measured geochemical data should be adequately representative of the full range of expected aquifer conditions both within and between years. For performance of CSIA, monitoring wells should be selected with concentrations at or above 5 parts per billion (ppb) for proper analysis of isotope composition and evaluation of the extent of biotic and abiotic degradation occurring in the plume. The smallest decision unit for making decisions regarding the occurrence of natural attenuation should be a distinct plume area or hydrogeologic unit (e.g., source area vadose zone). Because attributes that influence natural attenuation processes can vary	The statistical parameter of interest when estimating concentrations of PCE and degradation products in groundwater or soil is the maximum detected concentration at a single well or sampling location. In assessing natural attenuation occurrence and potential, biodegradation is the most important destructive attenuation mechanism, although abiotic destruction of some compounds can occur. Other, nondestructive attenuation mechanisms can also occur, including sorption, dispersion, dilution from recharge, and volatilization. Site data will be evaluated using the following lines of evidence as outlined in the OSWER Directive 9200.4-17 (1997). (1) Historical ground water and/or soil chemistry data that demonstrate a clear and meaningful trend of decreasing contaminant mass and/or concentration over time at appropriate monitoring or sampling points. In the case of a ground water plume, decreasing concentrations should not be solely the result of plume migration. (2) Hydrogeologic and geochemical data that can be used to indirectly demonstrate the type(s) of natural attenuation processes active at the site, and the rate at which such processes will reduce contaminant concentrations to required levels. For	Representative groundwater VOC concentration data, as well as other data noted in Step 3, are required to estimate the extent of natural attenuation of PCE that is occurring in the source area vadose zone and the downgradient groundwater plume. This evaluation will be conducted using multiple lines of evidence to determine the extent to which natural attenuation changes the concentrations of PCE and degradation products in the source area vadose zone and downgradient groundwater plume, and the controlling mechanisms (both destructive and nondestructive). For example, even if PCE concentrations are not decreasing in groundwater, combined CSIA evaluation and microbial analysis can be used to indicate that biotic or abiotic degradation is occurring at the Site, and that other mechanisms (e.g., diffusion from soil) could be causing groundwater PCE concentrations to remain stable.	Prior to initiation of the Phase 2 investigation, an evaluation of data from Phase 1 will be completed to identify data gaps for determining if natural attenuation is occurring at the Site and what mechanisms control attenuation. The specific data collection activities during Phase 2 include: ▪ Collection of geochemical data from all monitoring wells to provide a temporal and spatial distribution of geochemical conditions to evaluate the potential of abiotic or biotic degradation ▪ Collection of groundwater samples at existing and newly-installed wells to evaluate VOC concentrations and aquifer geochemistry, and to evaluate VOC trends over time, ▪ Collection of groundwater samples from select wells for compound-specific isotope analysis to evaluate attenuation of VOCs across the plume. ▪ Collection of subsurface soil data for total ferrous minerals analysis, and magnetic susceptibility to support evaluation of abiotic attenuation mechanisms.

2 - Principal Study Question	3 - Information Inputs [1]	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
		spatially, data from each unit or area must either be measured or predicted with reasonable certainty.	example, characterization data may be used to quantify the rates of contaminant sorption, dilution, or volatilization, or to demonstrate and quantify the rates of biological degradation processes occurring at the site. (3) Data from field or bench scale studies that directly demonstrate the occurrence of a particular natural attenuation process at the site and its ability to degrade the contaminants of concern (e.g. biological degradation processes).		
D1 (Source Mass) Is there sufficient mass of PCE in the vadose zone in the source area to act as an ongoing source of PCE in groundwater? ***Alternative study outcomes:*** ▪ If yes: Evaluation of response actions would need to consider alternatives that include source treatments to address contaminant mass in the vadose zone. ▪ If no: The evaluation of response actions may not need to include a detailed analysis of alternatives that include source treatment. ***Decision Statement:*** Evaluate whether there is sufficient source mass of PCE in the vadose zone to act as an ongoing source of PCE in groundwater. Delineation of PCE in the vadose zone and determination of PCE mass discharge at the source area will be used in the evaluation. If soil-to-groundwater migration is found to be occurring, evaluation of response actions would need to consider alternatives that include source treatments to address contaminant mass in the vadose zone.	The following information is needed to determine the PCE mass in the source area contributing to an increase in dissolved concentrations: ▪ Source area temporal and spatial groundwater VOC concentration data ▪ Source area spatial soil VOC data ▪ Source area spatial and temporal soil gas VOC data ▪ Vadose zone and saturated zone lithological and hydraulic data The analytical method for VOC analysis should be selected such that the RL for each VOC is below its respective MCL or applicable screening level for decision-making. The source mass evaluation should be derived using measured data collected during all phases of the RI (i.e. AOU1 RI, Phase 1 OU2 RI, and Phase 2 OU1 RI).	Currently the source area is not well defined, therefore the lateral extent of the study area is the vicinity of the VAMC Buildings 6 and 7 and the Sunnyside Park sewer line (**Figure 2-2**). The vertical extent of subsurface investigation is limited to the vadose and saturated groundwater zones beneath the source area to a depth of approximately 400 feet. There is limited space for installation of additional monitoring wells in the vicinity of Buildings 6 and 7 on the VAMC campus, thus installation of additional groundwater wells in this area may be physically constrained. VOC concentrations in groundwater vary over time due to migration of contaminants in the subsurface, and seasonal fluctuations in groundwater levels (e.g. due to infiltration from rainfall and irrigation and/or pumping from agricultural wells) that can alter groundwater velocities and flow paths. VOC concentrations in soil gas also vary over time, thus, measured VOC concentrations should be adequately representative of the full range of expected concentrations both within and between years. The smallest decision unit for making source response decisions is the individual source area (i.e. Building 6 and 7 on the VAMC campus and the Sunnyside sewer line). Because these two distinct source areas likely have a different mass of PCE acting as a source	The statistical parameter of interest when estimating concentrations of PCE and degradation products in groundwater or soil gas is the maximum detected concentration at a single well or sampling point over a specified time period. Determination of the presence or absence of sufficient PCE mass in the vadose zone will be conducted using measured and estimated hydrogeologic and VOC concentration data in conjunction with a numerical groundwater model and professional judgement.	Representative source area groundwater, soil, and soil gas VOC concentration data, as well as lithologic and hydraulic data are required to determine if there is sufficient mass of PCE in the vadose zone in the source area to act as an ongoing source of PCE in groundwater. This evaluation will be conducted using multiple lines of evidence to determine if source mass is present and if it acts as an ongoing source for groundwater. For example, lines of evidence indicating the absence of sufficient source mass in the vadose zone may include: ▪ Maximum concentrations of VOCs in source area soil that are less than the soil screening level protective of groundwater ▪ Soil gas VOC concentration data that demonstrate a meaningful decreasing concentration trend over time at source area sampling points ▪ Recent groundwater VOC concentration data indicating concentrations are less than the MCL over multiple quarters or sampling events ▪ Groundwater VOC concentration data that demonstrate a meaningful decreasing concentration trend	Prior to initiation of the Phase 2 investigation, an evaluation of data from Phase 1 will be completed to identify data gaps for determining if there is sufficient mass of PCE in the source area to act as an ongoing source of PCE in groundwater. Collection of additional data to support this evaluation in Phase 2 may include: ▪ Sampling existing source area soil gas probes. ▪ Collecting vadose zone soil samples from new borings/monitoring wells near source areas.

2 - Principal Study Question	3 - Information Inputs [1]	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
		to groundwater, data from each area must either be measured or predicted with reasonable certainty.		over time at source area monitoring wells ▪ Mass discharge estimates and/or groundwater modeling results indicating low plume strength in the source area Alternatively, the absence of some or all of these lines of evidence may indicate that there is sufficient contaminant mass in the vadose zone to act as an ongoing source to groundwater, and evaluation of response actions may need to consider alternatives that include source treatments to address contaminant mass in the vadose zone.	
D2 (Source Area Vapor Intrusion Risk) Would human exposure to site-related VOCs in the source area vadose zone via VI result in unacceptable risks? ***Alternative study outcomes:*** ▪ If yes: Evaluation of response actions would need to consider alternatives that include source treatments to address contaminant mass in the vadose zone. ▪ If no: The evaluation of response actions may not need to include a detailed analysis of alternatives that include source treatment. ***Decision Statement:*** Evaluate whether human exposure to site-related VOCs in the source area vadose zone near VAMC Buildings 6 and 7 by way of VI would result in unacceptable risks. If unacceptable risks are identified, evaluation of response actions would need to consider alternatives that include source treatments to mitigate VI risks.	The following information is needed to quantify human exposures and risks from source area VOCs in soil gas: ▪ Measured concentrations of VOCs in soil gas near and beneath VAMC Buildings 6 and 7 ▪ Measured concentrations of VOCs in indoor air at VAMC Buildings 6 and 7 ▪ Exposure parameters for human receptor populations ▪ Toxicity thresholds for evaluating non-cancer and cancer risks for human populations The analytical method should be selected such that the RL for each VOC is below its respective concentration-based toxicity threshold for decision-making. Exposure point concentrations (EPCs) for VOCs in soil gas and indoor air should be derived using measured data collected during all phases of the RI (i.e. AOU1 RI, Phase 1 OU2 RI, and Phase 2 OU1 RI). Exposure parameters for the human receptor populations of interests should be based on USEPA default exposure assumptions, derived from national exposure data (e.g., USEPA Exposure Factors Handbook), site-specific data, or using best professional judgement.	Potential human receptors for this site include indoor workers and other special populations (i.e., child care center and a residential scenario with the veterans home and Valor House) that may come in contact with source area contamination via VI and inhalation of indoor air at Buildings 6 and 7. The lateral study boundary is the area in and around VAMC Buildings 6 and 7. When quantifying air exposures inside buildings, because there can be indoor sources of VOCs (not related to source area soil/soil gas contamination), such as dry cleaned clothing, brake cleaners, and glues, characterizing background levels of VOCs from these non-site-related indoor sources is useful for interpreting site risks. The smallest decision unit for making risk management decisions should be a single building. Because each building can have building-specific attributes that influence indoor air concentrations, data from each potentially-impacted building must either be measured or predicted with reasonable certainty. VOC concentrations in indoor air can vary due to many factors, such as, but not limited to,	The statistical parameter of interest when estimating human exposures is the mean across the entire exposure area of interest and entire exposure timeframe of interest. The exposure area and timeframe depend upon the receptor of interest. For example, for residential indoor exposures, the exposure area is the house and the default exposure duration is 26 years (6 years as a child and 20 years as an adult). However, the EPC should represent the spatially- and time-weighted average. The USEPA RSL and VISLs will be used to compute non-cancer hazard quotients (HQs) and cumulative hazard indices (HIs) and cumulative cancer risk estimates. As appropriate, site-specific assumptions will be used to derive screening levels for the receptor-specific exposure scenario of interest. If estimated cancer risks are greater than 1E-04 and/or estimated HI is greater than 1, then risks will be deemed unacceptable and an evaluation of response actions would need to consider alternatives that include source treatments to mitigate VI risks. If estimated cancer risks are less than or equal to 1E-06 and/or estimated	The following are the null (H_0) and alternative (H_A) hypotheses for the evaluation of source area VI risks: H_0: Human exposures to site-related VOCs in the source area vadose zone via VI are greater than the level of concern. H_A: Human exposures to site-related VOCs in the source area vadose zone via VI are less than or equal to the level of concern. In making decisions about human health risks, two types of decision errors are possible: • A Type I (false negative) decision error would occur if a risk manager decides that VOC exposure is not of health concern, when, in fact, it is of concern (i.e., false rejection of the H_0 hypothesis). • A Type II (false positive) decision error would occur if a risk manager decides that VOC exposure is above a level of concern, when, in fact, it is not	Prior to initiation of the Phase 2 investigation, an evaluation of data from Phase 1 will be completed to identify additional data required to quantify risks from human exposure to site-related VOCs in source area soil gas via VI. Collection of additional data to support this evaluation in Phase 2 may include: ▪ Sampling existing source area soil gas probes. ▪ Collecting additional indoor air samples at VAMC Buildings 6 and 7.

2 - Principal Study Question	3 - Information Inputs (1)	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
	The toxicity thresholds used for risk management decision-making should be derived from two main sources of risk-based thresholds – the USEPA Regional Screening Levels (RSLs) and the USEPA Vapor Intrusion Screening Levels (VISLs).	source concentrations, building floor level, and air flow. For the purposes of risk estimation, it is most important to characterize air concentrations in rooms inside the building where the receptors have the highest exposure frequency. VOC concentrations in soil gas and indoor air have the potential to vary over time. For example, VOC concentrations in indoor air attributable to VI are likely to be highest in the winter and lowest in the summer. The human health risk assessment will quantify average long-term chronic exposures under both current and future site conditions. Thus, measured VOC concentrations should be adequately representative of the full range of expected concentrations both within and between years. If it is not possible to collect data to represent the full range of concentrations, to ensure decisions are risk-protective, data should be representative of time periods and locations that are likely to be from the high-end of the exposure distribution.	HI is less than or equal to 1, then risks will be deemed acceptable and a detailed analysis of remedial alternatives for the mitigation of source area contaminant mass would not be necessary. If estimated cancer risks are between 1E-06 and 1E-04, which is within the USEPA acceptable risk range, then risk managers may need to consider site-specific attributes to make appropriate risk management decisions.	(i.e., false acceptance of the H_0 hypothesis). Risk managers are most concerned about guarding against the occurrence of false negative decision errors, since an error of this type may leave humans exposed to unacceptable levels of VOCs. In general, the goal is to limit the probability of a false negative decision error to no more than about 5% (i.e., $\alpha = 0.05$). This is accomplished by using the 95% upper confidence limit of the arithmetic mean (95UCL) as the EPC, rather than the sample mean, in risk calculations. Use of the 95UCL to estimate risk helps account for limitations in the data, ensuring that risk estimates are more likely to overestimate than underestimate the true risk level. A minimum of 3 samples is needed to compute the 95UCL; however, the target number of samples to compute a reliable EPC is 8 to10 samples. The actual number of samples collected will be determined as part of the study design, taking into consideration the exposure scenario, exposure area size, anticipated spatial and temporal variability in concentrations, and project budget. If the number of samples collected is too small to calculate the 95UCL, the maximum concentration may be used in the risk evaluation as an estimate of the EPC. Risk managers are also concerned with the probability of making false positive decision errors. Although this type of decision error does not result in unacceptable human exposure, it may result in unnecessary expenditure of resources (i.e., in investigating and remediating source area soil that does not result in	

2 - Principal Study Question	3 - Information Inputs [1]	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
				unacceptable risks). The probability of a false positive decision error is greatest when the EPC is close to the decision threshold. In general, the goal is to limit the probability of a false positive decision error to no more than 20% (i.e., $\beta = 0.20$) when the true risk is within a factor of 2 of the level of concern. The required sample size to limit the false positive error rate will depend upon the underlying variability in the VOC concentrations for the medium of interest (i.e., the chemical-specific standard deviation). When the standard deviation is small, fewer samples are needed to limit the false positive error rate; when the standard deviation is large, a high number of samples are needed to limit the false positive error rate.	
D3 (Groundwater Risk) Would human exposures to site-related VOCs in groundwater within the plume area result in unacceptable risks? ***Alternative study outcomes:*** ▪ If yes: Evaluation of response actions would need to consider alternatives that mitigate exposure to VOCs in groundwater for the pathways where unacceptable risks were identified. ▪ If no: Evaluation of response actions would not include a detailed analysis of remedial alternatives for the mitigation of groundwater. ***Decision Statement:*** Evaluate whether human exposures to site-related VOCs in groundwater would result in unacceptable risks. If unacceptable risks are identified an evaluation of response actions would need to consider alternatives that mitigate exposure to VOCs in groundwater for the pathways where unacceptable risks were identified.	The following information is needed to quantify human exposures and risks from VOCs in groundwater: ▪ Measured concentrations of VOCs in groundwater from aquifers that could be used as drinking or irrigation water under current or future conditions ▪ Measured concentrations of VOCs in groundwater that could be a source of VI exposures ▪ Measured concentrations of VOCs in soil gas that could be a source of VI exposures ▪ Measured concentrations of VOCs in indoor air inside buildings that could be impacted by VI ▪ Measured concentrations of VOCs in shallow groundwater in the ESS area that could be a source of exposure for construction workers ▪ Exposure parameters for human receptor populations and pathways of potential concern for groundwater exposure scenarios ▪ Toxicity thresholds for evaluating non-cancer and cancer risks for human populations The analytical method should be selected such that the RL for each VOC is below its respective	Potential human receptors for this site include residents, building occupants, and construction workers that may contact contaminated groundwater via a drinking water supply well or through VI and direct contact pathways associated with shallow groundwater. The lateral study boundary is shown on **Figure 2-1**. Within the study area, the exposure areas of interest are determined by the groundwater plume extent. The exposure areas of interest include the ESS neighborhood, as well as other locations and buildings outside this neighborhood within the current footprint of the known groundwater plume (see **Figure 3-2**). However, because the lateral extent of the groundwater plume has not been fully defined, the spatial bounds of the exposure areas could expand or be reduced as more information about the Site is obtained. When quantifying air exposures inside buildings, because there can be indoor sources of VOCs (not related to groundwater	The statistical parameter of interest when estimating human exposures is the mean across the entire exposure area of interest and entire exposure timeframe of interest. The exposure area and timeframe depend upon the receptor of interest as discussed above. The EPC should represent the spatially- and time-weighted average. The USEPA RSL and VISLs will be used to compute non-cancer hazard quotients (HQs) and cumulative hazard indices (HIs) and cumulative cancer risk estimates. As appropriate, site-specific assumptions will be used to derive screening levels for the receptor-specific exposure scenarios of interest. If estimated cancer risks are greater than 1E-04 and/or estimated HI is greater than 1, then risks will be deemed unacceptable and an evaluation of response actions would need to consider alternatives that mitigate exposure to VOCs in groundwater for the pathways where unacceptable risks were identified. If estimated cancer	The following are the null (H_0) and alternative (H_A) hypotheses for the evaluation of groundwater risks: H_0: Human exposures to site-related VOCs in groundwater within the plume area are greater than the level of concern. H_A: Human exposures to site-related VOCs in groundwater within the plume area are less than or equal to the level of concern. As described above, to avoid the potential for a false negative decision error, the 95UCL on the mean should be used as the basis of the EPC for both human health and terrestrial ecological receptors. Use of the 95UCL limits the probability of a false negative decision error to no more than about 5%. The minimum of samples to compute the 95UCL is 3 samples; however, 8 to10 samples would be needed to compute a	Prior to initiation of the Phase 2 investigation, an evaluation of data from Phase 1 will be completed to identify additional data required to quantify human exposures and risks from VOCs in groundwater. Collection of additional data to support this evaluation in Phase 2 may include: ▪ Sampling existing soil gas probes. ▪ Collecting indoor air samples inside buildings that could be impacted by VI ▪ Collecting multiple rounds of groundwater VOC concentration data from new and existing monitoring wells

2 - Principal Study Question	3 - Information Inputs [1]	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
	concentration-based toxicity threshold for decision-making. EPCs for VOCs in various media should be derived using measured data collected during all phases of the RI (i.e. AOU1 RI, Phase 1 OU2 RI, and Phase 2 OU1 RI). Exposure parameters for the human receptor populations of interests should be based on USEPA default exposure assumptions, derived from national exposure data (e.g., USEPA Exposure Factors Handbook), site-specific data, or using best professional judgement. The toxicity thresholds used for risk management decision-making should be derived from two main sources of risk-based thresholds – the USEPA Regional Screening Levels (RSLs) and the USEPA Vapor Intrusion Screening Levels (VISLs).	contamination or source area soil/soil gas contamination), such as dry cleaned clothing, brake cleaners, and glues, characterizing background levels of VOCs from these non-site-related indoor sources is also useful for interpreting site risks. The smallest decision unit for making risk management decisions should be a single building (e.g., one residential home). Because each building can have building-specific attributes that influence indoor air concentrations, data from each potentially-impacted building must either be measured or predicted with reasonable certainty. Risks from VI will be quantified for properties within the groundwater plume boundary where there is the potential for VI to occur.	risks are less than or equal to 1E-06 and/or estimated HI is less than or equal to 1, then risks will be deemed acceptable and a detailed analysis of remedial alternatives for the mitigation of groundwater would not be necessary. If estimated cancer risks are between 1E-06 and 1E-04, which is within the USEPA acceptable risk range, then risk managers may need to consider site-specific attributes to make appropriate risk management decisions for groundwater.	reliable EPC. The goal is to limit the probability of a false positive decision error to no more than 20% when the true risk is within a factor of 2 of the level of concern.	
D4 (Surface Water Risk) [3] Would human and ecological exposures to site-related VOCs in surface water (i.e., springs, creeks, ponds, irrigation water) within the groundwater plume area result in unacceptable risks? ***Alternative study outcomes:*** ▪ If yes: Evaluation of response actions would need to consider alternatives that mitigate exposure to VOCs in surface water for the pathways and receptors where unacceptable risks were identified. ▪ If no: Evaluation of remedial alternatives would not include a detailed analysis of remedial alternatives for the mitigation of surface water. ***Decision Statement:*** Evaluate whether human and/or ecological exposures to site-related VOCs in surface water would result in unacceptable risks. If unacceptable risks are identified an evaluation of response actions would need to consider alternatives that mitigate exposure to VOCs in surface water for the pathways and receptors where unacceptable risks were identified.	The following information is needed to quantify human and ecological exposures and risks from VOCs in surface water: ▪ Measured concentrations of VOCs in surface water (i.e., springs, creeks, ponds, irrigation water) that could be impacted by contaminated VOCs associated with the site ▪ Exposure parameters for human receptor populations and pathways of potential concern for surface water exposure scenarios ▪ Toxicity thresholds for evaluating non-cancer and cancer risks for human populations ▪ Toxicity thresholds for evaluating exposures by aquatic receptors, plants, birds, and mammals The analytical method should be selected such that the RL for each VOC is below its respective concentration-based toxicity threshold for decision-making. EPCs for VOCs in surface water should be derived using measured data collected during all phases of the RI (i.e. AOU1 RI, Phase 1 OU2 RI, and Phase 2 OU1 RI). Exposure parameters for the human receptor populations of interests should be based on USEPA default exposure assumptions, derived from	Potential human receptors for this site include residents and outdoor workers that may contact contaminated surface water (i.e., springs, creeks, ponds, irrigation water). Potential ecological receptors may include aquatic receptors (e.g., aquatic plants, invertebrates, fish), terrestrial plants, birds, and mammals (both wildlife and domesticated pets) that may contact contaminated surface water. The lateral geographic boundary of the study area is shown on **Figure 2-1**. Within the study area, the exposure areas of interest are determined by the groundwater plume extent and the locations where impacted surface water is present. This includes both areas where contaminated shallow groundwater is surfacing, such as seeps and springs, as well as creeks and ponds that are infiltrated by contaminated shallow groundwater. The exposure areas of interest include the springs, creeks, and ponds within the ESS neighborhood, as well as other surface water locations outside this neighborhood within the current footprint of the known groundwater plume (**Figure 3-2**). The exposure areas of	**Human Health:** The statistical parameter of interest when estimating human exposures is the mean across the entire exposure area of interest and entire exposure timeframe of interest. The exposure area and timeframe depend upon the receptor of interest. The EPCs will be used to compute non-cancer HQs and HIs and cumulative cancer risk estimates. As appropriate, site-specific assumptions will be used to derive risk estimates for the receptor-specific exposure scenarios of interest. If estimated cancer risks are greater than 1E-04 and/or estimated HI is greater than 1, then risks will be deemed unacceptable and an evaluation of response actions would need to consider alternatives that mitigate exposure to VOCs in surface water for the pathways where unacceptable risks were identified. If estimated cancer risks are less than or equal to 1E-06 and/or estimated HI is less than or equal to 1, then risks will be deemed acceptable and a detailed analysis of remedial alternatives for the mitigation	The following are the null (H_0) and alternative (H_A) hypotheses for the evaluation of surface water risks: H_0: Human and/or ecological exposures to site-related VOCs in surface water within the plume area are greater than the level of concern. H_A: Human and ecological exposures to site-related VOCs in surface water within the plume area are less than or equal to the level of concern. As described above, to avoid the potential for a false negative decision error, the 95UCL on the mean should be used as the basis of the EPC for both human health and terrestrial ecological receptors. Use of the 95UCL limits the probability of a false negative decision error to no more than about 5%. The minimum of samples to compute the 95UCL is 3 samples; however, 8 to10 samples would be needed to compute a reliable EPC. The goal is to limit the probability of a false positive decision	Prior to initiation of the Phase 2 investigation, an evaluation of data from Phase 1 will be completed to identify additional data required to quantify human exposures and risks from VOCs in surface water. Collection of additional data to support this evaluation in Phase 2 may include: ▪ Collecting surface water VOC concentration data from springs, creeks or ponds that could be impacted by contaminated VOCs associated with the site

2 - Principal Study Question	3 - Information Inputs (1)	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
	national exposure data (e.g., USEPA Exposure Factors Handbook), site-specific data, or using best professional judgement. The human health toxicity thresholds used for risk management decision-making should be derived from the USEPA RSLs and the USEPA National Ambient Water Quality Criteria (NAWQC) for human health. The ecological toxicity thresholds should be based on USEPA NAWQC for aquatic life (i.e., both the criterion maximum concentration [CMC] and the criterion continuous concentration [CCC]) and other commonly used toxicity benchmark sources in the available scientific literature (e.g., Los Alamos National Laboratory ECORISK Database screening levels for surface water).	interest also include locations where impacted groundwater is being used for surface irrigation. As noted above, because the lateral extent of the groundwater plume has not been fully defined, the lateral bounds of the exposure areas could expand or be reduced as more information about the Site is obtained. The smallest decision unit for making risk management decisions should be a single property (e.g., for residential exposure scenarios), a single well (e.g., for irrigation water), or a unique surface water feature (e.g., a specific spring, creek, or pond). If there are spatial gradients in concentration within a surface water feature, it may be appropriate to split the feature into smaller exposure areas. VOC concentrations in surface water have the potential to vary over time. Additionally, there are also differences in the exposure potential as a function of time. Terrestrial receptors, such as humans, birds, and mammals, are likely to have more frequent exposure to surface water in the spring and summer, and less exposure during the fall and winter when surface water features are absent or frozen. The human health risk assessment will quantify average long-term chronic exposures under both current and future site conditions. Thus, measured VOC concentrations should be adequately representative of the full range of expected concentrations both within and between years. If it is not possible to collect data to represent the full range of concentrations, to ensure decisions are risk-protective, data should be representative of time periods and locations that are likely to be from the high-end of the exposure distribution.	of surface water for human health would not be necessary. If estimated cancer risks are between 1E-06 and 1E-04, which is within the USEPA acceptable risk range, then risk managers may need to consider site-specific attributes to make appropriate risk management decisions for surface water. **Aquatic Ecological Receptors:** The statistical parameter of interest when estimating aquatic ecological receptor exposures is the mean across the entire exposure area of interest; however, the exposure timeframe of interest depends upon the basis of the toxicity threshold. A four-day averaging period should be used when evaluating chronic exposures, while a one-hour averaging period should be used when evaluating acute exposures. Ideally, continuous surface water monitoring data would be collected that allows for the computation of both the four-day and one-hour averages. If continuous monitoring data cannot be collected, each sample should be evaluated as representing both the four-day average and the one-hour average and risks interpreted based on the frequency and magnitude of CMC and CCC exceedances. **Terrestrial Ecological Receptors:** The statistical parameter of interest when estimating terrestrial ecological exposures is the mean across the entire exposure area of interest and entire exposure timeframe of interest. Similar to human health, for terrestrial ecological receptors, the goal is to quantify long-term chronic exposures (e.g., year-long). For ecological receptors (aquatic and terrestrial), if estimated HQs are greater than 1, then ecological risks will be deemed unacceptable and an evaluation of response actions may need to consider alternatives that mitigate exposure to VOCs in surface water for the pathways	error to no more than 20% when the true risk is within a factor of 2 of the level of concern.	

2 - Principal Study Question	3 - Information Inputs [1]	4 - Study Boundaries	5 - Analytical Approach	6 - Performance/Acceptance Criteria	7 - Plan for Obtaining Data
			where unacceptable risks were identified. If estimated HQs are less than or equal to 1, then risks will be deemed acceptable and a detailed analysis of remedial alternatives for the mitigation of surface water would not be necessary for ecological receptors.		

[1] Step 3 of the DQO process identifies all of the data inputs required to answer the principle study questions. In some cases, adequate data for inputs identified in this step may be available from previous investigations (i.e. AOU1 RI and OU2 Phase 1 RI) and may not require collection of additional data in Phase 2.

[2] Mass flux is a rate measurement specific to a defined area, which is usually a subset of a plume cross section. Mass flux is expressed as mass/time/area (e.g., grams/day/square meter). Mass discharge is an integrated mass flux estimate (i.e., the sum of all mass flux measures across an entire plume) and represents the total mass of any solute conveyed by groundwater through a defined plane. Mass discharge is expressed as mass/time (e.g., grams/day).

[3] Evaluation of surface water risk will only be considered for areas not evaluated in the AOU1 HHRA and SLERA (EA 2019).

Appendix C
Laboratory Quality Assurance Manuals

EMAX QUALITY SYSTEMS MANUAL 2019 Rev. 5.3

EMAX-QS00

This manual is considered confidential within EMAX. The Quality Systems Manual is available for use by laboratory personnel through the EMAX browser and by means of controlled distribution. The procedure for controlled distribution of this manual is detailed in EMAX-DM02, Controlled Documents. The manual must not be altered other than by a duly authorized representative of EMAX.

If the document has been provided to external users or regulators, it is for the exclusive purpose of reviewing EMAX quality systems. The external party or parties shall not use it in any other way without the prior written permission of an authorized representative of EMAX Laboratories, Inc.

EMAX
LABORATORIES, INC.

1835 W. 205TH ST., TORRANCE, CA 90501
310-618-8889 (TEL)
310-618-0818 (FAX)
info@emaxlabs.com (email)
www.emaxlabs.com (web)

QUALITY SYSTEMS MANUAL

EFFECTIVE DATE: SEPTEMBER 09, 2019

DOCUMENT ID: EMAX-QS00

REVISION 5.3

APPROVED BY:

KENETTE PIMENTEL
QUALITY ASSURANCE & DATA VALIDATION MANAGER
(SIGNATURE)

YE MYINT
TECHNICAL OPERATIONS MANAGER
(SIGNATURE)

CASPAR PANG
CHIEF EXECUTIVE OFFICER & LAB DIRECTOR
(SIGNATURE)

Preface

EMAX Quality Systems (EMAX-QS00) was designed to guide its Laboratory Operations and Data Validation to deliver data conformant to environmental industry standards.

EMAX Laboratories, Inc., (EMAX) has provided comprehensive environmental laboratory services to governmental, state and local agencies since 1987. EMAX was founded to fill a need within the California marketplace for a flexible, reliable, and cost-effective analytical laboratory for consulting firms involved in the early stages of site investigation and remediation. EMAX has also provided fundamental services for the development of statewide and regional environmental compliance, enforcement, and remedial action programs.

Similarly, EMAX had the opportunity to provide large-scale analytical support to the initial and then burgeoning environmental restoration programs of the branches of the Department of Defense, Department of Energy, and United States Environmental Protection Agency. EMAX occupies a state-of-the-art, 27,000 square foot physical plant in Torrance, California.

The industry EMAX supports has undergone periods of growth, uncertainty, and consolidation. EMAX however, has maintained levels of stability and consistency. With a strong dedication to the continuation of our technical proficiency, we place extreme emphasis on in-house training, third-party review of procedures and protocols, and the audit and peer-review processes.

Over the years, EMAX has distinguished itself within the laboratory services industry by its stability in staff, its recognized technical proficiency, and its commitment to providing analytical deliverables in a wide variety of either agency-specified or client-developed reporting formats. We must share a large portion of our success in these areas with our clients, for it is a product of their dedication, input, and communication with us.

EMAX, a Small Disadvantaged Business (SDB), has been evaluated and audited by numerous governmental agencies and private-sector clients, and has provided analytical services in support of the overall environmental programs of the

- United States Environmental Protection Agency
- United States Air Force, The Air Force Center for Environmental Excellence
- United States Army, Army Corps of Engineers
- United States Navy, and
- United States Department of Energy

Therefore, it is incumbent for EMAX to update this manual to conform with the requirements of The NELAC Institute (TNI), EL-V1-2011, Modules 1, 2 and 4 and the DoD Quality Systems Manual (DoD/DOE QSM) for Environmental Laboratories, Version 5.3, Modules 1, 2 and 4 and Appendices A, B and C.

Updates

The following sections were updated in EMAX-QS00 Rev.5.3:

- Vol. 1 Mod. 2 Sec. 4.4 Review of Requests, Tenders and Contracts
- Vol. 1 Mod. 2 Sec. 4.16 Data Integrity Investigations
- Vol. 1 Mod. 2 Sec. 5.9. Quality Assurance of Environmental Testing
- Appendix A References
- Appendix B EMAX Organization and Management

TABLE OF CONTENTS

VOLUME 1, MODULE 1, PROFICENCY TESTING 8

1.0 INTRODUCTION 9
2.0 REQUIREMENTS FOR ACCREDITATION 9
2.1. Initial Accreditation 9
2.2. Continuing Accreditation 9
3.0 DOE Requirements for Participation 10
3.1. Initial Inclusion 10
3.2. Continued Participation 11

VOLUME 1, MODULE 2, QUALITY SYSTEMS GENERAL REQUIREMENTS 12

1.0 INTRODUCTION, SCOPE AND APPLICABILITY 13
2.0 REFERENCES 13
3.0 TERMS AND DEFINITIONS 13
4.0 MANAGEMENT REQUIREMENTS 13
4.1. Organization 13
4.2. Management Systems 15
4.3. Document Control 21
4.4. Review of Requests, Tenders and Contracts 21
4.5. Subcontracting Environmental Tests 23
4.6. Purchasing Services and Supplies 23
4.7. Service to the Customer 24
4.8. Complaints 24
4.9. Control of Non-conforming Environmental Testing Work 24
4.10. Improvement 25
4.11. Corrective Action 25
4.12. Preventive Action 27
4.13. Control of Records 27
4.14. Internal Audits 28
4.15. Management Review 29
4.16. Data Integrity Investigations 30
5.0 TECHNICAL REQUIREMENT 30
5.1. General 31
5.2. Personnel 31
5.3. Accommodation and Environmental Conditions 32
5.4. Environmental Methods and Method Validation 33
5.5. Equipment 35
5.6. Measurement Traceability 37
5.7. Collection of Samples 38
5.8. Handling Samples and Test Items 38
5.9. Quality Assurance of Environmental Testing 39
5.10. Reporting the Results 39
6.0 Hazardous and Radioactive Materials Management and Health and Safety Practices 42

TABLE OF CONTENTS

VOLUME 1, MODULE 4, QUALITY SYSTEMS CHEMICAL TESTING ____ 43

1.0 CHEMICAL TESTING ____ 44
1.1. INTRODUCTION ____ 44
1.2. SCOPE ____ 44
1.3. TERMS AND DEFINITIONS ____ 44
1.4. METHOD SELECTION ____ 44
1.5. METHOD VALIDATION ____ 44
1.6. DEMONSTRATION OF CAPABILITY (DOC) ____ 46
1.7. TECHNICAL REQUIREMENTS ____ 46

APPENDIX A REFERENCES
APPENDIX B EMAX ORGANIZATION AND MANAGEMENT
APPENDIX B-1 Management Structure
APPENDIX B-2 List of Standard Operating Procedures
APPENDIX B-3 List of Major Equipment
APPENDIX B-4 Accreditations
APPENDIX B-5 Health & Safety Practices

PRINT DRF

EMAX
QUALITY
SYSTEMS
MANUAL
2019
Rev. 5.3

VOLUME 1, MODULE 1, PROFICENCY TESTING

PROFICIENCY TESTING

1.0 INTRODUCTION

EMAX shall participate in proficiency testing (PT) as required by TNI and DoD standards. Generally, PT standards shall be purchased from ISO 17043 accredited PT providers.

2.0 REQUIREMENTS FOR ACCREDITATION

2.1. Initial Accreditation

2.1.1. Initial Accreditation for DoD ELAP

EMAX shall successfully complete at least two PT samples for each analyte-matrix-method where PT samples are commercially available.

2.1.2. PT Samples for Initial Accreditation

Primarily, EMAX shall obtain PT samples from PT providers that are accredited under International Organization for Standardization (ISO) 17043.
EMAX also participates in MAPEP PT studies. This PT participation can also be utilized for initial accreditation.

2.1.3. PT samples for non-ISO 17043 Accredited PT Provider

In the absence of ISO 17043 certified PT providers, EMAX shall obtain permission from the Accrediting Body to use a PT from a non-ISO 17043 accredited PT Provider prior to analyzing the PT sample.

2.1.4. PT Samples for Analyte-matrix-method not from a PT Provider

In the event that a particular analyte in a given method and/or matrix can not be obtained from any PT provider, EMAX shall inform the Accrediting Body in writing that no PT provider is available for that specific parameter. Other requirements to substantiate the PT requirements shall be duly fulfilled. EMAX shall continue to survey for its availability and shall participate with the PT study as expected.

2.1.5. Analysis Date of PT Samples

EMAX shall provide required PT results that are performed within 12 months, completed at least 15 calendar days apart.

2.1.6. PT Study Determination

For initial accreditation or additional analyte(s) desired to be added to an existing method-matrix accreditation, EMAX shall request from the PT provider to send PT reports directly to the Accrediting Body unless instructed otherwise.

2.1.7. Processing PT Samples

EMAX shall treat PT samples as regular field samples. They shall be received, logged and stored the same way as field samples. PT samples shall be prepared/extracted/digested (whichever is applicable) and analyzed with QC samples, e.g., method blank, lab control sample, etc., per method requirement. PT sample results shall be qualitatively and quantitatively evaluated as prescribed by the method.

2.2. Continuing Accreditation

2.2.1. Maintaining Accreditation

EMAX shall participate in PT studies at least twice a year at approximately six months apart (or >4 to <8 months apart). Regularly, EMAX joins the following PT round robins:

- Water Supply
- Water Pollution
- Soil / Hazardous Waste

- Air & Emissions
- Alaska Underground Storage Tank
- Arizona Hydrocarbons
- DOE MAPEP

2.2.2. EMAX PT History

EMAX had been participating with the above listed PT programs for more than 25 years and has successfully maintained a high success rate (98-100%).

EMAX shall maintain its practice to review PT reports from the providers upon receipt. Non-conforming results shall be assessed in accordance to EMAX-QA08, Corrective Action SOP. Resolution to the finding(s) shall be dealt accordingly and where necessary, preventive measures shall be instituted. In the event that the PT sample itself is the suspect for non-conformance, the provider shall be duly informed to take part in the root-cause analysis. Findings, corrective action and lessons learned drawn from root-cause-analysis shall be logged in the Non-Conformance database. Where new policies or procedures are enforced to correct the anomaly, all relevant personnel shall be involved and actions taken shall be properly documented. Corrective action shall be implemented to assure that resolution to finding(s) are in effect.

EMAX shall demonstrate due diligence that at least two out of the most recent three PTs shall successfully pass the PT acceptance criteria. EMAX shall retain PT records for at least five years.

2.2.3. Failure to Meet Criteria

It is of EMAX's best interest to maintain high score in Performance Testing. However, it is understood that failure to meet PT criteria at the frequency required, the analyte-method-matrix parameter shall be removed from the scope of accreditation.

2.2.4. PT Samples Same as Regular Environmental Samples

EMAX shall analyze and evaluate PT samples in the same manner as regular environmental samples. EMAX shall employ the same quality control, sequence of preparation and analytical steps, and replicates used when analyzing samples.

3.0 DOE REQUIREMENTS FOR PARTICIPATION

3.1. Initial Inclusion

3.1.1. Initial Inclusion to DOECAP Program

EMAX shall successfully complete at least 2 PT samples from MAPEP or an ISO 17043 accredited PT provider. The PT shall, at a minimum, include analyte-method-matrix applicable to reportable data under DOE contracts.

3.1.2. PT Sample for Initial Inclusion

EMAX shall maintain its radiological materials license from the Nuclear Regulatory Commission to be able to participate in analyzing MAPEP PT samples. MAPEP PT samples shall be obtained for all analyte-method-matrix parameters where available. Otherwise, PT samples shall be obtained from ISO 17043 accredited PT providers.

3.1.3. PT samples for non-ISO 17043 Accredited PT Provider

In the absence of ISO 17043 certified PT providers, EMAX shall obtain permission from the Accrediting Body to use a PT from a non-ISO 17043 accredited PT Provider prior to analyzing the PT sample.

3.1.4. PT Samples for Analyte-matrix-method not from a PT Provider

PROFICIENCY TESTING

In the event that a particular analyte in a given method and/or matrix cannot be obtained from any PT provider, EMAX shall inform the Accrediting Body in writing that no PT provider is available for that specific parameter. Other requirements to substantiate the PT requirements shall be duly fulfilled. EMAX shall continue to survey for its availability and shall participate with the PT study as expected.

3.1.5. Analysis Date of PT Samples

EMAX shall provide required PT results that are performed within 12 months, completed at least 7 calendar days apart. In the event that MAPEP PTs are utilized, two consecutive MAPEP PT series shall be provided.

3.1.6. PT Study Determination

For initial accreditation or additional analyte(s) desired to be added to an existing method-matrix accreditation, EMAX shall request from the PT provider to send PT reports directly to the Accrediting Body unless instructed otherwise.

3.1.7. Processing PT Samples

EMAX shall treat PT samples as regular field samples. They shall be received, logged and stored the same way as field samples. PT samples shall be prepared/extracted/digested (whichever is applicable) and analyzed with QC samples, e.g., method blank, lab control sample, etc., per method requirement. PT sample results shall be qualitatively and quantitatively evaluated as prescribed by the method.

3.2. Continued Participation

3.2.1. Maintaining Participation

EMAX shall maintain its participation with MAPEP and other PT programs to meet requirements of DOE contracts. PT results shall be made available to DOE upon request. EMAX shall demonstrate due diligence that at least two out of the most recent three PTs shall successfully pass the PT acceptance criteria.

3.2.2. EMAX PT History

EMAX shall demonstrate due diligence that at least two out of the most recent three PTs shall successfully pass the PT acceptance criteria for each analyte-matrix-method combination. EMAX shall retain PT records for at least five years.

3.2.3. Reporting Requirements to DOE Sites

Where applicable, EMAX shall provide PT results to current DOE projects, within 10 days of receipt of results from the PT provider.

3.2.4. Failure to Meet Criteria

Likewise, EMAX shall implement EMAX-QA08 for non-conforming PT results as described in Section 2.2.2. EMAX shall also perform corrective action for any unacceptable PT results. When two(2) out of three(3) PT samples results are unacceptable, it is understood that DOE contract holder may cease to send samples to EMAX until an acceptable remedial PT is completed.

3.2.5. PT Samples as Regular Environmental Samples

EMAX shall analyze and evaluate PT samples in the same manner as regular environmental samples. EMAX shall employ the same quality control, sequence of preparation and analytical steps, and replicates used when analyzing samples.

EMAX
QUALITY
SYSTEMS
MANUAL
2019
Rev. 5.3

VOLUME 1, MODULE 2, QUALITY SYSTEMS GENERAL REQUIREMENTS

QUALITY SYSTEMS GENERAL REQUIREMENTS

1.0 INTRODUCTION, SCOPE AND APPLICABILITY

EMAX-QS00 is developed to guide EMAX and its constituents to carry out its undertakings fairly, consistently and competently within the boundaries of environmental analytical discipline. It describes EMAX's overall management commitment to Quality Systems, standardization of analytical practices and continuous improvement from acquired knowledge and lessons learned. It is imperative to EMAX to have a clear vision on how requirements are achieved in consideration of the veracity of different specifications and data quality objectives from one project to another. Rest assured that compliance to this document validates competency, impartiality and consistency to any work contracted by EMAX.

This manual is based on TNI Standard, EL-V1- 2011(compliant to version 2009) and DoD/DOE QSM Version 5.3, 2019. It is designed parallel to the sections in the TNI Standard and the DoD/DOE QSM to ensure that all requirements are covered.

This document is applicable to all EMAX projects unless directed otherwise. Federal, State or Local regulations, as well as Project and/or Contract specific requirements shall supersede this document. It shall be available to clients, regulatory authorities and accrediting bodies whenever deemed necessary.

2.0 REFERENCES

TNI Standards, 2011 (compliant to version 2009)
DoD/DOE Quality Systems for Environmental Laboratories, Version 5.3, 2019
ISO/IEC 17025:2017 and ISO/IEC 17025:2005 General Requirements for the Competence of Testing and Calibration Laboratories

3.0 TERMS AND DEFINITIONS

Relative to majority of EMAX's projects and the applicability of terms and definitions within the environmental industry, EMAX has adopted the DoD/DOE QSM terms and definitions as published in its version 5.3, 2019.

4.0 MANAGEMENT REQUIREMENTS

4.1. Organization

4.1.1. EMAX holds all appropriate certifications, validations, approvals and licenses from federal, state and local agencies that mandates provision of legally defensible data for a wide variety of projects, from groundwater monitoring to investigation and remediation on National Priority List sites. These certifications, validations, approvals, and licenses define and document the range of laboratory activities that take place at EMAX while maintaining conformity with this manual, relative to the Standards referenced in Section 2.

The quality systems is designed to naturally practice impartiality on all of its activities and EMAX organization is structured to foster fairness in all of their actions within EMAX and its constituents.

4.1.2. EMAX shall ensure that its staff members, instrumentations, and facilities that carry out its activities adequately satisfy requirements of the standards embodied for this manual. Conformity to project specific requirements (PSR) defines client satisfaction and commitment to impartiality. To secure this responsibility, EMAX quality systems integrates project management and operations in such a way that PSRs are in the hands of staff members performing the analytical process on real-time basis. Refer to EMAX-QA01 for details.

4.1.3. EMAX operates one permanent facility. In the event that an activity necessitates to be conducted outside its permanent facility, EMAX shall ensure that such activity is managed similarly as required by this manual.

This organization also operates to ensure that there are measures employed so that its activities are free from undue pressures, may it be commercial, financial or functions internal and/or external to EMAX that may impair impartiality.

QUALITY SYSTEMS GENERAL REQUIREMENTS

4.1.4. EMAX is not a part of any organization. In the event that an opportunity will arise, EMAX shall ensure that key personnel involved in analytical testing and/or signatories to analytical results shall be clearly identified and probable conflicts of interest does not exist within EMAX and/or between EMAX and the associated organization.

Laboratory activities shall be conducted without bias. Test results shall not be influenced or be construed of being influenced by any of the following:

- business relationships between the laboratory and the client
- family or personal relationships between people in the laboratory who will be involved in laboratory activities and the client
- financial interests in a venture related to the test results.

4.1.5. EMAX shall -

a) empower its key personnel to carry out their responsibilities to ensure that established policies and procedures are consistently put into practice, continuous improvement is exercised, and due diligence in detecting divergence from normative practice as well as implementing preventive measures is second nature.

b) demonstrate that all of its employees are free from any undue internal or external pressures and/or influences that may adversely affect the quality of their work. This process is detailed in EMAX's Ethics Program (EMAX-QA10).

c) ensure that its customers' confidential information and propriety rights are protected. Electronic data shall be properly stored and transmissions shall be carried out as contractually prescribed. This is detailed in EMAX Code of Ethics, Appendix 1 of EMAX-QA10.

d) ensure that activities undertaken by EMAX or its representative outside its laboratory operations shall be conducted in a professional manner, where promotions of its capabilities are factual, impartial and unprejudiced, and shall exercise ethical values set forth in EMAX-QA10.

e) maintain an organizational chart to exhibit its organization and management structure. Refer to APPENDIX B-1.3, Figure 1. Relationships between quality management, technical operations and support services are described in APPENDIX B-1.1.

f) have defined responsibilities, authority and interrelationships of all personnel within the organization affecting quality of data produced. Refer to APPENDIX B-1.2.

g) have adequate supervisors to oversee its processes, to include training, instrument calibrations, data production and review. Refer to APPENDIX B-1.3, Figure 2.

h) have a Technical Operations Manager to oversee laboratory technical operations to make sure that necessities are sufficiently maintained, may it be personnel, services and/or supplies as well as technical knowhow in producing quality data.

i) have a Quality Manager who reports directly to EMAX's President and is responsible to ensure that quality systems is implemented and observed continuously. In the absence of the QA Manager, the QA/QC Coordinator shall be acting on behalf of the QA Manager. Refer to APPENDIX B-1.3, Figure 1.

j) ensure that the following key personnel are employed:
   i. President & Laboratory Director
   ii. Operations Manager
   iii. Laboratory Supervisors
   iv. Quality Assurance and Data Validation Manager
   v. Project Managers

vi. Information Systems Manager
vii. Business Manager

4.1.6. EMAX shall mandate its technical staff to review this manual to ensure awareness, understanding and implementation of the objectives of the quality systems; their responsibilities and consequence of their actions in executing their activities.

4.1.7. Appropriate communication processes are imbedded in EMAX's administrative SOPs (Quality Assurance, Quality Control, Data Management, and Information Systems). In addition, top management shall practice open-door policy to encourage honesty and transparency. Additional Requirements for QA and Operations Managers:

4.1.7.1. The QA manager or his/her designee shall -

a) serve as the focal point of QA/QC and be responsible for the oversight and/or review of quality control data

b) have functions independent from laboratory operations QA/QC functions

c) be able to evaluate data objectively and perform assessments without outside influence

d) have documented training and/or experience in QA/QC procedures and laboratory quality systems

e) have general knowledge of analytical methods for which data review is performed

f) arrange for or conduct internal audits annually

g) notify laboratory management of deficiencies and the quality system

h) monitor corrective actions

i) implement, maintain, and improve the management system by using available tools such as audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews in efforts to monitor trends.

4.1.7.2. The Operations Manager or his/her designee shall -

a) oversee day-to-day operations to include data generation, reporting and review;

b) have overall experience in the fields of accreditation for which EMAX has been accredited or where accreditation is being acquired;

c) have duties that shall include monitoring of quality control standards and validity of data produced;

d) be exclusive to EMAX and shall not serve any other laboratory bearing same function unless otherwise approved by the primary accrediting body.

e) assign a designee when absent for more than 15 consecutive calendar days and shall inform the primary accrediting body in writing when absent for more than 35 consecutive calendar days.

f) meet the qualifications described in Section 5.2.6.

4.2. Management Systems

4.2.1. EMAX shall function and maintain its operations through established standard operating procedures (SOP), systems in place and as guided by this manual. All personnel are trained, as appropriate, utilizing these documents. Such trainings are culminated by an acknowledgment that they have fully understood and attest to implement such. EMAX Browser was established to provide an easy access to these documents for laboratory personnel. All documents generated at EMAX shall be written in English.

All information pertaining data gathered and client information shall remain confidential unless otherwise instructed by the client.

4.2.2. The management team of EMAX is committed to assure that its quality system policies and objectives are habitually practiced and imbedded in its operations. Primarily, EMAX quality systems principles are based on:

a) Commitment to serve its clients such that contract agreement and PSR are attentively employed.

b) Assurance to maintain its standard practice of analytical testing where data collection and processing shall be precisely performed, accurately presented and scientifically valid.

c) Quality Systems Objectives

- To promote quality culture through leadership, quality management, and organizational learning;
- To uphold data integrity by addressing ethical practices through employee training, and practicing impartiality among employees;
- To provide a healthy work environment by maintaining a clean, well ventilated and lighted facility
- To afford adequate equipment to produce reliable data.
- To deliver client satisfaction by producing data of known quality on time.
- To establish a continuous process of improvement through lessons learned, preventive measures, and quality control.

d) Conformance to demonstration of personnel capability to perform environmental testing activities and adherence to established the policies and procedures affecting their work including proper documentation; and

e) Commitment to comply with the adopted Standards. It is of EMAX best interest to practice continuous quality systems improvement as part of its commitment to service. This can be sustained by:
- Meeting customer requirements;
- Operating in accordance with statutory and regulatory requirements; and
- Operating in accordance with the laboratory's documented ethics policy.
-

f) Commitment to confidentiality. In the event that EMAX is required by law or contractually obligated to release confidential information, the client shall be notified about the information to be released.

4.2.3. As evidence of its commitment to sustain the objectives of the quality systems, EMAX management team shall

a) Ensure that processes needed for managing quality systems are established, maintained and implemented

b) Conduct management review at least once a year. This process shall use operational metrics to gauge effectiveness of its systems, client satisfaction and fulfillment of contract obligations. This effort will also allow the management to discover and eliminate probable factors to cause quality issues, slow down deliveries and/or any existing concern affecting productivity as well as processes that do not have added values.

c) Ensure availability of resources, may it be personnel, equipment, facility or supply needs.

d) Safeguard information about the client obtained from other sources other than the client by keeping it confidential to the client unless otherwise agreed by the source.

4.2.3.1. In addition EMAX management shall

a) Define minimum qualifications, experience and skills necessary for all positions in the laboratory. Refer to APPENDIX B-1.2, for details.

b) Train all laboratory personnel to function as positioned. Refer to Training SOP (EMAX-QA05) for details Demonstration of Capability requirements.

c) Keep laboratory personnel training records up-to-date as described in EMAX-QA05.

d) Record laboratory and operational activities, as appropriate. Refer to laboratory logbooks for details.

e) Adequately supervise all laboratory personnel. Refer to APPENDIX B-1.3, Figure 2 for details.

f) Ensure that samples received are accepted, verified and logged in accordance to sample receiving SOP (EMAX-SM02).

g) Record quality of all data reported. Refer to analytical logs for details.

4.2.4. Project management system shall be responsible to disseminate project and contractual requirements to respective personnel. To augment this function, operations and quality assurance meetings shall from time to time reiterate the importance of fulfilling PSRs and contractual obligations.

Business Managers, Project Managers, Project Coordinators or any other personnel acting on behalf of EMAX, shall keep all information gathered or created by EMAX's activities, confidential, unless as required by law.

4.2.5. EMAX Standard Operating Procedures (SOP) organized as follows :

a) Analytical

b) Sample Management

c) Quality Assurance

d) Data Management

e) Information Systems

Approved versions of these SOPs are accessible by all laboratory personnel through EMAX Browser.

4.2.6. Roles and responsibilities of EMAX's technical management and QA manager are detailed in APPENDIX B-1.2. Additional responsibilities for QA and Operations managers are described in Section 4.1.7.1 and Section 4.1.7.2, respectively.

4.2.7. When changes are planned, EMAX management shall ensure that integrity of quality systems is not jeopardized. Such plans shall be carefully reviewed to make certain that contradictions and/or conflicts to other facets of quality systems do not exist. This process shall require review and approval from the QA Manager and Laboratory Director prior to implementation.

4.2.8. Data Integrity

4.2.8.1. EMAX ethics program details process of data integrity training, in-depth monitoring and documentation. Refer to EMAX-QA10 for details. This program shall include:

a) Steps for confidential reporting of data integrity concerns

b) Process of informing EMAX management in the event that there is an ethical concern and the need for further investigation is deemed necessary.

c) Detection and deterrence of improper and unethical actions. This process shall include the following aspects:
   i. Records that the ethics policy was read and signed by all employees
   ii. Objective evidence that annual ethics training is observed

iii. Analyst shall explain, sign and date when data was manually changed.
iv. Where available, data acquisition software's electronic tracking or audit trail functions shall be enabled

4.2.8.2. The quality assurance manager shall keep EMAX-QS00 current; shall review it annually and update it as necessary.

4.2.8.3. The quality manual structure shall be compliant to the standards referenced in this manual.

a) The title manual is titled as "EMAX Quality Systems Manual" and its document ID shall be EMAX-QS00.

b) The laboratory name is EMAX Laboratories, Inc.(EMAX), located at 1835 W. 205th St., Torrance, CA 90501.

c) The primary person responsible for EMAX is Mr. Caspar Pang. He can be reached at 310-618-8889 or CPang@emaxlabs.com

d) EMAX laboratory organizational structure is illustrated at APPENDIX B-1.3, Figure 2.

e) EMAX approved signatories

1) The President & Laboratory Director is the primary signatory for EMAX.

2) In the absence of the Laboratory Director, the QA Manager and the Operations Manager assume the technical functions of the Laboratory Director. The assumption of this duty carries with it the responsibility to assure that all of their actions shall be in accordance, but not limited to EMAX approved policies and procedures, EMAX Quality Systems, EMAX Standard Operating Procedures, Project Specific Requirements, Project Contract Specifications, and other directives related to EMAX laboratory activities.

3) Alternatively, the Project Managers, limited to the specific project(s) they handle, may also assume the technical functions of the Laboratory Director. Likewise, this duty carries with it the responsibility to assure that all of their actions shall be in accordance, but not limited to EMAX approved policies and procedures, EMAX Quality Systems, EMAX Standard Operating Procedures, Project Specific Requirements, Project Contract Specifications, and other directives related to EMAX laboratory activities.

4) List of approved signatories is included in EMAX E-1.3, Figure 1.

f) Concurrence and signature page is in the first page of this document

g) Objectives of quality systems are in Section 4.2.2.c.

h) EMAX quality policy statement is in Section 4.2.2.

i) Table of contents, references and appendices is in page 5.

4.2.8.4. EMAX Quality Systems shall include the following:

a) Maintenance, calibration and verification procedures for conducting tests, refer to EMAX SOPs.

b) Major equipment and reference measurement standards, facilities and services, refer to APPENDIX B-3, Equipment, Facilities and Services.

c) Verification practices, refer to Proficiency Tests.

d) Procedures for reporting analytical results, refer to Analytical SOPs.

e) Organization and management structure, refer to APPENDIX B-1.3, Figures 1 and 2.

f) Document control procedure, refer to EMAX-DM02.

g) Job description of key staff, refer to APPENDIX B-1.2.

h) Procedures for achieving traceability of measurements are embedded in SOPs as a function of documentation of data acquired, may it be through instrument electronic data output or manually transcribed as observed.

i) List of analytical SOPs, refer to APPENDIX B-2, List of EMAX SOPs.

j) Procedure for reviewing new work, refer to EMAX-QA01, Project Management.

k) Procedures for handling samples, refer to EMAX SOPs for Sample Management.

l) Procedure for non-conforming events, refer to EMAX-QA08, Corrective Action.

m) Procedures for departures from documented policies and procedures.

Departures from documented policies and procedures or from standard specifications should be saved, and if it is unavailable, the laboratory management will treat it on a case by case basis taking into consideration the impact of the quality of data.

Departures and deviations from SOP that are due to project DOQ/PRS are handled by the project management system. Other departures and deviations are classified as minor or major changes. When a departure and/or deviation from SOP will have no impact on data quality, the change is deemed to be a minor change requiring approval of either the immediate supervisor or the Technical Operations Manager.

Any departure and/or deviation from SOP that may affect the quality of data is deemed a major change and requires the approval of both the Technical Director and the QA Manager. When granting such approval, the Laboratory Director and the QA Manager shall consider whether the justifying circumstances are of such significance that an addendum to the SOP, a revision of the SOP or a new SOP is needed.

n) Procedures for dealing with complaints

EMAX shall treat customer satisfaction with high priority. All customer complaints shall be considered and assessed appropriately. A Project Manager (PM) shall be designated as primary point of contact at the inception of the project and shall serve as the conduit between the customer and EMAX. The PM shall be responsible in resolving issues from the time sampling supplies are ordered to the time the data deliverables are delivered. The PM is also responsible for responding to future questions that may arise from submitted data, hard copy and/or electronic. EMAX-QA08 details the guidelines for taking care of customer complains.

o) Procedures for protecting confidentiality and propriety rights

EMAX will not intentionally disclose to any person (other than the representative(s) designated by the client) services rendered or information received and/or generated by EMAX.

All employees, upon joining EMAX are informed of the importance of the policy on protecting confidentiality and EMAX'S propriety rights. All data received and generated at EMAX shall remain confidential within EMAX Laboratories, Inc. Reports and other pertinent information is only disclosed by authorized personnel to the clients concerned. No one is permitted to remove or make copies of any EMAX records, reports or documents without prior management approval. Disclosure of confidential information could lead to dismissal.

p) Procedures for audits and data review, refer to EMAX-QA07 and EMAX-DM01.

q) Procedures for personnel training and competency, refer to EMAX-QA05.

r) Policy for the use of unique electronic signatures, where applicable.

Electronic data and/or electronic logbooks shall bear the identification of the analyst that performed the process. They shall be identified with first initial and the first 5 characters of the analyst's last name (e.g.

KPimen for Kenette Pimentel) or their last name should their last name be <6 characters (e.g. JDoe for John Doe). This identification is equivalent to electronic signature. The electronic signature shall be authenticated by the initials of the secondary data reviewer during data review.

s) Procedure for procurement of standards, refer to EMAX-QC02 and EMAX-QA09.

t) Data management procedures including validation, verification and purging of electronic data and data systems, refer to EMAX SOPs for Data Management.

u) Procedures for manual entry of raw data from analytical measurements that are not interfaced to LIMS and the verification of records for accuracy of manually entered data, refer to EMAX-DM01 – Data Flow and Review.

v) Procedures for making changes to electronic data, refer to EMAX-DM01 – Data Flow and Review.

w) Procedures for electronic data processing, reporting and storage, refer to Analytical SOPs, section on Report Generation.

x) Procedures for data review, for general data review procedures, refer to EMAX-DM01 – Data Flow and Review and for method specific data review procedures, refer to analytical SOPs.

y) List of current certifications or accreditations, refer to APPENDIX B-4, List of Accreditation.

z) Health and Safety, refer to APPENDIX B-5.

aa) Material (Waste) Management, refer to APPENDIX B-5.

4.2.8.5. EMAX SOPs are categorized as follows:

1) Quality Assurances SOPs are established to manage and detail the execution to the overall quality assurance objectives and policies.

2) Supplemental Quality Control SOPs are established controls auxiliary to Analytical and Quality Assurance SOPs.

3) Sample Management SOPs are established policies and procedures for sample custody from cradle to grave.

4) Analytical SOPs are established tests manuals adopted from approved reference methods e.g., EPA Methods, Standard Methods, or other industry approved methods.

5) Data Management Procedures are established guidelines for data control from generation to archival.

6) Information Systems Procedures are guidelines to manage electronic media from software development to electronic data archival.

7) Radiation Safety SOPs are established policies and procedures for handling samples from DOE-sponsored projects or samples suspected to have limited quantity of radioactive material.

EMAX SOPs listed above are developed and maintained to reflect its current practice. These documents are:

a) detailed to depict procedures that can be understood by a qualified personnel to execute the job;

b) downloaded to the network (EMAX Browser) for employees' easy access;

c) published bearing effective date and revision number and the signatures of the approving authorities;

d) established as standalone procedures so they can be performed as written

e) developed and maintained for all accredited analytes or method;

f) internally developed detailing the steps to execute the requirements of the referenced method including any deviation from the published method, refer to APPENDIX B-2. Deviations from the referenced

method are detailed in the SOPs' supplementary notes section. EMAX SOP for Writing SOP (EMAX-QA00) for details of SOP structure;

g) reviewed for accuracy and adequacy and updated as necessary. Technical SOPs are reviewed by competent personnel at least once a year to include Chemical Hygiene, Waste Management and Radiation Protection applicable to DOE sponsored projects.

4.3. Document Control

4.3.1. General

EMAX established a standard operating procedure for writing SOP (EMAX-QA00) to standardize the process on generating, maintaining and controlling standard operating procedures. Document Control SOP (EMAX-DM02) is established to cover document identification, filing, distribution, control, changes, retention, protection, preservation, archival, retrieval, and disposal of controlled documents.

Document control policies and procedures related to or supplementary to analytical processes (e.g. calibration of support instruments) are imbedded in the analytical SOPs or in SOPs specific to the activity being performed.

4.3.2. Document Approval and Issue

4.3.2.1. All documents in the laboratory as part of the quality system are reviewed and approved by the Laboratory Director and the QA & DV Manager prior to issue. EMAX Browser was established to provide an easy access to these documents for laboratory personnel. The browser table of contents is maintained to list the SOPs, its current version and the latest document review date. The QA & Data Validation Department controls the documents loaded in the browser so that only the most recent edition is available for use to preclude the use of invalid and/or obsolete documents. Other distributions are recorded on the document control ledger kept with the original SOP.

4.3.2.2. The controls for document approval and issue ensure that:

a) only authorized editions are available for use and are accessible throughout the lab;

b) SOPs are reviewed by both the user and the QA & DV Department to ensure that current practice is consistent with the approved procedures as well as the referenced methods;

c) obsolete documents are removed and replaced upon approval and replacements are made available for access;

d) obsolete documents are voided and archived for at least 5 years from the date it was voided;

e) new editions, revisions and/or changes to SOPs are emailed to relevant supervisors for them to disseminate and mandate document review to significant staff members;

f) document reviews shall be completed by accomplishing document review form (DRF);

g) in the event that additional or special instruction is required to conform to PSR, such instructions are controlled by the project code.

4.4. Review of Requests, Tenders and Contracts

4.4.1. EMAX Project Management SOP (EMAX-QA01) describes the procedures for the review of requests, tenders and contracts. The policies and procedures of this SOP include:

a) PSRs, including the methods to be used, quality control procedures, reporting formats, deliverables and other requirements to fulfill contract agreement.

b) A process to ensure that the laboratory has the capability and resources to meet the PSR;

c) Appropriate selection of environmental test method to meet the PSR.

4.4.1.1. Where new methods are to be developed or additional analytes are required by a project, the Operations Manager is consulted purposely to review capability and readiness to deliver the environmental tests in query. The review process includes demonstration of capability, successful participation of proficiency testing (PT), detection limits, or other essential quality control requirements. The Project Manager (PM), where applicable, reviews current accreditation status of the laboratory and informs the client of the results of this review if it indicates any potential conflict, deficiency, lack of appropriate accreditation status, or inability on the laboratory's part to complete the client's work.

4.4.1.2. Conformity to a project specification shall be communicated to and agreed with the customer, unless inherently defined. EMAX shall also advise the client when the required test method is considered to be inappropriate or obsolete..

4.4.1.3. Any differences between the request or tender and the contract are resolved before any work commences. However, in instances where disparity, ambiguity or any other related circumstance arise thereafter, it shall be properly communicated and resolved. Contracts are generally drawn as written agreements to attest that the contract is acceptable to both parties. Where contract was agreed upon orally, they are followed by written statement (i.e., email or letter) confirming the agreement. Any deviations requested by the client shall only be considered if it does not affect the integrity of the EMAX or the validity of the results

4.4.2. In summary, the PSR documents the PMs' review of the project requirements. These records shall be maintained by the PMs including agreed variances, pertinent discussions relating to client and/or project requirements. Records of PMs' completeness review of data produced during the execution of the contract shall be documented in the analytical record of each data package. Records of data package technical review shall be maintained with the data package retained at EMAX.

4.4.3. Where part of the project is subcontracted, the PM is responsible for the review of contract requirement and make sure that the sub-tier lab is capable of providing the requirements of the project. Contingent upon the absence of sub-tier lab that is not qualified (e.g., no accreditation) to do the work but has the capacity to perform the work, the PM shall obtain consent from the client prior to using the sub-tier lab. Likewise, records of PMs' completeness review of shall include subcontracted work.

4.4.4. In instances where deviation from the contract is dictated by a circumstance, the PM shall inform the client on a timely manner. Changes initiated by the PM are acknowledged by their dated initials. Where changes impact generation of deliverables (e.g., analyses, report forms, EDD formats, etc.), the PM is required to discuss the matter with the Laboratory Director and the Operations Manger.

Waivers from QSM requirements are mostly executed during project proposal stage. Hence, such are already approved when contract is awarded. In the event that adherence to approved requirements become a challenge and/or not feasibly achievable during the project implementation stage, a waiver shall be requested in writing to the appropriate project contact person on a project-specific basis. It shall include technical justification relating to the specific project for the waiver. Documentation of approval for the waiver shall be maintained by EMAX Project Management and readily available for review.

4.4.5. When change orders are initiated or if a contract needs to be amended after work has commenced, the PM shall go through the same review process and disseminate the amendments to all pertinent personnel. Likewise, when there are major changes at EMAX (e.g., suspension, revocation or withdrawal of accreditation, change of ownership, etc.), the PM is also responsible to inform the client.

4.5. Subcontracting Environmental Tests

4.5.1. Generally, subcontracted work decision to where the work is going is made by the client prior to contract award. When EMAX is tasked to select the subcontract lab, EMAX shall ensure that subcontracted laboratories meet the requirements of the project. This process is detailed in EMAX SOP for Subcontracting (EMAX-QA01).

4.5.2. EMAX shall obtain client consent upon qualifying a sub-tier lab before work is subcontracted, preferably in writing. In the event that oral approval was provided, the PM shall send a written statement (i.e., email, fax or letter) confirming the verbal approval.

4.5.3. EMAX is responsible to the client for the subcontractor's work, except in the case where the client or a regulatory authority specifies which subcontractor is to be used or when client consent was not obtained or granted.

4.5.4. A register of all subcontractors that EMAX uses for environmental tests and a record of the evidence of compliance are maintained by the designated PM.

4.5.5. In the event that EMAX is obligated to choose a sub tier lab, subcontracted labs shall at a minimum possess and maintain a NELAP accreditation in compliance to NELAP standards and meets all applicable requirements in performing the tests and capable of submitting results at the required format.

4.5.6. For DoD and/or DOE sponsored projects, the PM shall ensure that the subcontract lab meets the requirement of the latest version of DoD/DOE QSM prior awarding the subcontract.

4.5.7. Subcontracted labs shall possess and maintain DoD ELAP accreditation in compliance to DoD and/or DOE-sponsored projects.

4.5.8. For DoD and/or DOE-sponsored projects, unless otherwise subcontractor was selected by the client, the PM shall obtain project-specific approval from the DoD or DOE client before samples are analyzed.

4.5.9. In the event that the subcontracting lab has more than one facility, each participating facility shall go through the same qualification process as described in this manual.

4.5.10. Currently, all analyses performed at EMAX are completed from sample receiving to data package delivery (hardcopy and EDD). In the event that EMAX will have a need to outsource a segment of its management systems or outsource personnel, these services shall be subjected to abide by EMAX's management system and comply with the requirements of this manual. For DoD or DOE-sponsored projects, such services shall be subject for review and approval of the client.

4.6. Purchasing Services and Supplies

4.6.1. Procurement in general is under the supervision of the Operations Manager. Qualifying vendors and suppliers as well as purchasing are subject to the approval of the Operations Manager. Purchasing SOP (EMAX-QA09) describes EMAX's purchasing system, its policies, procedures and links to quality control of purchased materials, where applicable. This SOP is applicable for the procurement of laboratory instruments, analytical standards, reagents, chemicals, supplies, services and other related materials that the lab may need.

Calibration services by third party shall be outsourced to an ISO/IEC 17025 accredited calibration laboratory.

4.6.2. Analytical Standards, solvents, reagents, chemicals or any other item that may affect the quality of environmental tests are not used until they have been verified to conform to requirements defined in the methods for the environmental tests concerned. Chemical QC SOP (EMAX-QC01) details the policies and procedures for acceptance/rejection of chemicals prior to its use. Records of actions taken to check compliance are endorsed, reviewed and maintained by the QA & Data Validation Department.

4.6.3. Approved purchased orders are utilized to place and receive orders. Materials are inspected upon receipt for accuracy. If received items concur with the packing slip and the purchase order, these documents are forwarded for payment approval. Otherwise, the Operations Manager is informed about any discrepancy.

4.6.4. The process of qualifying vendors and suppliers is detailed in EMAX-QA09. Documents generated during the process are maintained by the Operations Manager.

4.7. Service to the Customer

4.7.1. The primary function of a PM is to serve as an interface between EMAX and the customer. A PM is designated for each project to review and clarify project requirements and monitor the laboratory's performance in relation to the work performed. While each PM may be serving more than one client, confidentiality to other clients is mandatory. This policy is also true and apparent to any constituent of EMAX anywhere they may be at any given time.
Each PM shall maintain and document communications with the client. The PM shall notify clients when the following are encountered:

a) the client has specified incorrect, obsolete, or improper methods;

b) methods require modification to obtain project data quality objectives (e.g., difficult matrix, poor-performing analyte);

c) absence of PSRs (e.g., Quality Assurance Project Plan (QAPP), Statement of Work (SOW), Sampling and Analysis Plan (SAP), detection levels); or

d) the laboratory has encountered problems with sampling or analysis that may impact results (e.g., improper preservation of sample holding time, turn-around-time).

4.7.2. Good project management shall foster and build strong working relationships with the clients. Hence, it is incumbent for PMs to make an effort to seek feedback from their clients. Both positive and negative feedback would help direct focus on management systems review and improve customer relations. This improvement process will not only sustain business health but will also show that EMAX genuinely cares about its customers' needs and opinion.

4.8. Complaints

EMAX treats customer satisfaction with high priority. All customer complaints are entertained and assessed appropriately. The PM is responsible in making sure that issues are resolved from the time sampling supplies are ordered to the time the data deliverables are completed. The PM is also responsible for responding to future questions that may arise from submitted data, hard copy and/or electronic.

Corrective Action SOP (EMAX-QA08) includes policy and procedure for the resolution of complaints received from clients or other parties. Complaints are treated on a case-by-case basis. Lessons learned from these incidents are incorporated in the improvement process, where new policies may be implemented to prevent recurrence. Records of all complaints, investigations and corrective actions taken by the laboratory are maintained.

4.9. Control of Non-conforming Environmental Testing Work

4.9.1. Quality control procedures are imbedded in every analytical SOP. These procedures also contain corrective actions for parameters that are nonconforming. This process shall ensure that:

a) The Project Manager is informed by the Operations Manager of the nonconforming event with recommendation(s) on how to resolve the issue. Where data quality is affected, direction on how to

proceed is provided by the QA Manager. Where necessary (e.g., data quality is jeopardized), work shall be temporarily halted by the QA Manager until issue of concern is resolved.

b) An evaluation of the significance of the nonconforming work is assessed.

c) Corrective action is taken in a timely manner.

d) Where data quality is or may be impacted, the PM notifies the client.

e) When work is halted due to a non-conforming event, resumption of work is authorized by the QA Manager. In the absence of the QA Manager, the Laboratory Director may authorize resumption upon verifying that the non-conforming event is back to control.

4.9.2. Non-conforming events indicative of a need for further evaluation and/or measures to prevent recurrence is necessary, hence corrective action is initiated. This process is described in Section 4.11.

4.9.3. For DoD/DOE-sponsored projects, when corrective action is initiated due to potential data quality issue, the PM shall duly inform the client representative in accordance to the PSR. Records of corrective actions or proposed corrective actions to resolve the nonconformance shall be submitted to the client within 30 business days of discovery.

4.10. Improvement

4.10.1. EMAX Quality systems management propels a continuous improvement cycle. It has to be effective, efficient, and equitable.

a) Effectiveness is measured by how well its quality policy and objectives are portrayed by the data quality audit results and corrective and preventive measures.

b) Efficiency is reflected by the receptiveness and ease in implementing established quality measures.

c) Equitability is significant to maintain business existence. Identifying and discarding a non-value added activity and focusing on customer satisfaction is a key factor to equitable operation.

4.11. Corrective Action

4.11.1. General

Policies and procedures established to resolve problems and restore proper functioning of laboratory operations when error, deficiencies or out-of-control situations arise are detailed in SOP for Corrective Action (EMAX-QA08).

4.11.2. Cause Analysis

Cause analysis is a fact-finding process purposely to identify the contributory factors that led to the non-conforming event. It shall include the timing, location and degree of the consequences caused by the nature of the incident. This should be carried out systematically and is best accomplished by team effort, openness and good documentation. Essential parts of cause analysis are:

a) Defining the non-conforming event – what happened
b) Collection of data – objective evidence, interviews, when did it happen
c) Identifying possible causes – sequence of events leading to the problem, conditions allowing the problem to occur, other factors contributory to the problem. Was it systems, human or organization failure?
d) Identify impact on data quality – identify associated data and determine if data quality is jeopardized.
e) Identify lessons learned

4.11.3. Selection and Implementation of Corrective Actions

Meaningful cause analysis will draw significant considerations in configuring essential corrective action. Such consideration shall not only correct the problem but also prevent recurrence and occurrence of foreseen circumstances due to lessons learned from the nonconforming event.

Corrective action implementation shall be applied relative to all activities affected by the problem as well as possible sections where it could happen.

Documentation of non-conforming events is logged in the Non-Conformance Report (NCR) database.

4.11.4. Monitoring of Corrective Actions

The Operations Manager is responsible for monitoring the implementation and effectiveness of the corrective action. The QA Manager shall review the NCR database quarterly to determine appropriateness of corrective action taken and/or probable trends or recurrence. In the event that the corrective action implemented is unsuccessful, the Operations Manager and the QA Manager shall revisit the issues of concern and find measures to resolve the issue.

4.11.5. Additional Audits

When alleged breach of established policies and procedures or non-compliance with this manual is observed, the QA Manager or his/her designee, shall conduct an audit on appropriate areas of activity in accordance with Section 4.14 of this manual as soon as possible.

4.11.6. Documenting Corrective Action

Analytical Quality Control Procedure (QCP) is incorporated in every work order. The QCP includes corrective action for quality control parameters. In the event that other anomalies occur or corrective action in the QCP does not resolve the problem, the Supervisor is informed and a non-conformance report is initiated. Refer to Corrective Action SOP (EMAX-QA08).

4.11.7. Systematic Errors

Systematic error is bound to happen because it is inherent to the system. Therefore this type of error must be identified during cause analysis and corrective action shall necessitate change in the associated system to correct the problem.

4.11.8. Nonconformance Record Tracking System

EMAX has developed a NCR database where nonconformance events and corrective actions are logged. This process is an integral part of EMAX-QA08 and it includes:

1) Event description; corrective action recommendation (CAR) corrective action taken (CAT). The event description is initiated by the person that discovered the non-conforming event and informs the Supervisor or the responsible person for initiating CAR (e.g., the PM, if it requires client notification, the QA if it concerns data quality, PT). The CAR is initiated by the Supervisor or the responsible party. The CAT is initiated by the person responsible for implementing the CAR.
2) The Supervisor is responsible in making sure that actions are taken in a timely manner as well as the acceptability of course of action. Where data quality is or may be jeopardized, the PM is informed so that the client is notified.
3) These non-conformances and actions taken are logged in a database maintained by the QA & Data Validation Department.

The QA Manager or his/her designee performs a periodic review to check for appropriateness of the CAT, trending analyses or recurring problems. Where the QA Manager observes unsuitable CAT, trending or recurring events, the issue is brought to the Operations Manager and when necessary, involve the Laboratory Director for verification, evaluation and discussion of probable cure to the existing concern.

EMAX shall obtain approval from DoD ELAP Accreditation Bodies or the DOECAP Operations Team when changes to approved corrective actions implemented to address findings from a DoD or DOECAP assessment, as appropriate. Failure to obtain such approval in a timely manner may result in discontinued work until verification is completed.

4.12. Preventive Action

4.12.1. EMAX has established pro-active processes where identified potential sources of non-conformances or probable cause of problems may occur.

4.12.2. These processes include but not limited to the following:

1) Standard Operating Procedures are established so that proper course of action is followed across the board.
2) Chemicals used are passed through quality control prior to its use to prevent accidental use of underrated or adulterated chemicals.
3) Operations capacity is verified prior to accepting work and turning down work when project specifications are not within EMAX capability.
4) Appropriate personnel training and adequacy of commissioned equipment.
5) Continuous quality systems improvement obtained from technical experience, customer satisfaction, participation of performance testing, trend analysis (LCS, NCR and PT) and assessments (internal and external).

4.13. Control of Records

4.13.1. General

Document Control SOP (EMAX-DM02) describes the policies and procedures of document management. This covers document identification, distribution, control, changes, retention, protection, preservation, archival and disposal of controlled documents.

All records are expected to be legible. They are stored and retained in such a way that they are readily retrievable in a suitable environment to prevent damage or deterioration and to prevent loss. These records are retained for a minimum period of five years.

Only authorized personnel can access the records warehouse.

Access controls to electronic records are established for LIMS and administrative files. All users of Company data must be authorized to access the appropriate systems and their resources. Access is controlled and monitored in accordance with Company policy. Copies of data, regardless of location, have the same data security and access control requirements as operational data. The elements involved in controlling and monitoring this access include identification, authentication, and authorization. Access levels are determined by the user's or user group's data access privileges. Access is granted by means of a computer account, which serves as identification. A computer account is created based on an approved request for network account.

Backing Up Files SOP (EMAX-IS09) describes policies and procedures of backing up files from a server, fixed disk or removable disk to a backup medium. All critical LIMS and administrative data are backed up on a regular basis. Files stored in servers are backed up daily. Workstation data are backed up at a frequency established by the user who generates the data. This frequency is influenced by the rate of generation of new data, the rate with which the data changes and the effort required to recreate information, if it is lost. Backup and archival methods shall be defined for all data files. Backup data shall be used to recover data when files have been destroyed. Archived data shall be kept for future reference. Retention times shall be defined for archived data. Provisions shall be made for off-site storage of daily and archival back ups.

4.13.2. Technical Records

Technical records shall include all data collected to generate sample results, associated QC samples, calibrations and detection limits. These records shall include but not limited to internal chain of custody, raw data, instruments IDs and initials of staff members who participated in the analytical process (sample preparation, analysis and data review).

Data collection shall be traceable to the staff member that performed the task.

Transcription error correction is detailed in EMAX-QA08, Section 4.1, where corrected record should remain readable and the correction entered is initialed and dated. All original electronic records shall be retained. When corrections are due to reasons other than transcription errors (e.g. manual integration), the reasons for the correction are documented.

4.13.3. Additional Requirements

Cradle-to-grave documentation system is applied to every sample received at EMAX. These records shall:

a) Include all information pertinent laboratory activities conceived to allow historical reconstruction of data deliverables. Laboratory logbooks or forms related to the laboratory activities are integrated with the specific SOP. Internal chain of custody logbooks record the history of inter-laboratory transfers of samples and/or extracts.
b) Be retained for at least five years.
c) Be available to the accreditation body.
d) Be retrievable when records are stored only on electronic media.
e) Have access logs, where applicable, to archived records.
f) Have Information necessary for the historical reconstruction of data. These records shall include:
    i. all raw data for samples (lab QC and field), calibrations, logbooks
    ii. related SOPs
    iii. lab sample identification
    iv. relevant dates – collection, lab receipt, preparation, analysis, calibrations, detection limit study/verification
    v. relevant time – collection, lab receipt, preparation, analysis, calibrations, detection limit study/verification
    vi. instrument identifications, maintenance logs, reference to operating conditions
    vii. all manual calculations
    viii. relevant analysts/operators – initials/signatures or electronic identification
    ix. sample preparation information – sample amount, final volume, cleanup, SOPs, date, time, instrument IDs, standards/reagents, notations, etc.
    x. test results
    xi. standards and reagents information – standard/reagents logs, source/vendors, receipt, preparations,
    xii. calibration criteria, frequency and acceptance
    xiii. data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions
    xiv. quality control protocols and assessment
    xv. electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries
    xvi. method performance criteria including expected quality control requirements
    xvii. proficiency test results
    xviii. records of demonstration of capability for each analyst
    xix. records of names, initials and signatures for all individuals who are responsible for signing or initialing laboratory record
g) All manual data entry shall be recorded legibly in permanent ink.
    i. individuals rectifying an incorrect entry previously made shall date and initial the correction made;
    ii. Corrections, other than transcription errors shall include reason for the correction.
h) In the event that EMAX transfers ownership or goes out of business, unless there is pre-existing data transfer instructions, its clients shall be duly informed with a request of records transfer instructions. All records shall be transferred according to the client's instructions. In case of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records shall be followed.

4.14. Internal Audits

4.14.1. Internal audits include both technical and systems audits. Technical audits verify compliance with method-specific requirements as well as operations related to the analytical process, data reduction, and data review. The process includes all actions related to data generation and the assurance of its quality. Systems audits verify compliance with the laboratory's quality systems, based on Standards governing this manual and applicable regulatory requirement to accomplish existing projects. Part of the systems audit would be a review of the policies and procedures for Quality Assurance, Quality Control, Sample Management, Data Management, Project Management, and Information Systems.

4.14.2. Internal Assessment SOP (EMAX-QA07) describes the policies and procedures to carry out internal systems audit (ISA). The QA Manager shall plan and organize audits at a predetermined schedule. Such audits shall be carried out by the QA Manager or qualified personnel who are, wherever resources permit, independent of the activity to be audited.

4.14.3. The audit schedule shall ensure that all areas of the laboratory are reviewed over the course of one year. Audit personnel shall be trained and qualified in the specific quality system element or technical area under review. Updated checklists shall be secured preferably from accrediting bodies (ABs) and shall be utilized to assure completeness.

4.14.4. Immediate corrective action is required when critical findings where the correctness or validity of calibrations or test results is observed. Clients shall be duly informed in writing, as necessary.

4.14.5. ISA report shall include 1) the area of activity audited, 2) the audit findings, and 3) corrective actions that arise from them. Refer to EMAX-QA07 for procedures in documenting internal audit. The Operations Manager and the Laboratory Director receive a copy of the Internal Assessment Report. The Operations Manager is responsible that the corrective actions are discharged within the agreed time frame as indicated in the report. Time frame for corrective actions is based on the magnitude of the finding and its impact on the defensibility and use of data.

4.14.6. It is the responsibility of the QA Manager to follow-up, verify, and record the implementation and effectiveness of the corrective action taken. Where resolution or timeliness of the implementation of corrective actions is in question, the Operations Manager must inform the QA Manager and the Laboratory Director for re-evaluation and/or redirection.

4.14.7. In the event that clients need to be informed about a finding,

- it is of EMAX's best interest to carry it out as soon as the information verification is completed;
- corrective action target completion date shall be concurred with the client;

4.14.8. Internal assessment shall be conducted with full cooperation of laboratory operations. Auditing team shall have full and free access to work areas, documents, instrumentations, laboratory personnel and all activities affecting quality.

4.15. Management Review

4.15.1. Members of the executive management submit their annual report to the QA Manager. The QA Manager consolidates the reports and draws an executive summary of annual management review. The management annual review is submitted to the Laboratory Director of the company for review. The Laboratory Director or a designee arranges an executive meeting to review the laboratory's quality system and environmental testing activities, their continuing suitability and effectiveness, and to introduce necessary changes or improvements. The review shall take account of:

- the suitability of policies and procedures;
- reports from managerial and supervisory personnel;

- the outcome of recent internal audits;
- corrective and preventive actions;
- assessments by external bodies;
- the results of inter-laboratory comparisons or proficiency tests;
- changes in the volume and type of the work;
- client feedback;
- complaints; and
- other relevant factors, such as quality control activities, resources, and staff training.

4.15.2. Findings from management reviews and the actions that arise from them are included in the report. The Laboratory Director presides the executive meeting and directs course of action where changes are necessary.

The Operations Manager is responsible to ensure that identified action items concerning operations (e.g., initiate hiring or reshuffling and/or training of personnel, purchase or repair of equipment, facility improvement or maintenance, and other action items related to operations) are carried out within an appropriate and agreed timescale.

The QA Manager is responsible to ensure that identified action items concerning quality systems (e.g., policies and procedures, quality controls, and other action items related to quality systems improvement) are carried out within an appropriate and agreed timescale.

4.16. Data Integrity Investigations

Ethics Program SOP (EMAX-QA10) details the process of promoting ethical values in the workplace, prevention of potential conflicts of interest, consequences of wrongful use of resources, mismanagement of contract agreements and proper conduct when dealing with ethical dilemma.

Discovery of potential issues shall be handled in a confidential manner and must be kept at a low profile while conducting evaluation, full investigation, or other appropriate actions needed to complete the process and have issues clarified.

All investigations that result in finding of inappropriate activity related to data integrity shall be documented and shall include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients. All documentation of these investigation and actions taken shall be maintained for at least five years.

Any occurrence of inappropriate and/or prohibited laboratory practices shall be reported to the DoD ELAP Accreditation Body (AB) within 15 business days of discovery. These include findings of such inappropriate practices by laboratory staff and/or customer stakeholders. EMAX shall fully execute its zero tolerance policy regarding breach of ethical conduct. Records of associated corrections taken and proposed corrective actions to prevent recurrence shall be submitted to the AB within 30 business days of discovery.

EMAX shall also fully cooperate with its AB and the Environmental Data Quality Workgroup (EDQW) accordingly and shall treat these infractions seriously.

5.0 TECHNICAL REQUIREMENT

QUALITY SYSTEMS GENERAL REQUIREMENTS

5.1. General

5.1.1. Factors affecting correctness and reliability of environmental test results:

- human factors (5.2);
- accommodation and environmental conditions (5.3);
- environmental test methods and method validation (5.4);
- equipment (5.5);
- measurement traceability (5.6);
- sampling (5.7); and
- handling of samples (5.8).

5.1.2. All measurements are subject to uncertainty. Total uncertainty of measurements is relative to the consistency or variability of how measurements are conducted. EMAX established environmental test methods and procedures, standardized training, and qualification of personnel, in conjunction to daily equipment check and procurement of service contracts for major equipment. EMAX designed its facility considering factors that may effect stability of equipment, minimize probable sources of contamination, health and safety of its workers, and ease for maintaining tidiness and cleanliness.

5.2. Personnel

5.2.1. EMAX technical staff undergoes training regardless of educational attainment and/or experience. Training is initiated with SOP reading, applicable quality controls and discussion of primary duties and responsibilities. With adequate supervision, the on-the-job training follows. The trainee will then have a chance to practice and build experience in the real work scenario applying all the necessary steps and quality controls that is required to complete the job. Training is culminated by the trainee's demonstration of capability.

5.2.2. Training SOP (EMAX-QA05) details the general process of conducting training. The Supervisor is tasked to conduct the training or assigns a mentor. The mentor identifies the SOPs needed for the training as well as all the necessary training needs to successfully execute and complete the training including a schedule/list of activities that needs to be completed. The progress of training is dependent on the background and skills of the trainee. The mentor shall document the training activities and shall determine when the trainee is ready for the job.

5.2.3. Currently, all laboratory personnel are employed by EMAX. In the event that there will be a need for additional technical and/or key support personnel, they shall undergo the same training, supervision and qualification process as the regular EMAX employees and must work in accordance with the EMAX's quality systems as well as health and safety practices.

5.2.4. Laboratory personnel training files are maintained and updated periodically by the QA & Data Validation Department. These records include:

- EMAX Resume to include their educational background, experience and current job description
- In-house training records
- Demonstration of Capabilities
- External Training (if any)

Minimum job descriptions are detailed in APPENDIX B-1.

5.2.5. Training records are forwarded to the QA & Data Validation Department for evaluation and certification to authorize specific personnel to perform particular types of environmental testing. The certification qualifies them to

conduct the first level of review on test results, give opinions and interpretations to test results, and to operate equipment specific to the test performed. The certification is approved by the Operations Manager and the QA & DV Manager.

5.2.6. Additional Personnel Requirements

Technical Directors employed shall possess the qualifications prescribed at TNI Section 5.2.6, as applicable.

5.2.7. Data Integrity Training

Every new employee goes through data integrity training and is renewed on an annual basis thereafter together with the current employees. Ethics Program SOP (EMAX-QA10) details EMAX business ethics, its policies and procedures, code of ethics, prohibited practices and how and when to report data integrity including its documentation process. Employees are required to have full understanding of their obligations and commitments related to data integrity. They, too, are made to be aware of that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to disciplinary action including immediate termination or be brought in the court of law. Data integrity trainings are authenticated by the participant's dated signature.

5.3. Accommodation and Environmental Conditions

5.3.1. Facilities

Significance of every facet of the laboratory was taken into consideration when EMAX designed its facilities. Its energy sources, lighting, and environmental conditions are planned and constructed to facilitate expected output of environmental tests.

To ensure that environmental conditions do not jeopardize the required quality of any measurement, controls are established and monitored. Fume hoods are adequately provided so that particular care when sub-sampling and environmental testing are undertaken. Technical requirements for accommodation and environmental conditions that can affect the results of environmental tests are monitored and documented.

5.3.2. Monitoring

All environmental conditions that require control are monitored and documented in accordance with the relevant specifications, methods and procedures or where they influence the quality of the results. Due attention are paid as appropriate to the technical activities concerned (e.g. Leaching requires controlled temperature, DOE lab requires frisking, etc.). Environmental tests shall be stopped when the environmental conditions jeopardize the results of the environmental tests.

In instances where monitoring or controls of any of the above-mentioned items are specified in a test method or by regulation, a course of action is included in the SOP relevant to the activity being performed to meet and document adherence to the laboratory facility requirements.

5.3.3. Cross-contamination Prevention

The laboratories were constructed to have effective separation between neighboring areas in which there are incompatible activities. Volatiles Laboratory is located farthest from the extraction lab and has independent mechanical controls; laboratories for DOE samples are isolated with independent mechanical controls and restricted entries.

All foreseen measures are taken to prevent cross-contamination. Sample storage for Volatiles is located in the Volatiles Laboratory. Samples from DOE sights that are identified as restricted samples are stored in the DOE Laboratory.

EMAX-QC08 SOP provides procedure for handling/isolating highly contaminated samples. Samples or extracts designated for volatile organics analysis are segregated from other samples and extracts. Samples suspected of containing high concentrations of volatile organics shall be further isolated from other volatile organics samples.

Storage blanks are used to determine if cross-contamination may have occurred. EMAX-QC03 describes the procedure and criteria for evaluating storage blanks, appropriate to the types of samples being stored.

5.3.4. Access Control

Specific areas in the laboratory can have limited access. Access is controlled but not limited to:

- Sample control room – where samples can only be accessed in the presence of a sample custodian or designee
- Warehouse – where sampling supplies, reagents, etc. are stored
- Analytical labs – are accessed by authorized analysts only during off hours
- Guests – requires escorting

5.3.5. Housekeeping

Good housekeeping promotes safe and environmentally friendly work area. It is an integral part of EMAX's Health and Safety program. Laboratory staff are trained to systematically organize and clean their work area every after use. This routine does not only prevent cross-contamination but also exemplifies respect to co-workers.

The Supervisors are responsible to make sure that their work areas are properly maintained and the Safety Officer conducts periodic inspection to ensure that the practice is sustained.

5.4. Environmental Methods and Method Validation

5.4.1. General

All analytical methods within EMAX's scope of accreditation are adaptations from published reference methods (e.g., USEPA Methods, Standards Methods for the Examination of Water and Wastewater, etc.). These SOPs provide details of the analytical process from sample storage to data reporting. Essential quality control procedures, deviation from the reference method and handling PSRs are also included in each of these SOPs. These SOPs are also updated as necessary.

5.4.2. Selection of Methods

Project Management SOP (EMAX-QA01) describes how methods for environmental testing, including methods for sample preparation, are dispensed to meet the needs of the client and which are appropriate for the environmental tests it undertakes.

Project method requirement shall include target analytes identified by the client on a project-specific basis. If project-specific information is not available, EMAX standard target analyte list shall be used.

If client did not specify the method or the method requested is obsolete, the PM shall suggest an appropriate method to the client. If the client concurs or has a different method preference, the client's preferred method shall be applied.

Where a project requires a method and/or analyte that is not within EMAX's scope of accreditation, EMAX shall make an effort to obtain accreditation. However, if accreditation is not feasible or the process is not completed prior to project commencement, EMAX shall inform the client about the accreditation status. Where client chooses to proceed with the analysis, EMAX shall identity those method(s) and/or analyte(s) in its report and their current accreditation status.

5.4.3. Laboratory Developed Methods

Method Development SOP (EMAX-QA03) details the process of method development for new methods and/or existing methods where new technology or a different technique can be applied to improve the method.

Hypothesis shall be drawn based on similar concepts from developed methods and shall be adjusted accordingly as the method development progresses. Process documentation is imperative in method development for proper course of action and generation of SOP. Method development shall be concluded with method proficiency. Method proficiency shall include detection limit study, demonstration of capability, bias and precision studies and successful analysis of PT sample (when available).

5.4.4. Non-Standard Methods (Not Applicable)

5.4.5. Validation of Methods (Not Applicable)

5.4.6. Estimation of Uncertainty

Factors affecting measurement uncertainty is estimated based on the complexity of the process. Note that the analytical process is only a fraction of the total measurement uncertainty for a target analyte. Hence, EMAX can not make measurement uncertainty corrections on sample results.

In the event that a project will require uncertainty of measurement to be applied to sample results, EMAX shall secure guidance for estimating measurement uncertainty from the client to prevent misinterpretation of results.

In the absence of guidance measurement uncertainty shall:

a) Base its estimation of measurement uncertainty on laboratory control samples (LCS). LCSs are subjected to all possible sources of measurement uncertainty (e.g., sample sub-sampling, sample preparation, sensitivity of instrumentation, instrument calibrations, qualitative and quantitative evaluation). Hence, reasonable estimation of measurement uncertainty is revealed by the statistical evaluation of the LCS results at 99% confidence level.

b) Method specific measurement uncertainties as published.

c) Shall only apply measurement uncertainties when the project requires and the client concurs with EMAX's estimation of measurement uncertainty.

5.4.7. Control of Data

5.4.7.1. Data evaluation and review process are detailed in every analytical SOP. In addition, Data flow and Review SOP (EMAX-DM01) describes the general process of data generation and review. These SOPs include policies and procedures for:

a) checking transcription and calculation errors

b) data review against quality control procedures

c) counterchecking manual calculations

d) proper manual integration and its documentation

5.4.7.2. Where EMAX uses automated data acquisition, processing, recording, reporting, storage or retrieval of test data or calibration data, EMAX has established the following:

a) Documentation and validation of software developed in-house are detailed in the following SOPs:

- Software Documentation (EMAX-IS01)
- Software Testing and Quality Assurance (EMAX-IS03)

b) Data Security SOP (EMAX-IS08) details the policies and procedures for protecting laboratory programs and electronic data. Furthermore, EMAX has developed Ethics Program (EMAX-QA10) to uphold integrity and confidentiality of data entry or collection, data storage, data transmission and data processing.

c) Hardware Maintenance SOP (EMAX-IS12) details computers attached to automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of environmental test data.

Commercial off-the-shelf software (e.g. word processing, database and statistical programs) in general use within their designed application range is considered to be sufficiently validated. However, laboratory software configuration or modifications must be validated as in 5.4.7.2a.

d) Access controls, backup and archival, and routine disaster recovery are established and maintained by the Network Administrator to maintain security of data. Network users are issued passwords by the

Network Administrator. EMAX network is limited access. Only authorized personnel can amend computer records as permitted by their passwords.

e) The Network Administrator shall conduct initial computer security awareness training prior to issuance of a password. A refresher course shall be conducted on an annual basis by the Network Administrator.

f) Internal assessment shall include inspection of electronic data management. At a minimum, a check if the backup and recovery process is exercised at a frequency specified in EMAX-IS09.

g) The PM shall inform the client about any changes that may affect electronic deliverables and its associated data (raw data, calibration data, processed reports, etc.).

h) Spreadsheets that are used for laboratory templates are read-only and protected to prevent accidental or intentional eradication or deletion of embedded formulas and macros. Spreadsheet verification is accomplished prior to release for use. Documentation of verifications is maintained by the QA and DV Department.

i) For computer software developed in-house, policies and procedures are detailed in the following SOPs:

- Software Documentation (EMAX-IS01)
- Software Development Methodologies (EMAX-IS02)
- Software Testing and Quality Assurance (EMAX-IS03)
- Software Maintenance (EMAX-IS04)

j) Record to demonstrate the validity of laboratory generated software are stored in EMAX Network.

k) Electronic Data Security measures are detailed in EMAX-IS08.

5.5. Equipment

5.5.1. EMAX major Instruments are listed in APPENDIX B-3. In addition, all support instruments to support quality control and analytical processes are adequately provided for the laboratory. These instruments are obtained and maintained as required by the Standards governing this manual.

5.5.2. Instrument (and its software, as applicable) calibration capability is always a deciding factor in considering instrument acquisition. Both qualitative and quantitative sensitivity and accuracy are carefully looked at prior to purchase. Operations have to meet sensitivity and all calibration requirements before an instrument is put to use.

5.5.3. Proper use of equipment is a significant part of training. It includes daily and periodic instrument care and detecting apparent instrument breakdown and troubleshooting when doable in-house. Analysts shall complete demonstration of capability prior to operating any equipment on their own.

5.5.4. Each instrument is uniquely identified and is traceable to the data it generates. Data acquisition software (where applicable) is identified in the instrument maintenance logs.

5.5.5. A database of major instruments is maintained by the QA & DV department where instruments are logged and given an instrument number when acquired. At a minimum, the instrument information includes but not limited to the following:

a) serial number

b) manufacturer

c) model

d) detector (where applicable)

e) data acquisition software (where applicable)

f) date put to service

g) location in the lab

h) instrument condition (new or used)

i) comments to record any other relevant information about the instrument

Every analytical instrument has a maintenance logbook where instrument checks are recorded. These logs shall have the instrument number, the data acquisition software version and current equipment parameter setup.

5.5.6. EMAX facility was built to suit for proper instrument upkeep. Each instrument is strategically and logistically positioned in consideration for ease in handling, prevention of contamination and health hazard.

5.5.7. Analytical SOPs detail the process of handling instruments when out of control. While troubleshooting is ongoing, the instruments are clearly identified by an "OUT-OF-SERVICE" tag. Instrument maintenance is recorded in their respective instrument maintenance logs and shall indicate when it was back to control.

5.5.8. All instruments shall bear identification traceable to all other information relevant to its use and maintenance where applicable (e.g., records of: calibration, maintenance, analysis, etc.).

5.5.9. All instruments currently used at EMAX are purposely intended for its permanent facility use only and all maintenance services are done in-house. When external use of any of the instruments is necessary or instruments are sent out for repair, the same quality control checks shall be applied prior to instrument use to ensure that the instrument is properly functioning.

5.5.10. Continuing calibration checks shall be carried out in accordance to the method or the PSR.

5.5.11. Correction factors, where applicable, shall be applied accordingly. Where software is elemental in applying the correction factors, (e.g., Inter-Element Correction factors, retention time windows, calibration factors, etc.) the data acquisition software is updated in accordance to the method and/or instrument requirement prior to instrument use. Where correction factors are applied in support instruments, (e. g. thermometers) readings shall be recorded as observed and corrected values.

5.5.12. Access control of instruments is password protected to prevent accidental and/or intentional changes to instrument parameter setups including correction factors where applicable.

5.5.13. Support Instruments

5.5.13.1. Support instruments contributory to accuracy of analytical results (e.g., balances, ovens, refrigerators, freezers, incubators, water baths, thermometers, micropipettes) shall be maintained to deliver required accuracy.

a) Records of repair and maintenance activities shall be maintained by the user.

b) Calibrations shall be verified at least once a year using traceable reference standards. Reference standards used for calibration verification in-house shall be sent to a NIST certified calibration laboratory for calibration at least once a year. Support instruments that does not have a valid calibration shall be

- Removed from the workbench; or
- If calibration results require correction factor, the device (e.g., thermometer, balance) shall bear the correction factor that needs to be applied to correct the measurement.

c) All raw data obtained during the calibration shall be recorded in their respective calibration logs.

d) Calibration checks for balances shall be performed prior to its use for the day. Balances shall be checked within the expected range of use with standard weights having a valid certificate calibration.

Temperatures of refrigerators and freezers shall be checked daily with a calibrated thermometer.

Temperatures of ovens and water baths shall be checked daily or prior to its use for the day with a calibrated thermometer.

Thermometers shall be calibrated against a thermometer reference standard having a valid certificate from a NIST certified calibration laboratory or equivalent.

e) Calibration checks for volumetric dispense devices (except Class A and glass microliter syringes) shall be performed on a quarterly basis.

f) Performance checks, frequency and acceptance criteria for support instrument calibrations shall be performed as required by the application for which the instrument is used, otherwise adhere to the following:

1) EMAX-QC03 for refrigerator/freezer monitoring
2) EMAX-QC04 for balance calibration check and standard mass verification
3) EMAX-QC05 for thermometer calibration check and standard thermometer verification
4) EMAX-QC06 for micropipette calibration check and volumetric/non-volumetric measuring labware accuracy and precision check
5) EMAX-QC01 for reagent water monitoring

5.6. Measurement Traceability

5.6.1. General

Analytical SOPs detail the required instrumentation, calibration process, analytical standards to be used and calibration validity.

5.6.2. Specific Requirement

5.6.2.1. Instruments, analytical standards, calibrations (initial, verification or continuing) raw data and reports generated are all linked through documentation process for traceability purposes. Analytical standards are purchased from vendors whose measurements are linked to SI units of measure and are prepared using support instruments traceable to SI units of measure.

5.6.2.2. A valid calibration must exist prior to instrument use. Where possibility of traceability is not possible, analytical process must demonstrate that method requirement is fulfilled.

5.6.3. Reference Standards and Reference Materials

5.6.3.1. Reference Standards. Where possible, reference standards are purchased as certified standards traceable to SI units of measure. Certificate of analysis shall include the purity, traceability and ISO accreditation or equivalent.

5.6.3.2. Reference Materials. Where possible, reference materials are purchased as analytical grade traceable to SI units of measure.

5.6.3.3. Intermediate Checks. Reference standards and reference materials are verified using secondary source when commercially available.

5.6.3.4. Handling and Storage. EMAX-QC02 details the process of analytical standard handling and storage.

5.6.4. Documentation

5.6.4.1. Reference Standards or reference materials used for calibration shall be applicable for field samples, quality control samples and performance tests samples. Records of such shall be traceable in laboratory logbooks.

5.6.4.2. Documentation and labeling of analytical standards are detailed in EMAX-QC02.

5.7. Collection of Samples

5.7.1. Sampling

Sample management establishes a process to ensure that samples are properly received and stored. It also ensures that statistically relevant representative samples are taken and that all information relevant to the sample and sub-sampling processes are recorded accordingly.

5.7.2. Project Management

EMAX-QA01 details the process of disseminating PSRs. This process will also handle special instructions (changes, additions and/or deletions from a method or program requirement) to achieve project data quality objectives.

5.7.3. Documentation

The internal chain-of-custody (ICOC) shall document the traffic of samples going in and out of the sample control room, who relinquished/withdrew/returned the sample(s) and when the transfers were done. Details of such process are described in EMAX-SM01.

PSR is enclosed in every work order distributed in the bench. This contains all requirements necessary to complete the work order. The process of accomplishing the PSR is detailed in EMAX-QA01.

Sample preparation logs are used to record sub-sample amounts taken for extraction, digestion, leaching, or preparation for analysis. The process is detailed in sample preparation or analytical SOPs.

5.8. Handling Samples and Test Items

5.8.1. Handling Samples

EMAX-SM01 details the process of sample control. Every sample received is uniquely identified. Sample integrity shall be maintained during receipt, storage and retention and residual samples are disposed according to established policies and procedures.

5.8.2. Test Items

Every sample analyzed is uniquely identified, may it be quality control samples, field samples, calibration samples, instrument performance samples, etc. EMAX-SM04 details the process of identifying different analytical codes in order to identify test samples distinctively. This identification is linked to all records (sample ID, logbooks, raw data, reports, calibrations, standards, etc.) for traceability purposes.

Procedures for sample handling specific to each method are described or referenced in every analytical SOP.

5.8.3. Documentation

Sample handling, testing and any deviation from the standard practice or any anomaly noted during the process shall be recorded in the appropriate document. When the irregularity encountered affects the data quality objective of the project, the client shall be informed accordingly.

5.8.4. Facilities

Sample control room and extract storage are maintained to ensure proper sample preservation and prevent cross-contamination. Refer to EMAX-QC04 for details.

Likewise, sample preparation and analytical laboratories are maintained to facilitate an environmentally friendly workplace keeping cautious consideration of contamination prevention.

5.9. Quality Assurance of Environmental Testing

5.9.1. Validity of test results is dependent on acceptability of applicable quality control measures.

a) Appendix 1 of the analytical SOPs includes quality control procedure (QCP) that must be observed for every analytical process. Refer to analytical SOPs for details.

b) Generally, project data quality objectives are incorporated in the project statement of work (SOW) or quality assurance project plan (QAPP). Project control limits are utilized as an on-going basis to control every sample analyzed. In the absence of project control limits, in-house control limits are applied.

c) EMAX-QA06 – Control Chart SOP describes the process of generating and updating control limits. In addition, this SOP details the process of monitoring the different analyses performed at EMAX. This practice will identify if there are variations that are inherent to the process or random in nature. This SOP also describes interim control limits to analytes with no statistical data.

d) Sample(s) shall be retested where validity of result is in question (e.g., contamination is suspected, over calibration range, etc.). Tentatively identified target analytes are confirmed as directed by the method.

e) 200% data review is performed prior to data packaging, PM's completeness review is performed prior to data package delivery and QA review is performed on 10% of data packages produced on a quarterly basis. If data quality issues are discovered during the review, the client shall be notified within fifteen (15) business days of the discovery of the issue.

5.9.2. Re-analysis of field samples shall be performed when quality control determinant does not warrant data to be reported. In the event that re-analysis is not possible, the client is informed unless otherwise pre-existing instruction is stipulated in the contract.

5.9.3. Quality control samples are prepared and analyzed with field samples at a frequency required by the project. In the absence of PSR, in-house QCP is applied.

5.10. Reporting the Results

5.10.1. General

Sample result summary forms contain all pertinent information relevant to results being reported. Details on how results are reported depend on PSR (e.g., criteria for reporting non-detects, data flagging, significant figures, etc.). Typically, these forms are accompanied with quality control summary forms where conformance (or non-conformance) to control limits is demonstrated and a case narrative summarizing the condition of the analytical process. When required, calibration summaries, preparation/analysis logs, or any other PSR are also included in reporting the results.

5.10.2. Test Reports

EMAX standard reporting formats for analytical results are included in every analytical SOP. Where project deliverables require a different reporting format, the PM shall coordinate with the parties involved to determine the necessary measures to meet the project requirement.

At a minimum, test reports contain the following:

a) Cover Letter – contains the following:

1. Report date
2. Report ID – EMAX batch No.
3. Attention line – report recipient as specified in the project
4. Client Name
5. Address – where report is to be delivered as specified in the project
6. Subject – Laboratory Report
7. Project Name
8. Date samples were received
9. Cross reference table with client sample IDs, EMAX lab IDs, collection date, matrix and analyses requested.
10. Authorized report signatory and function
11. Applicable accreditation(s)

b) Sample result summary forms contain the following:

1. Analytical Method applied
2. Client and Project
3. Sample IDs (client and EMAX)
4. Relevant Dates (sampled, received, prepared/extracted/digested/leached, analyzed)[1]
5. Dilution factor
6. Matrix
7. Moisture content (all soil samples are reported in dry weight unless otherwise specified by the project)[2]
8. Data file ID
9. Calibration reference
10. Preparation batch
11. Sample results with applicable qualifiers as required by the project[3]
12. Detection limits (LOQ, LOD, DL)
13. Surrogates, as applicable (results, spike amount, recoveries)

c) Quality Control samples summary forms (if required) contain the following:

1. QC sample IDs (client and EMAX)
2. QC sample result
3. Percent recovery
4. RPD (where applicable)
5. Project QC limits

d) Calibration Summary forms (if required) contain the following:

[1] TNI EL-VM2-2011/DoD/DOE QSM 5.1, 5.10.11.a
[2] TNI EL-VM2-2011/DoD/DOE QSM 5.1, 5.10.11.b
[3] TNI EL-VM2-2011/DoD/DOE QSM 5.1, 5.10.11.e

1. Calibration data file IDs
2. Calibration Date
3. Results
4. Recoveries
5. Method required limits, unless otherwise specified by the project

e) Case Narrative discusses the following:

1. Holding time
2. Calibrations
3. Applicable QCs (MB, LCS/LCD, MS/MSD/MD, MRL, etc.)
4. Sample Analysis (manual integration, anomaly encountered, opinions and interpretations[4], etc.)

5.10.3. Reporting Analytical Conditions

5.10.3.1. Analytical conditions shall be reported in the case narrative and shall include the following:

a) Applicable test methods. Where deviation from applicable test method is imposed by a sample condition or any unavoidable circumstance, discussion of such shall be included in the case narrative.

b) Statement of quality control compliance or non-compliance

c) Reporting measurement uncertainty is only applied when required by the project.

d) Condition of sample analysis. In instances where results are outside the norm, requiring interpretation, it shall include description of the uncharacteristic result (e.g., manual integration, non-conformance to holding time, sample storage temperature out-of-range, etc.).

e) Statement of compliance to method and project requirement.

5.10.3.2. Data qualifiers shall be applied as required by the project. In the absence of project data qualifiers, EMAX standard qualifiers shall be applied. Refer to EMAX-DM01 for details.

5.10.3.3. Sample condition is documented in sample receipt form (SRF) as they are received in the lab. If samples received are not in a manner as expected (preservation, container, amount), discrepancies are noted in the SRF.

5.10.4. Calibration Certificates

EMAX Laboratories, Inc. does not issue calibration certificates.

5.10.5. Opinions and Interpretations

When opinions and interpretations are mandated by the condition of sample test results, they shall be discussed in the case narrative. The discussion shall include the basis upon which the opinions and/or interpretations have been made.

5.10.6. Test Results From Subcontractors

Test performed by subcontractors are reported as delivered by the subcontracting lab to EMAX. Likewise, the subcontractor shall be liable to the authenticity, correctness and accuracy of its report. Unless stipulated in the contract, the subcontractor shall be required to report the results in writing or electronically as specified by the project. EMAX shall retain a copy of the subcontractor's report and stores it using EMAX archival system.

[4] DoD/DOE QSM 5.1, 5.10.5

5.10.7. Electronic Transmission of Results

Electronic transmission of results shall be carried out as instructed by the project requirement. All reasonable steps to preserve confidentiality shall be taken into consideration.

5.10.8. Format of Report

A cover letter, chain of custody, sample receipt documentation, reports of all analyses within a sample delivery group (SDG) and other related documents essential to the sample results being reported are collated into a data package. The data package is identified by its SDG. The procedure for data package assembly is detailed in EMAX-DM03, unless otherwise directed by the project.

When informed that the data is intended for regulatory report[5], report format shall be prepared as stipulated in the contract. If EMAX is responsible for generating the regulatory reports, the PM shall coordinate with the responsible parties to generate the report in accordance to the reporting requirement. However, if the client requires a different format, all pertinent information to complete the regulatory report shall be provided to the client.

5.10.9. Amendments to Test Reports

Where amendments to transmitted test report are necessary, a supplemental report shall be provided. The cover letter shall include what was changed and/or added and the subject line shall read:

"Supplement to Laboratory Report, [EMAX batch number]"

Where a part of the report is to replace a particular page, that page is numbered with the same number as the previous report stamped with "Revised Report". Where a page(s) is inadvertently missed and is to be added in the previous report, the whole report is repaginated and resubmitted.

When it is necessary to issue a complete new test report and no specific instruction for report re-submittal was provided by the project, the cover letter shall state that it is a full re-submittal and shall replace the previously submitted report and the subject line shall read:

"Re-submittal to Laboratory Report, [EMAX batch number]"

6.0 HAZARDOUS AND RADIOACTIVE MATERIALS MANAGEMENT AND HEALTH AND SAFETY PRACTICES

DoD/DOE QSM 5.3 Section 6.0 shall be implemented as applicable.

[5] TNI EL-VM2-2011/DoD/DOE QSM 5.1, 5.10.10 Exceptions

EMAX QUALITY SYSTEMS MANUAL 2019 Rev. 5.3

VOLUME 1, MODULE 4, QUALITY SYSTEMS CHEMICAL TESTING

1.0 CHEMICAL TESTING

1.1. Introduction

This module details EMAX's quality culture for environmental testing activities in conjunction to Modules 1 and 2. The core of EMAX's quality culture is to continuously deliver authentic and technically valid results. This can be accomplished by consistency and conformity to quality systems and customer expectations.

1.2. Scope

The important aspects of quality control are included in this module. Specific SOPs detailing the actual practice are referenced in this module.

1.3. Terms and Definitions

This section shall use the same terms and definitions as described in DoD/DOE QSM 5.1 Module 2, Section 3.

1.4. Method Selection

Predominantly, project statement of work (SOW), sampling plan (SAP) or quality assurance project plan (QAPP) dictates the required reference method. Where there is no specific reference method stipulated, the PM shall confer and reach an agreement with the client on which method to apply. If the client shall seek advice or its choice of method is not a valid reference method, EMAX shall provide guidance based on prior knowledge.

In general, EMAX utilizes USEPA methods; Standard Methods for the Examination of Water and Wastewater; State published methods (e.g., 8015AZ, CA Method 399-M) and other methods recognized by the environmental industry (e.g., RSK-175).

EMAX SOPs for regulatory methods are written compliant to the method with no deviations from the method requirements. Analysts performing such methods have full understanding about the regulatory requirements of these methods.

Other analytical SOPs are also written adapting the applicable reference method. Any deviations from the method are detailed in the supplementary section of the SOP.

1.5. Method Validation

1.5.1. Validation of Method

a) The primary considerations for method validation are accuracy, reproducibility and determination of detection limits. Method validation shall ensure that each target analyte are qualitatively identified and quantified accurately as specified in the analytical SOP. Method validations shall be performed as described by the procedures of the relevant SOP including method deviations (if any).

b) Non-reference methods shall undergo method development as described in EMAX-QA03.

c) Non-reference methods shall also be validated as stated in 1.5.1 a). In addition, method precision and bias shall be evaluated.

d) For DoD or DOE-sponsored projects, unless required by the project, consent to use non-standard methods shall be obtained prior to implementation.

e) Method re-validation is a must when modifications cause changes in stoichiometry, technology, instrument tuning acceptance or quantitation ions.

1.5.2. Limit of Detection and Limit of Quantitation

Determination of detection limits is detailed in EMAX-QA04. This SOP describes the process of establishing, validating and verifying detection limits (DL), limit of detection (LOD) and limit of quantitation (LOQ) and their relationships.

1.5.2.1. Limit of Detection (LOD)

Where reference methods specify the LOD (or determination LOD), validation or verification, the reference method requirement shall prevail. Otherwise, LOD shall be established as stated above.

DL is determined for each analyte in a given method adapting SW846 Chapter 1, Method Detection Limit section or based from historical data. A quality systems matrix shall be used in the study. It shall be subjected to the same process as sample results are determined to include sample preparation, applicable extraction cleanup, analysis, data evaluation, result quantitation and qualitative identification.

a) LOD for each analyte per matrix is included in every analytical SOP. The samples prepared for LOD determination were also subjected to sample preparation, applicable extraction cleanup, analysis, data evaluation, result quantitation and qualitative identification.

b) Study samples for LOD are spiked at 2-4 times the DL value. LOD is determined by the least concentration where the analyte is qualitatively and positively detected.

c) LOD study is not applicable for analytes for which spiking solutions or quality control samples are not available or otherwise inappropriate.

d) A method blank from the same matrix used for the LOD samples shall be prepared and analyzed along with the LOD samples to confirm absence/presence of interferences in matrix used for the study.

e) Likewise, if there is a change in the method that may impact the sensitivity of the method as stated in 1.5.1.e, LOD verification is a required.

f) LOD verification shall be implemented in accordance with DoD/DOE QSM 5.1, Module 4, Section 1.5.2.1.f).

g) Frequency of LOD verification shall be done quarterly. For methods that are infrequently performed, LOD verification may be performed per analytical batch.

1.5.2.2. Limit of Quantitation (LOQ)

a) LOQ for each analyte per matrix is included in every analytical SOP. Similarly, these samples are processed like LOD.

b) LOQ study is not applicable for analytes for which spiking solutions or quality control samples are not available or otherwise inappropriate.

c) For multi-point calibration methods, study samples for LOQ are spiked at the concentration of the lowest calibration point. LOQ must be within the established LOQ acceptance limits.

d) LOQ must be greater than LOD.

e) Frequency of LOQ verification shall be done quarterly. For methods that are infrequently performed, LOQ verification may be performed per analytical batch.

1.5.3. Evaluation of Precision and Bias

a) Reference Methods. In the absence of reference method guidance, determine precision and bias for each analyte per quality system matrix according to Section 1.6 of this module or from historical LCS data of at least 20 points.

b) Non-reference Methods. Currently EMAX does not have any non-reference methods employed. In the event that EMAX shall develop one, precision and bias shall be established as prescribed in TNI EL-V1M4-2011.1.5.3.b or TNI Standard latest version.

1.5.4. Evaluation of Selectivity

Selectivity is evaluated to extent of which analytes of interest can be qualitatively identified and quantitatively determined without interferences from other target analytes on a given matrix. Acceptance criteria for selectivity are generally prescribed by the reference method and are commonly demonstrated by the instrument performance check. Refer to analytical method for details.

1.6. Demonstration of Capability (DOC)

1.6.1. General

The biggest contributor of the quality output are the personnel dealing with the entire analytical process. This process starts from the time samples are received until all the deliverables are submitted. It is a must that all laboratory personnel who take part in the process are adequately qualified.

Only competent personnel are assigned to perform a given task. Every laboratory employee hired shall undergo training and initial demonstration of capability is completed regardless of education, experience or training prior to its employment at EMAX. Continuing demonstration of capability shall be evaluated once a year thereafter.

Training records of laboratory personnel shall be maintained by the Quality Assurance and Data Validation (QA & DV) department. SOP for training is detailed in EMAX-QA05.

1.6.2. Initial DOC

Initial DOC is performed prior to initial use of a method and every time a method re-validation is done (refer to 1.5.1.e of this module). Initial DOC is also completed when in a period of twelve (12) months the method was not used or the analyst has not performed such method.

1.6.2.1. Initial DOC shall be documented as described in EMAX-QA05.

1.6.2.2. Method requirements for demonstrating proficiency must be observed. If the method does not specify initial demonstration of capability, the procedures described in EMAX-QA05 shall be completed.

1.6.3. Ongoing DOC

The process of evaluating ongoing DOC is also described in EMAX-QA05.

1.7. Technical Requirements

1.7.1. Initial Calibration (ICAL)

1.7.1.1. Instrument Calibration

Accuracy of test results is highly dependent on instrument calibration. Understanding method calibration specificity and instrument operational instructions are essential factors in achieving proper calibration. To ensure that calibration is appropriately pursued specific to method/instrument, instrument parameter setup and calibration procedures are detailed in every analytical SOP.

a) Calibration is initiated by performing ICAL. Analytical SOPs detail the ICAL process including analytical standards (source, preparation, concentration levels, and target analytes), instrument parameter setup, analysis, evaluation, calculations, acceptance criteria, application and review.

b) Raw data are uniquely identified. They are retained in its original form and edited form (if any) traceable to the instrument identification and ICAL.

c) Primarily, analyte results shall be quantitated based on ICAL, unless otherwise instructed by the method and/or by the project.

d) ICAL are verified with an initial calibration verification standard (ICV) obtained from a secondary source. The primary standard and the secondary source standard may be obtained from same vendor as long as the

secondary source standard is identified as a second source standard (e.g., independently prepared from a different source material). Acceptance criteria shall be based on the project requirement.

e) Acceptance criteria for calibrations are also specified in specific analytical SOPs, unless otherwise instructed by the project.

f) Any value detected below the established LOQ shall be reported in accordance to project requirement.

g) Generally, quantitation range is bracketed by the lowest and the highest calibration points, unless otherwise instructed by the method. Any value above the quantitation range shall be diluted to an estimated factor to bring values within the calibration range or shall be flagged in accordance to the project requirement.

h) Where calibration is standardized with a zero and a single point calibration (IPC, ICP-MS), linear range must be established and verified as required by the method and LOQ verification must be analyzed in addition to the method calibration requirements.

i) When initial calibration does not meet the established acceptance criteria, analytical run shall be aborted; cause of non-conformance shall be evaluated and necessary corrective action shall be applied. Sample analysis shall not commence until a valid initial calibration is completed.

j) All target analytes, as well as surrogates and internal standards (as applicable) shall be included in the ICAL. In the absence of PSR, procedure for initial calibration as described in the analytical SOPs shall be implemented. Analytical SOPs also include qualitative identification, quantitative determination and ICAL evaluation. Refer to analytical SOPs for details.

1.7.2. Continuing Calibration

Where applicable, continuing instrument calibration verification (CCV) is performed as specified by the method, unless otherwise instructed by the PSR. Instrument calibration verification shall include the following elements:

a) Procedure

Detailed procedure on how continuing calibration is performed. Refer to analytical SOPs.

b) Analytes

CCV standards shall include all target analytes except for multi-component analytes (e.g., aroclors, chlordane, toxaphene, TPHs). CCV for such analytes shall be run separately, as necessary.

Concentration of CCV shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range[6].

c) Frequency

i. At the beginning of every analytical batch

ii. At interval required by the method

iii. When the last calibration verification is expired

d) Records

All pertinent records shall be retained to permit reconstruction of the CCV. Traceability to the instrument, ICAL, method, analysis date, analyte (name, concentration, response, etc.) shall be made apparent.

e) Acceptance Criteria

[6] DoD/DOE QSM 5.1, Module 4,1.7.2.c.iv

CCV shall be evaluated against the acceptance criteria as described in the specific analytical SOP. All CCVs must be evaluated and reported in accordance to project requirement. If a CCV is outside the acceptance criteria, corrective action shall be applied.[7]

For DoD/DOE-sponsored projects:

i. If CCV is outside the acceptance criteria and no samples were run after the CCV, two consecutive CCVs can be analyzed to rule out incongruous results.

ii. If both CCVs passed then the results may be reported.

iii. If one of the CCVs failed, determine and fix the problem, then recalibrate.

iv. Re-analyze all samples associated with the non-compliant CCV.

v. In the event that re-analysis cannot be done, the PM shall be notified ASAP for further action.

For other projects:

i. If CCV is bias high and no analyte was detected on the associated samples, then results can be reported. CCV condition shall be discussed in the case narrative.

ii. If CCV is bias low and target analytes are detected above the action level, results can be reported. CCV condition shall be discussed in the case narrative.

iii. Otherwise, determine and fix the problem. Recalibrate and re-analyze all samples associated with the non-compliant CCV.

1.7.3. Quality Control

Quality control samples required for each method are included in the respective SOPs. They are prepared and analyzed at a frequency prescribed by the project.

For DoD/DOE-sponsored projects, in the absence of PSR, DoD/DOE QSM, Appendix B, method specific quality control requirements shall be implemented.

In the absence of PSR, the in-house requirements shall be in effect.

1.7.3.1. Negative Control – Method Performance: Method Blank

a) Function. Method blank (MB) is used to confirm that no contaminant is introduced during the analytical process; hence it is prepared and analyzed using the same process as the field samples.

b) Frequency. MB is prepared in every preparation batch.

c) Matrix. MB matrix shall be similar to the field sample matrix being analyzed. EMAX utilizes reagent water for water samples, target analyte interference-free solid matrix for other soil samples.

Where methods require surrogate spikes, method blank sample is also spiked with surrogate.

d) Application. MB is required, when extraction, clean-up, digestion, leaching or purging process is involved or when something is added into the sample and/or the sample goes through a process where contamination may occur.

1.7.3.2. Positive Control – Laboratory Control Sample

1.7.3.2.1. Function. Laboratory control sample (LCS) is used to determine the efficiency of the analytical process to include sample preparation, clean-up (when performed) and analysis. This control sample is prepared and analyzed using the same process as the field samples.

[7] DoD/DOE QSM 5.1, Module 4,1.7.2.d

1.7.3.2.2. Frequency. LCS is prepared in every preparation batch.

1.7.3.2.3. Matrix. LCS matrix shall be similar to the field sample matrix being analyzed. EMAX utilizes reagent water for water samples, target analyte interference-free solid matrix for other soil samples.

Alternatively, a certified reference material (CRM) may be used for LCS.

Component. LCS shall be spiked with analytes and surrogate(s) as required by the project; otherwise, refer to analytical SOPs for details.

Concentration. If LCS spike concentration for the project is not specified, LCS concentration shall be at or below the midpoint of the calibration range.

1.7.3.2.4. Application. This is applicable to all methods where analytical standards used are amenable to spiking (or where CRM) and standard is commercially available.

1.7.3.3. Sample Specific Control

1.7.3.3.1. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

a) Function. MS/MSDs are analyzed to determine the effectiveness of the analytical process in the sample matrix. These samples are generally designated by the project to fit the project data quality objectives. These samples are prepared and analyzed the same way as the field samples that are included in the preparation batch.

b) Frequency. MS/MSDs are prepared and analyzed at the frequency prescribed by the project. In the absence of MS/MSD specific requirement, the method requirement is applied.

c) Matrix. Generally, for every given matrix, one MS/MSD sample is designated (or chosen in the lab) in every 20 field samples or a fraction thereof granting that sufficient samples are received.

Component. MS/MSD shall be spiked with analytes and surrogate(s) as required by the project; otherwise, refer to analytical SOPs for details.

Concentration. If MS/MSD spike concentration for the project is not specified, MS/MSD concentration shall be at or below the midpoint of the calibration range.

MS QC Limits. Project MS/MSD QC limits shall be used unless directed to use EMAX QC limits. EMAX QC limits are statistically derived from historical data. Refer to EMAX-QA06 for details.

d) Application. This is applicable to all methods where analytical standards used are amenable to spiking and standard is commercially available.

1.7.3.3.2. Surrogate

a) Function. Surrogate(s) is also a positive control with properties that mimic the analytes of interest. Its measurement will be indicative of the effectiveness of detecting the target analytes in the sample.

b) Frequency. Where methods require surrogate(s), each sample (including MB, LCS, MS/MSD samples) is spiked with surrogate(s).

c) Component. Surrogate(s) to be used are specified by the project. Otherwise, surrogate(s) to be used shall be as listed in the analytical SOPs.

Concentration. Unless surrogate concentration is specified by the project or mandated by the method, concentration levels for surrogate(s) shall be as prescribed in the analytical SOPs.

Surrogate QC Limits. Project Surrogate QC limits shall be used unless directed to use EMAX QC limits. EMAX QC limits are statistically derived from historical data. Refer to EMAX-QA06 for details.

d) Application. This is applicable to all methods where surrogate is required by the method.

1.7.3.4. Data Reduction

Procedures for data reduction are included in every analytical SOP.

1.7.3.5. Reagent Quality, Water Quality and Checks

a) Reagents are obtained in accordance to method specific purity requirements. Where reagent purity is not specified, analytical reagent grade or better is obtained. Refer to analytical SOPs for details.

b) Reagent water is either produced in-house or purchased commercially. Both sources of reagent water are checked for its usability prior to use. Refer to EMAX-QC01 for details.

c) Quality control for chemicals specific to their use is detailed in EMAX-QC01. LCS/MS Spiking standards for extractable for every preparation and titrants are verified prior to use.

1.7.3.6. Selectivity

Procedure for qualitative identification of every analyte is detailed in the specific analytical SOP.

a) Tentative analyte identifications are confirmed following the reference method confirmation technique.

b) Reporting analytes requiring confirmation shall follow the PSR. In the absence of PSR, the method reporting requirements shall be implemented. Otherwise, result shall be reported from the primary column or detector. Should there be a scientific valid reason not to report results as described above, the rationale shall be documented and discussed in the case narrative.

c) If tentatively identified analytes cannot be confirmed, the client shall be notified. Unconfirmed results shall be identified by a qualifier flag as specified by the project and shall be discussed in the case narrative.

1.7.4. Data Acceptance/Rejection Criteria

1.7.4.1. Negative Control – Method Performance: Method Blank

The result of method blank shall be a measure to determine if the samples that were prepared and processed with it are contaminant-free.

Acceptance Criteria: If result is < ½ of LOQ then results are considered free from contamination, otherwise corrective action is required if:

a) the concentration > ½ the LOQ and is >1/10th the amount measured in any associated sample or 1/10th the regulatory limit, whichever is greater.

b) the analyte is a common lab contaminant and concentration is >LOQ

Corrective Action: Determine and eliminate the source of contamination. Reprocess all associated samples with necessary control samples.

1.7.4.2. Positive Control – Method Performance: Lab Control Sample

a) The result of LCS is a determinant of the accuracy of the process. Project LCS control limits shall be used unless directed to use EMAX LCS limits. EMAX QC limits are statistically derived from historical LCS data. Refer to EMAX-QA06 for details.

Acceptance Criteria: If result is within LCS control limits then the process is in control, otherwise LCS shall be assessed as follows:

i. When result is biased high and associated samples are non-detects, then the data may be reported with appropriate data qualifier. The PM shall be informed for further instruction.

ii. When result is biased low and associated sample results exceed the regulatory limit/action level, then the data may be reported with appropriate data qualifier. The PM shall be informed for further instruction.

b) Allowable Marginal Exceedances (ME). Driven by the nature of analytical process, at times long list of analytes may tend to have a few analytes that are marginally out of the specified/determined control limits. This may not necessarily signify that the process is out of control.

ME limits are established at ±4 standard deviation from the mean. Result outside control limits but inside ME limits is determined as an *[allowable]* marginal exceedance.

Where projects permit ME application of allowable ME shall adhere to rules set forth by the project. For DoD/DOE-sponsored projects, ME is not allowed for target analytes identified as critical deciding factors (e.g., risk drivers). If ME is allowed and no specific rules are stipulated in the statement of work or in the QAPP, the following guidance shall be applied:

Number of Analytes in LCS	Number of Allowable ME
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

c) For DoD/DOE-sponsored project, application of ME shall be applied if the project statement of work or QAPP allows ME application or consent to apply ME is obtained.

d) Analytes observed exceeding of control limits two (2) out of three (3) consecutive LCS and are not categorized as poor performers are indicative of systemic problem. Determine cause and resolve the issue. Hence, ME is not to be allowed for these analytes.

1.7.4.3. Sample Specific Control

a) Matrix Spike/Matrix Spike Duplicate

Recovery of MS/MSD is a determinant of accuracy of results on a given matrix. RPD of MS and MSD results is a determinant of precision assuming that the matrix is homogeneous. Project MS/MSD control limits shall be used unless directed to use EMAX MS/MSD limits. EMAX QC limits are statistically derived from historical MS/MSD data. Refer to EMAX-QA06 for details.

Recoveries for MS/MSD are compared to the MS/MSD QC limits and RPD is compared to RPD limits. Project required corrective action shall be adhered to if any analyte exceeded the specified control limits. Otherwise, corrective action specified in the analytical SOPs shall be implemented.

b) Matrix Duplicate

Matrix duplicate (MD) samples are analyzed mainly to determine precision and/or sample homogeneity. Although this QC sample is generally prescribed for metals and inorganic analyses, it may also be done for organic analyses mostly driven by the project data quality objectives.

When MD is analyzed, the relative percent difference or absolute percent difference is determined and compared to the project RPD (or %D) limits. In the absence of project RPD (or %D), in-house limits are applied. Results are reported as required by the project.

c) Surrogates

Recovery of surrogates on a given sample is a gauge of accuracy of target analytes detected on a given sample. Recoveries shall be evaluated against project surrogate control limits otherwise, in-house limits shall be used. When recovery exceeded surrogate control limits, project prescribed corrective action shall be applied. When re-analysis is necessary, it shall be performed unless prohibiting factors are present (e.g., re-analysis will cause damage to instrument or chromatography obviously display qualitative and quantitative quandary).

1.7.5. Sample Handling

a) Thermal Preservation

Samples are generally received on coolers cooled by ice unless thermal preservation is not required. Temperature is checked and documented in accordance to EMAX-SM02 SOP for Sample Receiving.

b) Chemical Preservation

Samples that are chemically preserved are also checked and documented during sample receipt with the exception of the samples that are not amenable to be opened or sub-sampled prior to analysis (e.g., samples for volatile analyses, oil and grease). Details for this procedure are also described in EMAX-SM02.

c) Holding Time

Sample holding time is checked against the COC sampling date and time. Maximum holding time for analyses are listed in EMAX-SM02.

For a test with recommended maximum holding time measured in hours, the holding time is tracked by the hour. For a test with recommended maximum holding time measured in days, the holding time is tracked by the day. For a test with a recommended maximum holding time measured in months, the holding time shall be tracked by the month. One month is defined as 30 days.

d) Start Time Determination

For extraction, the moment that extraction solvent touches the sub-sample determines the start time. For analysis, the moment that the extract (or sample) is introduced into the analytical instrument determines the start time.

EMAX
QUALITY
SYSTEMS
MANUAL
2019
Rev. 5.3

APPENDIX
A – REFERENCES
B – EMAX ORGANIZATION & MANAGEMENT

REFERENCES

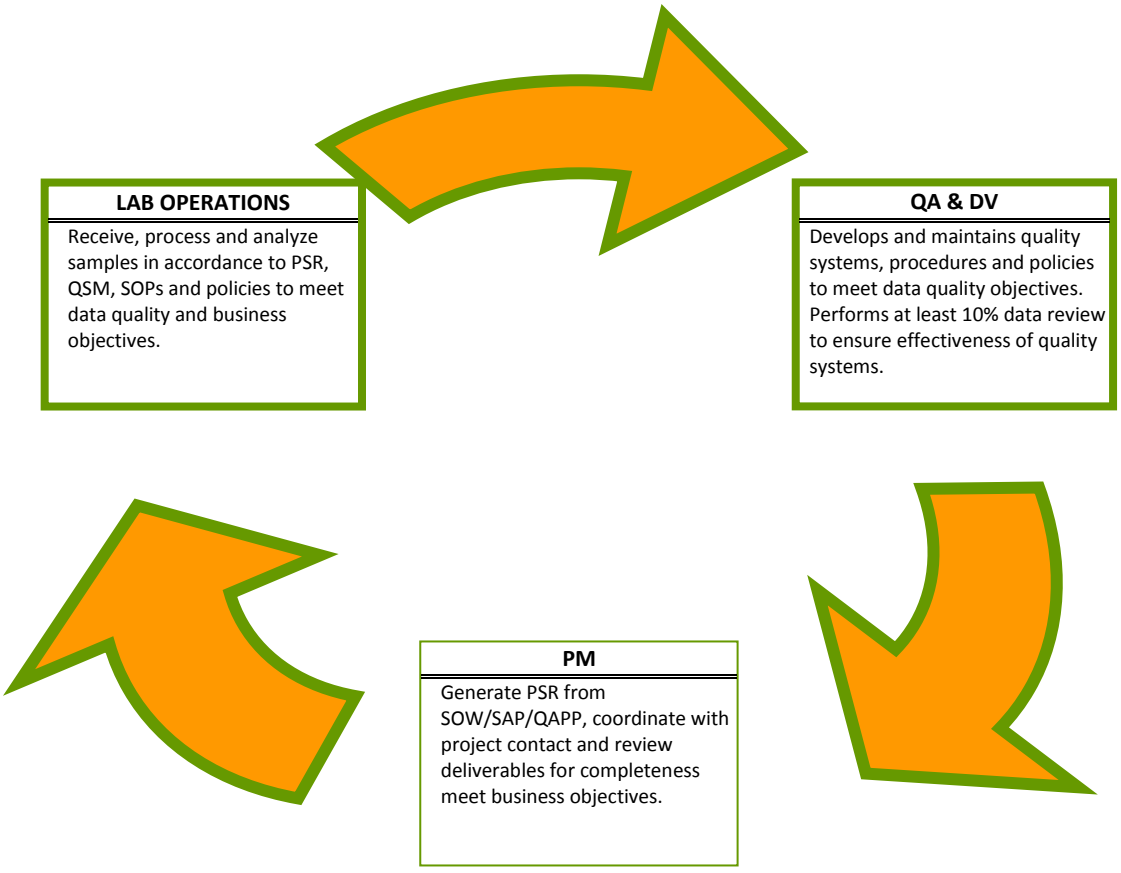
1. The NELAC Institute (TNI) Standard, Environmental Laboratory Sector, Volume 1, 2011
2. Department of Defense (DoD) Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories , DoD QSM Manual version 5.1, 2017
3. International Standard ISO/IEC 17025:2005(E)
4. U. S. EPA Drinking Water Methods for Chemical Parameters
5. U. S. EPA Methods for Chemical Analysis of Water and Wastes
6. U. S. EPA Hazardous Waste Test Methods / SW-846
7. Standard Methods for the Examination of Water and Wastewater, 21st Edition (2011) and 20th Edition (2005)

EMAX ORGANIZATION & MANAGEMENT
B-1 Management Structure

B-1.1 MANAGEMENT STRUCTURE

EMAX has over 70 staff members in its 27,000 square foot facility. Management Structure is detailed in Figure 1 and Laboratory structure is detailed in Figure 2.

Its technical functions are accomplished by the collective and unified effort of three departments, the Quality Assurance & Data Validation, Project Management, and Lab Operations. Although these departments function independently, internal communications and good collaboration are integrated to achieve one common goal – deliver data in accordance to project specific requirements. In summary these functions are illustrated below.



B-1.2 MINIMUM QUALIFICATIONS AND RESPONSIBILITIES

B-1.2.1 LABORATORY DIRECTOR

B-1.2.1.1 Minimum Qualifications

- Bachelor's degree with a minimum of 24 units of chemistry , 24 units of business management and
- Five years of laboratory experience directly related to environmental analytical services, including at least three years of supervisory experience.

B-1.2.1.2 Minimum Responsibilities

- Provide intellectual leadership in technical operations, project management and logistics of domain technical knowledge;
- Provide a healthy work environment
- Interface with Operations and Quality Assurance in the formulation and implementation of the Quality Systems;
- Ensure clear and consistent communication, and coordinate a team of professionals and their respective departments to promote positive corrective action in solving encountered problems;
- Drive project management activities to influence quality output with the goal of enhancing customer satisfaction;
- Review the effectiveness of Quality Systems and instigate improvement accordingly;
- Delegate deputies in case of absence of the Laboratory Director, Technical Operations Manager and/or Quality Assurance Manager;

B-1.2.2 TECHNICAL OPERATIONS MANAGER

B-1.2.2.1 Minimum Qualifications

- Bachelor's degree in chemistry or any science/engineering discipline and
- Five years of laboratory experience in analytical field, including at least three years of supervisory experience.

B-1.2.2.2 Minimum Responsibilities

- Provide direction in technical operations, guidance of project execution and assessment of operations capacity.
- Certify quality of technical staff to perform the test methods specified in this manual.
- Plan and adjust work operations to meet various project requirements or quick turn-around-time without sacrificing the quality and quantity of work;
- Coordinate and integrate the work activities and resources of the different departments or organizational segments;
- Analyze laboratory organizational structure and operational problems and develop timely and economical solutions;
- Establish performance goals and assess progress toward their achievement;
- Effectively deal with the department supervisors and assume their tasks in their absence.
- Assumes technical responsibilities of the Laboratory Director as necessary and perform secondary data review as needed;
- Devise ways to accommodate work operations to new and changing programs or requirements such as method development and staffing

EMAX ORGANIZATION & MANAGEMENT
B-1 Management Structure

B-1.2.3 QA MANAGER

B-1.2.3.1 Minimum Qualifications

- Bachelor's degree in chemistry or any science/engineering discipline and
- Five years of laboratory experience, including at least one year of applied experience dealing with QA principles and practices in an analytical laboratory.

B-1.2.3.2 Minimum Responsibilities

- Serve as the focal point for quality assurance and be responsible for maintaining the Quality Systems Manual;
- Performs at least 10 % of data package produced; evaluates data objectively and perform assessments independently from laboratory operations, without outside or management influence;
- Evaluates the appropriateness and effectiveness of corrective action taken for non-conforming events and notify the laboratory management of any deficiency requiring resolution or further action;
- Responsible for annual internal assessment of the laboratory and ensure that assessment findings are acted upon on a timely manner;
- Certify quality of technical staff to perform the test methods specified in this manual;
- Responsible for ensuring continuous improvement of the quality systems through process knowledge, the use of lessons learned from non-conforming events, proficiency testing, internal audits, control charts and management reviews;
- Assumes technical responsibilities of the Laboratory Director as necessary;

B-1.2.4 PROJECT MANAGER

B-1.2.4.1 Minimum Qualifications

- Bachelor's degree in chemistry or any science/engineering discipline and
- Three years of analytical laboratory experience including sample analysis, data validation, and QA activities.

B-1.2.4.2 Minimum Responsibilities

- Review project requirements;
- Generate technical summary of project specific requirements (PSR), including test methods, reporting limits, QC procedures and QC limits;
- Review data packages for completeness in accordance to PSR
- Maintain a line of communication and documentation of all transactions with the client.
- Initiate request for variance whenever necessary and disseminate project change orders to respective parties.

EMAX ORGANIZATION & MANAGEMENT
B-1 Management Structure

B-1.2.5 DEPARTMENT SUPERVISORS

B-1.2.5.1 Minimum Qualifications

- Bachelor's degree in chemistry or any science/engineering discipline and
- Three years of analytical laboratory experience, including at least one year of supervisory experience.

B-1.2.5.2 Minimum Responsibilities

- Assign and review the work of subordinates;
- Train and work effectively with subordinates;
- Accomplish the quantity of work expected within set limits of cost and time employing the quality systems of EMAX;
- Plan own work and carry out assignments effectively;
- Arrange for or conduct maintenance of instrumentation to produce quality data and minimize downtimes;
- Review and resolve reported anomalies;
- Communicate and resolve work related concerns or challenges and institute preventive measures moving forward;
- Ensure initial and continuing demonstration of capability of subordinates;
- Review and revise test methods related to respective work as necessary;
- Understand and advance management goals as these affect day-to-day work operations; and
- Develop improvements or design new test methods through method development process;

B-1.2.6 ANALYSTS

B-1.2.6.1 Minimum Qualifications

- Bachelor's degree in chemistry or any science/engineering discipline or in lieu of minimum education requirement, four years experience in operating and maintenance in the related field of service.
- Two years experience in related field of service, such as GC/MS, GC, ICP, etc.

B-1.2.6.2 Minimum Responsibilities

- Complete initial demonstration of capability before assuming responsibility and ensure continuing demonstration of capability in accordance with the quality systems;
- Plan own work and carry out assignments effectively
- Review and employ appropriate test methods and SOPs related to respective works;
- Perform first level of 100% review on sample results in accordance to work order specifications, method requirements and as described in EMAX-DM01.
- Report anomalous circumstances to the Supervisor or the Operations Manager; and
- Accomplish the quality and quantity of work expected within set limits of cost and time;

EMAX ORGANIZATION & MANAGEMENT
B-1 Management Structure

B-1.2.7 TECHNICIANS

B-1.2.7.1 Minimum Qualifications

- High school diploma and a college level course in general chemistry or one-year experience of laboratory work.

B-1.2.7.2 Minimum Responsibilities

- Complete initial demonstration of capability before assuming responsibility and ensure continuing demonstration of capability in accordance with the quality systems;
- Plan own work and carry out assignments effectively
- Review and employ appropriate test methods and SOPs related to respective works;
- Report anomalous circumstances to the Supervisor or the Operations Manager; and
- Accomplish the quality and quantity of work expected within set limits of cost and time;

B-1.2.8 NETWORK ADMINISTRATOR MANAGER

B-1.2.8.1 Minimum Qualifications

- Bachelor's degree in computer or any science/engineering discipline with advanced training in information management, database management systems, or systems requirements analysis.
- Three years experience in data or systems management or programming including one year experience in laboratory information management system operations.

B-1.2.8.2 Minimum Responsibilities

- Implement and administer network security policy as described in EMAX-IS08;
- Perform and monitor network backup jobs and process restore requests as well as perform configuration of workstations and mail server as described in EMAX-IS09;
- Manage network password and access control by establishing and maintaining users in regard to their files, rights, and account restrictions. Establish virus protection and perform virus disaster recovery as described in EMAX-IS10;
- Execute software or system releases as requested and maintain documentation of new hardware purchase as described in EAMX-IS12;
- Perform proactive review of systems performance and alerts to ensure that systems are properly protected on a regular basis;
- Review system logs to identify signs of potential problems;
- Review and revise as necessary, all SOPs related to network activities.
- Stay current on technologies affecting current systems infrastructure issues
- Respond to users request on problems encountered regarding hardware, commercially purchased software or any other related network.
- Install, configure, and maintain software and applications needed by the operations.
- Initiate improvements on design systems to meet the industry demand.

EMAX ORGANIZATION & MANAGEMENT
B-1 Management Structure

B-1.2.9 SAMPLE MANAGEMENT SUPERVISOR

B-1.2.9.1 Minimum Qualifications

- Associate's degree with two years of laboratory experience in sample management and at least one year of supervisory experience.

B-1.2.9.2 Minimum Responsibilities

- Supervise sample management as described in the sample management SOPs;
- Check that the sample control room is within the prescribed condition as described in EMAX-QC03.
- Train and work effectively with subordinates;
- Accomplish the quantity of work expected within set limits of cost and time employing the quality systems of EMAX;
- Plan own work and carry out assignments effectively;
- Arrange for or conduct maintenance of sample storage refrigerators/freezers to minimize downtimes;
- Review and resolve reported anomalies when they are encountered;
- Communicate and resolve work related concerns or challenges and institute preventive measures moving forward;
- Ensure initial and continuing demonstration of capability of subordinates;
- Review and revise test related SOPs respective to work practices and/or project requirements as necessary;
- Understand and advance management goals as these affect day-to-day work operations; and
- Initiate improvements on sample management systems to meet the industry demand.

B-1.2.10 HEALTH AND SAFETY OFFICER

B-1.2.10.1 Minimum Qualifications

- Bachelor's degree in chemistry or any science/engineering discipline, with 40-hour training on Hazardous Waste Management.
- One-year experience in administering health and safety regulations.

B-1.2.10.2 Minimum Responsibilities

- Responsible for lab safety policies and procedures and lab safety orientation of new hires.
- Management of employee injuries and exposures to include following up with occurrences, looking at trends and instituting work practice control changes if necessary.
- Regular safety audits which should include fire and electrical safety, ergonomics, chemical hygiene, general housekeeping, waste management, personal protective equipment, and infection prevention.
- Periodic health and safety training for laboratory personnel which includes spill training, fire extinguisher training, fire drills, and several other related health and safety topics.

- Responsible for waste management ensuring proper labeling and disposal of laboratory generated wastes and residual samples.

B-1.2.11. QA & DATA VALIDATION SUPPORT STAFF

B-1.2.11.1 Minimum Qualifications

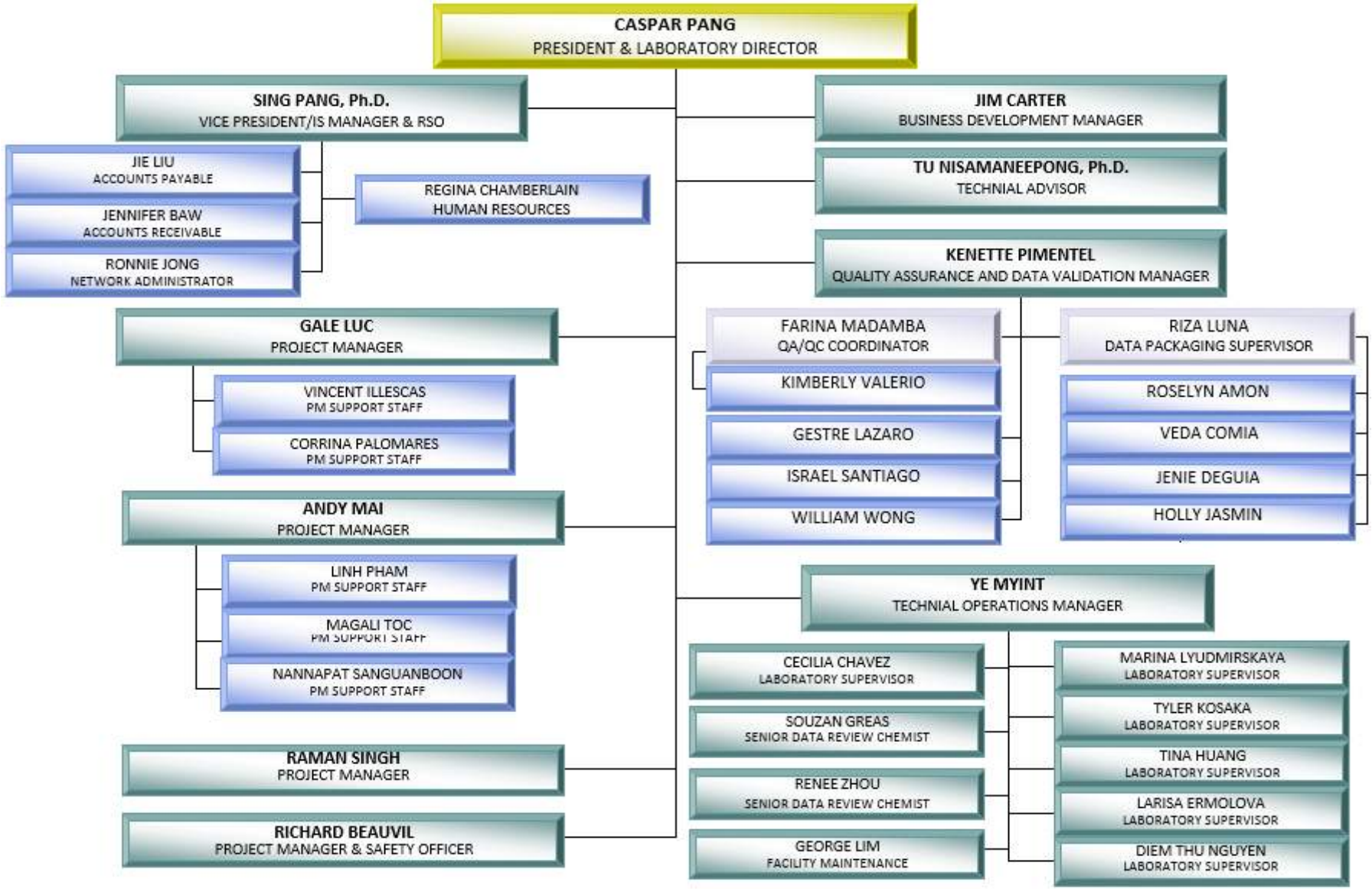
- High School Diploma.
- Two years experience as office support staff or associate degree with computer literacy.

B-1.2.11.2 Minimum Responsibilities

- Assist with quality assurance activities.
- Perform processing and assembly of data deliverables.
- Process requests for laboratory logbooks and templates.
- Update and maintain employee training records.
- Process SOP revisions.
- Update and maintain EMAX Browser.
- Perform other duties as assigned.

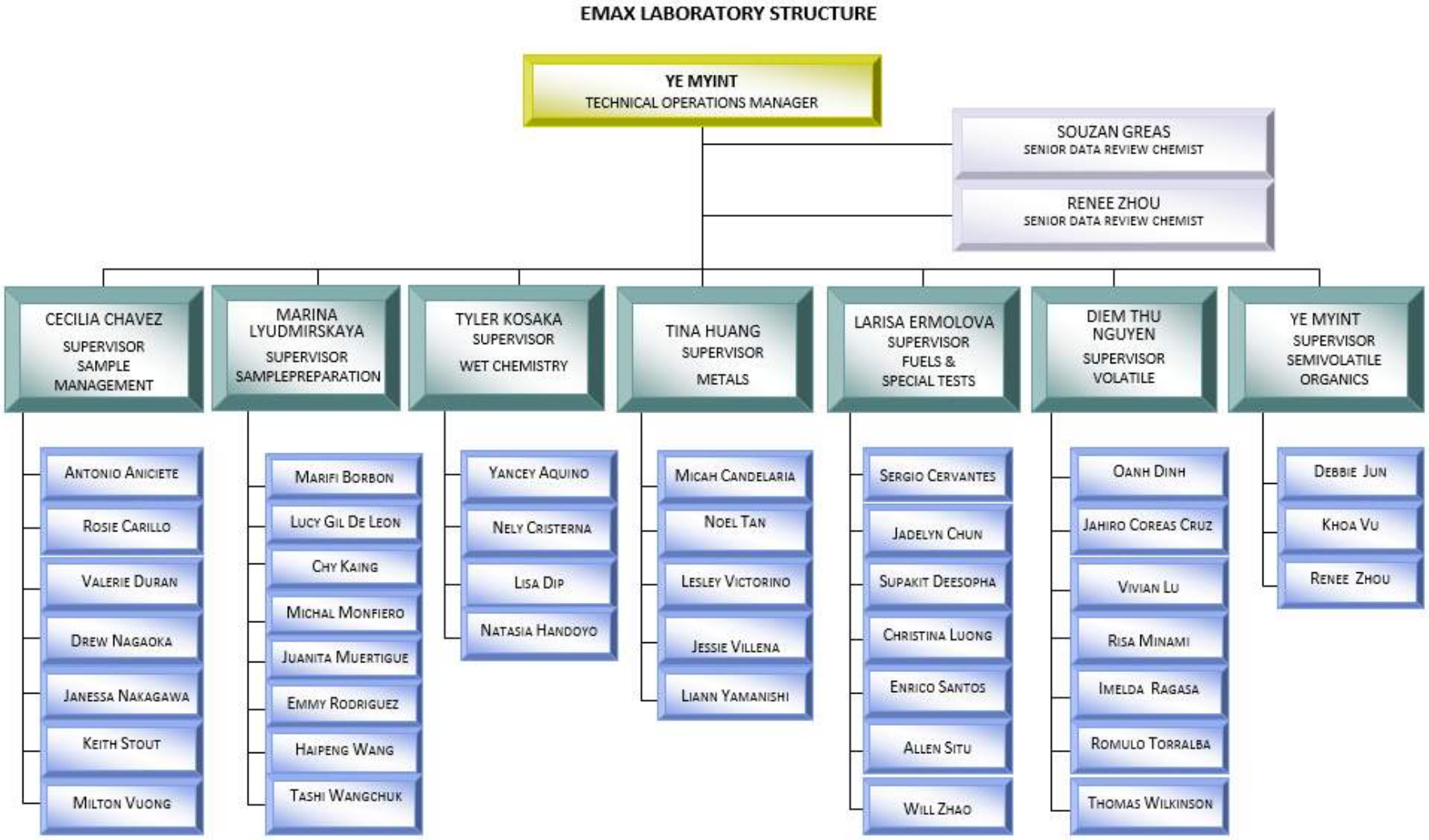
EMAX ORGANIZATION & MANAGEMENT
B-1 Management Structure

Figure 1: MANAGEMENT STRUCTURE



EMAX ORGANIZATION & MANAGEMENT
B-1 Management Structure

Figure 2: LABORATORY STRUCTURE



EMAX ORGANIZATION & MANAGEMENT
B-2 List of Standard Operating Procedures

EMAX SOP LISTS			
ANALYTICAL			
EPA METHODS			
SOP #	Rev. #	Effective Date	TITLE
EMAX-120.1	8	9/19/2018	SPECIFIC CONDUCTANCE
EMAX-150.1	8	9/19/2018	pH MEASUREMENT
EMAX-180.1	9	9/19/2018	TURBIDITY BY NEPHELOMETRIC METHOD
EMAX-200.7	5	8/24/2017	DETERMINATION OF METALS AND TRACE ELEMENTS BY ICP-AES
EMAX-200.8	6	10/30/2018	TRACE METALS BY ICP-MS
EMAX-218.6	7	7/26/2019	HEXAVALENT CHROMIUM
EMAX-245.1	4	7/15/2016	MERCURY (MANUAL COLD VAPOR)
EMAX-300.0	12	8/24/2017	ION CHROMATOGRAPHY ANALYSIS
EMAX-300M	4	12/7/2018	ION CHROMATOGRAPHY ANALYSIS FOR ORGANIC ACIDS
EMAX-314.0	5	2/28/2017	PERCHLORATE BY ION CHROMATOGRAPHY ANALYIS
EMAX-410.4	5	9/19/2018	CHEMICAL OXYGEN DEMAND (COLORIMETRIC, MANUAL)
EMAX-420.1	4	9/18/2017	TOTAL PHENOLS BY SPECTROPHOTOMETRY
EMAX-504.1	6	6/10/2019	EDB AND DBCP
EMAX-524.2	9	7/17/2015	PURGEABLE ORGANIC COMPOUNDS IN WATER BY GC/MS
EMAX-608	2	12/24/2014	ORGANOCHLORINE PESTICIDES & PCBs
EMAX-624	4	6/29/2011	VOLATILE ORGANICS BY GC/MS
EMAX-625	1	8/22/2011	SEMIVOLATILE ORGANICS BY GC/MS

EMAX ORGANIZATION & MANAGEMENT
B-2 List of Standard Operating Procedures

EMAX SOP LISTS

ANALYTICAL

STANDARD METHODS

SOP #	Rev. #	Effective Date	TITLE
EMAX-2120B	5	8/24/2018	COLOR (COLORIMETRIC-PLATINUM-COBALT)
EMAX-2130B	7	9/14/2018	TURBIDITY BY NEPHELOMETRIC METHOD
EMAX-2150B	1	10/11/2017	ODOR
EMAX-2310B	3	9/12/2018	ACIDITY
EMAX-2320B	5	8/24/2017	ALKALINITY
EMAX-2340B	4	8/24/2018	HARDNESS BY CALCULATION
EMAX-2340C	4	4/30/2018	HARDNESS, TOTAL
EMAX-2510B	6	9/12/2018	SPECIFIC CONDUCTANCE
EMAX-2540B	4	4/30/2018	TOTAL SOLIDS
EMAX-2540C	9	4/30/2018	TOTAL DISSOLVED SOLIDS
EMAX-2540D	6	4/30/2018	TOTAL SUSPENDED SOLIDS
EMAX-2540F	4	4/30/2018	SETTLEABLE SOLIDS
EMAX-3500-FeB	5	9/18/2017	FERROUS IRON BY PHENATHROLINE
EMAX-4110B	5	12/3/2015	ION CHROMATOGRAPHY ANALYSIS
EMAX-4500-CIB	3	9/12/2018	CHLORIDE (ARGENTOMETRIC)
EMAX-4500-CNE	6	4/17/2019	CYANIDE, TOTAL
EMAX-4500HB	5	9/12/2018	pH MEASUREMENT
EMAX-4500-NH3F	5	8/24/2017	AMMONIA-N
EMAX-4500-NO2B	3	4/30/2018	NITROGEN, NITRITE (SPECTROMETRIC)
EMAX-4500-NO3E	4	4/30/2018	NITROGEN, NITRATE-NITRITE BY COPPER-CADMIUM REDUCTION
EMAX-4500-PE	5	4/30/2018	PHOSPHORUS (COLORIMETRIC) ALL FORMS
EMAX-4500-S2D	4	4/30/2018	SULFIDE (COLORIMETRY)
EMAX-4500-S2F	6	11/7/2018	SULFIDE (TITRIMETRIC, IODINE)
EMAX-4500-S2H	3	9/25/2017	CALCULATION OF UN-IONIZED HYDROGEN SULFIDE
EMAX-4500-SiO2C	4	4/30/2018	SILICA (COLORIMETRIC)
EMAX-4500-TKNC	4	4/30/2018	TOTAL KJELDAHL NITROGEN
EMAX-4500-TKNF	1	4/30/2018	TOTAL KJELDAHL NITROGEN

EMAX ORGANIZATION & MANAGEMENT
B-2 List of Standard Operating Procedures

EMAX SOP LISTS			
ANALYTICAL			
STANDARD METHODS			
SOP #	Rev. #	Effective Date	TITLE
EMAX-5210B	7	4/30/2018	BIOCHEMICAL OXYGEN DEMAND
EMAX-5220D	4	9/12/2018	CHEMICAL OXYGEN DEMAND (COLORIMETRIC, MANUAL)
EMAX-5310B	4	9/18/2017	TOTAL ORGANIC CARBON
EMAX-5520B	3	8/22/2018	OIL & GREASE (HEM & SGT-HEM)
EMAX-5540C	4	4/17/2019	METHYLENE BLUE ANIONIC SURFACTANTS (MBAS)

EMAX ORGANIZATION & MANAGEMENT
B-2 List of Standard Operating Procedures

EMAX SOP LISTS

ANALYTICAL

SW846 METHODS

SOP #	Rev. #	Effective Date	TITLE
EMAX-1010	5	6/8/2016	IGNITABILITY
EMAX-1664	8	9/18/2017	OIL & GREASE (HEM & SGT-HEM)
EMAX-6010	8	11/1/2018	TRACE METALS BY ICP
EMAX-6010C	2	4/12/2017	TRACE METALS BY ICP
EMAX-6020	10	11/15/2018	TRACE METALS BY ICP-MS
EMAX-6020CA	0	6/5/2019	TRACE METALS BY ICP-MS
EMAX-6850	2	1/18/2019	PERCHLORATE BY HPLC/MS
EMAX-7196	6	8/23/2019	CHROMIUM(VI)
EMAX-7199	7	7/30/2019	CHROMIUM(VI)
EMAX-7470	8	7/13/2016	MERCURY
EMAX-7471	9	7/13/2016	MERCURY
EMAX-8011	6	6/10/2019	EDB/DBCP
EMAX-8015D	7	9/20/2017	DIESEL RANGE ORGANICS (DRO)
EMAX-8015G	5	1/24/2014	GASOLINE RANGE ORGANICS (GRO)
EMAX-8015O	1	7/20/2018	ALCOHOLS AND GLYCOLS BY GC
EMAX-8081	8	6/16/2014	ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY
EMAX-8082	5	7/2/2014	POLYCHLORINATED BIPHENYLS(PCB) AND POLYCHLORINATED TERPHENYLS (PCT) BY G
EMAX-8082CON	1	7/8/2014	POLYCHLORINATED BIPHENYLS(PCB) CONGENERS BY GAS CHROMATOGRAPHY
EMAX-8141	6	1/18/2019	ORGANOPHOSPHORUS COMPOUNDS BY GC
EMAX-8141A	0	5/6/2019	ORGANOPHOSPHORUS COMPOUNDS BY GC
EMAX-8151	6	4/5/2016	CHLORINATED HERBICIDES
EMAX-8260	10	6/6/2014	VOLATILE ORGANIC COMPOUNDS BY GC/MS
EMAX-8260C	1	10/23/2015	VOLATILE ORGANIC COMPOUNDS BY GC/MS
EMAX-8260SIM	1	9/6/2011	VOLATILE ORGANIC COMPOUNDS BY GC/MS SIM
EMAX-8270	6	7/10/2014	SEMIVOLATILE ORGANICS BY GC/MS
EMAX-8270D	1	7/29/2014	SEMIVOLATILE ORGANICS BY GC/MS
EMAX-8270SIM	2	7/5/2011	SEMIVOLATILE ORGANICS BY GC/MS SIM

EMAX ORGANIZATION & MANAGEMENT
B-2 List of Standard Operating Procedures

EMAX SOP LISTS

ANALYTICAL

SW846 METHODS

SOP #	Rev. #	Effective Date	TITLE
EMAX-8310	7	9/20/2017	POLYNUCLEAR AROMATIC HYDROCARBONS
EMAX-8330	8	11/12/2015	NITROAROMATICS & NITRAMINES BY HPLC
EMAX-8332	3	10/29/2015	NITROGLYCERINE BY HPLC
EMAX-9014	8	11/29/2016	CYANIDE, TOTAL
EMAX-9040	9	9/19/2018	pH MEASUREMENT
EMAX-9045	4	6/22/2017	pH, SOLID AND WASTE SAMPLES
EMAX-9050	4	9/19/2018	SPECIFIC CONDUCTANCE
EMAX-9056	8	12/3/2015	ION CHROMATOGRAPHY ANALYSIS
EMAX-9060	4	10/11/2017	TOTAL ORGANIC CARBON
EMAX-9065	3	1/29/2013	TOTAL PHENOLS BY SPECTROPHOTOMETRY
EMAX-9070	0	7/16/2012	OIL & GREASE (HEM & SGT-HEM)
EMAX-9071	1	9/7/2012	OIL AND GREASE
EMAX-9095	3	8/20/2012	PAINT FILTER LIQUID TEST
EMAX-9253	4	10/1/2018	CHLORIDE (TITRIMETRIC SILVER NITRATE)
EMAX-M8260SIM	0	3/14/2011	1,4-DIOXANE BY GC/MS SIM
EMAX-M8270SIM	2	10/31/2011	1,4-DIOXANE BY GC/MS, MODIFIED METHOD 8270SIM
EMAX-MCD	3	9/22/2009	MOISTURE CONTENT DETERMINATION
EMAX-RCN	3	8/19/2011	REACTIVE CYANIDE
EMAX-RSE	4	4/30/2012	REACTIVE SULFIDE
EMAX-TCPSIM	2	9/2/2011	1,2,3-TRICHLOROPROPANE BY GC/MS SIM
EMAX-TO15	5	11/19/2015	DETERMINATION VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR BY GC/MS

EMAX ORGANIZATION & MANAGEMENT
B-2 List of Standard Operating Procedures

EMAX SOP LISTS

ANALYTICAL

SPECIAL TEST

SOP #	Rev. #	Effective Date	TITLE
EMAX-8015AZ	3	7/20/2018	AZ C10-C32 HYDROCARBONS IN SOIL
EMAX-8073	1	3/17/2015	AGGRESSIVE INDEX BY CALCULATION
EMAX-8139	0	7/5/2011	CYANURIC ACID BY TURBIDIMETRIC METHOD
EMAX-939M	3	4/12/2017	ORGANOLEAD ANALYSIS BY GFAA
EMAX-AK101	2	3/8/2013	AK GASOLINE RANGE ORGANICS
EMAX-AK102/103	4	3/30/2017	AK DIESEL/RESIDUAL RANGE ORGANICS
EMAX-D1945	0	3/22/2013	TEST METHOD FOR NATURAL GASES
EMAX-RSK175	4	4/4/2016	DISSOLVED GASES
EMAX-TOCWB	3	3/30/2017	TOTAL ORGANIC CARBON (TOC) BY WALKLEY BLACK METHOD

EMAX ORGANIZATION & MANAGEMENT
B-2 List of Standard Operating Procedures

EMAX SOP LISTS

SAMPLE PREPARATION

SW846 METHODS

SOP #	Rev. #	Effective Date	TITLE
EMAX-1311	3	10/30/2015	TCLP FOR ORGANIC AND INORGANIC ANALYTES
EMAX-1312	1	9/7/2010	SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP)
EMAX-3010	7	12/14/2018	ACID DIGESTION, TOTAL METALS FOR AQUEOUS
EMAX-3050	6	12/14/2018	ACID DIGESTION, TOTAL METALS SOLIDS
EMAX-3060	1	1/22/2015	ALKALINE DIGESTION FOR HEXAVALENT CHROMIUM
EMAX-3520	5	3/19/2012	EXTRACTION, CONTINUOUS LIQUID LIQUID
EMAX-3540	3	1/18/2019	EXTRACTION, SOXHLET
EMAX-3546	0	8/6/2013	EXTRACTION, MICROWAVE
EMAX-3550	5	5/20/2016	EXTRACTION, PULSE SONICATION
EMAX-3580	3	6/27/2019	WASTE DILUTION
EMAX-3620	3	11/9/2016	CLEANUP, FLORISIL
EMAX-3640	3	10/21/2010	CLEANUP, GEL PERMEATION
EMAX-3660	2	10/10/2017	CLEAN-UP, SULFUR
EMAX-3665	3	10/10/2017	CLEANUP, SULFURIC ACID/PERMANGANATE
EMAX-5030	4	9/22/2017	PURGE & TRAP
EMAX-5035	4	9/22/2017	CLOSED SYSTEM PURGE & TRAP
EMAX-WET	3	10/10/2017	WASTE EXTRACTION TEST

EMAX ORGANIZATION & MANAGEMENT
B-2 List of Standard Operating Procedures

EMAX SOP LISTS

DATA MANAGEMENT

QUALITY SYSTEMS

SOP #	Rev. #	Effective Date	TITLE
EMAX-DM01	7	2/8/2017	DATA FLOW AND REVIEW
EMAX-DM02	4	8/4/2014	DOCUMENT CONTROL
EMAX-DM03	4	11/15/2016	DATA PACKAGE ASSEMBLY
EMAX-DM04	1	11/2/2011	MANAGEMENT OF LIMS RAW DATA
EMAX-DM05	0	4/24/2015	REPORTING PERFORMANCE TEST RESULTS

INFORMATION SYSTEMS

QUALITY SYSTEMS

SOP #	Rev. #	Effective Date	TITLE
EMAX-IS01	3	6/17/2014	SOFTWARE DOCUMENTATION
EMAX-IS02	2	6/17/2014	SOFTWARE DEVELOPMENT METHODOLOGY
EMAX-IS03	2	6/17/2014	SOFTWARE TESTING AND QUALITY ASSURANCE
EMAX-IS04	3	6/17/2014	SOFTWARE MAINTENANCE
EMAX-IS05	2	6/18/2014	EDD GENERATION AND VALIDATION
EMAX-IS06	1	3/1/2004	HISTORICAL FILE MAINTENANCE
EMAX-IS07	2	3/1/2004	ACQUISITION OF SOFTWARE PACKAGES
EMAX-IS08	5	6/24/2014	DATA SECURITY
EMAX-IS09	3	7/15/2010	BACKING UP FILES
EMAX-IS10	3	7/15/2010	VIRUS PROTECTION
EMAX-IS11	2	12/21/2012	PROJECT SUPPORT FILES
EMAX-IS12	1	7/15/2010	HARDWARE MAINTENANCE
EMAX-IS13	0	5/12/2007	SHUTDOWN AND STARTUP PROCEDURES

EMAX ORGANIZATION & MANAGEMENT
B-2 List of Standard Operating Procedures

EMAX SOP LISTS

QUALITY ASSURANCE

QUALITY SYSTEMS

SOP #	Rev. #	Effective Date	TITLE
EMAX-QA00	5	10/31/2017	WRITING STANDARD OPERATING PROCEDURES
EMAX-QA01	7	1/9/2017	PROJECT MANAGEMENT
EMAX-QA02	4	11/23/2016	UTILIZATION OF SUBCONTRACT LABORATORIES
EMAX-QA03	3	3/22/2017	METHOD DEVELOPMENT
EMAX-QA04	8	2/6/2019	DETECTION LIMIT (DL)
EMAX-QA05	9	3/13/2019	TRAINING
EMAX-QA06	6	10/10/2017	CONTROL CHART
EMAX-QA07	5	11/15/2016	INTERNAL ASSESSMENT
EMAX-QA08	8	10/1/2018	CORRECTIVE ACTION
EMAX-QA09	5	11/30/2016	PURCHASING
EMAX-QA10	4	9/22/2017	ETHICS PROGRAM

QUALITY CONTROL

QUALITY SYSTEMS

SOP #	Rev. #	Effective Date	TITLE
EMAX-QC01	4	2/15/2017	QUALITY CONTROL FOR CHEMICALS
EMAX-QC02	2	8/16/2010	ANALYTICAL STANDARD PREPARATION
EMAX-QC03	4	10/31/2017	REFRIGERATOR CONTROL
EMAX-QC04	6	1/9/2017	BALANCE CALIBRATION
EMAX-QC05	6	10/10/2017	CALIBRATION OF THERMOMETERS
EMAX-QC06	6	10/15/2015	VOLUMETRIC LABWARE AND MICROPIPETTE VERIFICATION
EMAX-QC07	3	2/6/2019	LABWARE CLEANING
EMAX-QC08	1	7/15/2010	HANDLING HIGHLY CONTAMINATED SAMPLES
EMAX-QC09	2	8/16/2010	FUME HOOD INSPECTION PROGRAM

EMAX ORGANIZATION & MANAGEMENT
B-2 List of Standard Operating Procedures

EMAX SOP LISTS

RADIATION SAFETY
QUALITY SYSTEMS

SOP #	Rev. #	Effective Date	TITLE
EMAX-RS01	5	12/12/2011	SURVEY METER USE
EMAX-RS02	3	12/12/2011	RECEIVING RADIOACTIVE MATERIALS
EMAX-RS03	6	12/12/2011	CONTROL OF RADIOACTIVE SAMPLES
EMAX-RS04	3	12/12/2011	EMERGENCY RESPONSE & DECONTAMINATION OF RADIOACTIVE MATERIAL
EMAX-RS05	4	12/12/2011	RADIOLOGICAL WORKER PERSONNEL MONITORING
EMAX-RS06	4	12/12/2011	FACILITY MONITORING PROGRAM
EMAX-RS07	2	12/12/2011	RADIATION SAFETY INTERNAL AUDIT

SAMPLE MANAGEMENT
QUALITY SYSTEMS

SOP #	Rev. #	Effective Date	TITLE
EMAX-SM01	8	6/15/2018	SAMPLE MANAGEMENT
EMAX-SM02	11	8/8/2019	SAMPLE RECEIVING
EMAX-SM03	6	3/20/2017	WASTE DISPOSAL
EMAX-SM04	1	5/11/2012	ANALYTICAL AND QC SAMPLE LABELING
EMAX-SM05	4	4/22/2015	SAMPLE CONTAINERS, HANDLING AND SHIPPING
EMAX-SM06	2	5/1/2012	TRIP BLANK PREPARATION
EMAX-SM07	1	6/15/2012	OVERSEAS QUARANTINED SOILS

EMAX ORGANIZATION & MANAGEMENT
B-3 List of Major Equipment

EMAX LABORATORIES, INC.

LIST OF MAJOR ANALYTICAL EQUIPMENT

No.	Type	Detector	Use	Manufacturer	Model	Location	Date Acquired
1	GC	MS	524 624 8260	Hewlett Packard	5890/5970	VOLATILES	6/1/1996
2	GC	MS	524 624 8260	Hewlett Packard	5890/5970	VOLATILES	6/1/1996
3	GC	MS	524 624 8260	Hewlett Packard	5890/5870	VOLATILES	6/1/1996
5	GC	MS	524 624 8260	Hewlett Packard	5890/5870	VOLATILES	6/1/1996
6	GC	MS	524 624 8260	Hewlett Packard	5890/5870	VOLATILES	6/1/1996
67	GC	MS	524 624 8260	Hewlett Packard	5890 II/5971B	VOLATILES	9/1/2001
83	GC	MS	TO15	VARIAN	SATURN 2000	VOLATILES	9/25/2002
94	GC	MS	524 624 8260	Hewlett Packard	5890A	VOLATILES	3/24/2003
111	GC	MS	524 624 8260	Agilent	7890A/G3171A	VOLATILES	2/26/2008
124	GC	MS	524 624 8260	Hewlett Packard	5890 SII	VOLATILES	9/21/2010
125	GC	MS	524 624 8260	Agilent	7890A/5975C	VOLATILES	12/17/2010
126	GC	MS	524 624 8260	Agilent	6890N/5973	VOLATILES	1/4/2011

EMAX ORGANIZATION & MANAGEMENT
B-3 List of Major Equipment

EMAX LABORATORIES, INC.

LIST OF MAJOR ANALYTICAL EQUIPMENT

No.	Type	Detector	Use	Manufacturer	Model	Location	Date Acquired
8	GC	Dual ECD	8081 8082	Hewlett Packard	5891 II	SEMIVOLATILES	6/1/1996
9	GC	Dual ECD	8151	Hewlett Packard	5892 II	SEMIVOLATILES	6/1/1996
12	GC	Dual FPD	8141	Hewlett Packard	5890	SEMIVOLATILES	6/1/1996
16	GC	Dual ECD	8081 8082	Hewlett Packard	5890	SEMIVOLATILES	6/1/1996
71	GC	Dual ECD	8082	Hewlett Packard	5890 II	SEMIVOLATILES	12/18/2001
113	GC	MS	625 8270	Agilent	7890A/G3440A	SEMIVOLATILES	9/11/2008
116	GC	MS	625 8270	Agilent	7890A-5975C MSD	SEMIVOLATILES	6/5/2009
117	GC	Dual ECD	8081 8082	Perkin Elmer	Clarus 600GC	SEMIVOLATILES	1/22/2010
118	GC	MS	625 8270	Thermo Fisher	Trace DSQII MS	SEMIVOLATILES	3/1/2010
119	GC	MS	625 8270	Thermo Fisher	Trace DSQII MS	SEMIVOLATILES	3/1/2010
130	GC	Dual ECD	8081 8082 608	Perkin Elmer	PE Clarus 680	SEMIVOLATILES	12/15/2011

EMAX ORGANIZATION & MANAGEMENT
B-3 List of Major Equipment

EMAX LABORATORIES, INC.

LIST OF MAJOR ANALYTICAL EQUIPMENT

No.	Type	Detector	Use	Manufacturer	Model	Location	Date Acquired
13	GC	TCD/FID	CO2	Hewlett Packard	5890	HPLC, FUELS, IC & TOC	6/1/1996
39	GC	FID/PID	8015 (Purgeables)	Hewlett Packard	5890	HPLC, FUELS, IC & TOC	6/1/1996
50	GC	FID	RSK175 / Glycol	Hewlett Packard	5890	HPLC, FUELS, IC & TOC	6/1/1996
55	GC	FID	8015 (Purgeables)	Hewlett Packard	5890	HPLC, FUELS, IC & TOC	8/1/2000
57	IC	Conductivity	314.0 300.0M	Dionex	LC25	HPLC, FUELS, IC & TOC	9/28/2000
59	IC	UV/VIS	218.6/7199	Dionex	LC20	HPLC, FUELS, IC & TOC	6/1/1996
72	GC	FID	8015 (Alcohols)	Hewlett Packard	5890 II	HPLC, FUELS, IC & TOC	12/18/2001
81	HPLC	UV	8310 8330	Waters	717 Plus	HPLC, FUELS, IC & TOC	6/7/2002
100	IC	Conductivity/UV/VIS	300.0	Metrohm	761 Compact IC	HPLC, FUELS, IC & TOC	4/11/2005
105	GC	FID	8015 (Extractable)	Agilent	G1530N,G1540N	HPLC, FUELS, IC & TOC	4/5/2006
106	IC	Conductivity/UV/VIS	300.0M	Dionex	ICS-1000 with AS50 Autosampler	HPLC, FUELS, IC & TOC	5/8/2006
107	IC	Conductivity	300.0	Metrohm	800 Series	HPLC, FUELS, IC & TOC	10/4/2006
112	IC	Conductivity	300.0	Metrohm	881IC PRO	HPLC, FUELS, IC & TOC	6/2/2008
115	HPLC	UV/PDA	8330	ThermoFisher	60057-60002	HPLC, FUELS, IC & TOC	5/27/2009
121	GC	FID	8015 (Extractable)	Perkin Elmer	Clarus 680	HPLC, FUELS, IC & TOC	8/9/2010
131	HPLC	MSQ	6850	Dionex/ThermoFisher	LPG-3400SD	HPLC, FUELS, IC & TOC	1/10/2012
133	IC	UV VIS	218.6 7199	Metrohm	887	HPLC, FUELS, IC & TOC	5/14/2012
149	GC	FID	8015 (Purgeables)	Hewlett Packard	5890	HPLC, FUELS, IC & TOC	8/12/2019

EMAX ORGANIZATION & MANAGEMENT
B-3 List of Major Equipment

EMAX LABORATORIES, INC.

LIST OF MAJOR ANALYTICAL EQUIPMENT

No.	Type	Detector	Use	Manufacturer	Model	Location	Date Acquired
20	GFAA	GFAA	Organo Lead	Varian	Zeeman SpectAA 400	METALS	6/1/1996
47	Mercury Analyzer	Cold Vapor	Mercury by Cold Vapor	Leeman PS-200II	200II	METALS	10/26/1999
74	Mercury Analyzer	Cold Vapor	Mercury by Cold Vapor	Leeman PS	HYDRA-AA	METALS	12/1/2001
98	ICP	MS	Metals by ICP-MS	Agilent	7500	METALS	9/15/2003
108	ICP	AES	Metals by ICP	Thermo	ICAP 6500	METALS	12/1/2006
127	ICP	MS	Metals by ICP-MS	Agilent	7500 CX	METALS	1/7/2011
148	ICP	MS	Metals by ICP-MS	Agilent	7800	METALS	8/1/2019

EMAX LABORATORIES, INC.

LIST OF MAJOR ANALYTICAL EQUIPMENT

No.	Type	Detector	Use	Manufacturer	Model	Location	Date Acquired
28	Flash Point	Thermometer	Ignitability	Pensky Martens	Semi-Automatic	GENERAL CHEMISTRY	6/1/1996
53	pH Meter	pH Probe	pH	Orion	420A	GENERAL CHEMISTRY	4/1/2000
70	Spectrometer	Photodiode	Spectrometric Methods	Thermo Spectronic	Spectronic 20D	GENERAL CHEMISTRY	12/1/2001
104	Conductivity Meter	Platimum	Specific Conductance	TRACEABLE	61161	GENERAL CHEMISTRY	1/2/2006
109	BOD Probe	Dissolved Oxygen Probe	BOD-5day			GENERAL CHEMISTRY	
110	Turbidimeter	Turbidimeter	Turbidimetric	HF INDUSTRIES	HF-MICRO 100	GENERAL CHEMISTRY	8/1/2007
114	PC-Titrate	pH Probe	Alkalinity	MAN-TECH	PC-1300-475	GENERAL CHEMISTRY	10/1/2008
128	pH Meter	pH Probe SN 35805-10	pH 7199	Oakton	pH 510	GENERAL CHEMISTRY	1/1/2009

EMAX ORGANIZATION & MANAGEMENT
B-4 Accreditations

FEDERAL GOVERNMENT VALIDATIONS
DEPARTMENT OF DEFENSE, ELAP
CALIFORNIA RADIOACTIVE MATERIAL LICENSE
DEPARTMENT OF FOOD AND AGRICULTURE, PERMIT TO RECEIVE SOILS FROM OVERSEAS

STATE CERTIFICATIONS
ALASKA
ARIZONA
CALIFORNIA
HAWAII
KANSAS
MARYLAND
NEVADA
NEW HAMPSHIRE
OKLAHOMA
UTAH
WASHINGTON

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

Approved By	Richard Beauvil, Safety Officer	8/26/19 Date
Approved By	Caspar Pang, President	8/26/2019 Date

Document ID	**EMAX-CHP-08**
Revision No.	8
Date Released	08/26/19
Issue No.	**EMAX-CHP-08-**
Issued To	

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

CHEMICAL HYGIENE PLAN

Table of Contents

		Pages
1.0	**Introduction**	**4**
1.1	Objective	4
1.2	Responsibility	4
2.0	**Control Measures**	**5**
2.1	Prior Approval of Hazardous Chemicals	5
2.1.1	Select Cancer Causing Agents	5
2.1.1.1	Regulated as a Carcinogen by OSHA	5
2.1.1.2	National Toxicology Program (NTP) Carcinogenic List	5
2.1.1.3	World Health Organization Carcinogenic Agents	5
2.1.1.4	State of California Reproductive Toxin Chemical List	5
3.0	**Standard Operating Procedures**	**6**
3.1	Chemicals	6
3.1.1	General Rule	6
3.1.2	Acids and Bases	6
3.1.3	Flammable Liquids	7
3.1.4	Toxic Materials	7
3.1.5	Personal Protective Equipment	7
3.1.5.1	Glove Awareness Policy	8
3.2	Compressed Gas Cylinders	10
3.2.1	General	10
3.2.2	Inspection	10
3.2.3	Storage/Transport/Use	11
3.3	Specific Gases	11
3.3.1	Argon	11
3.3.2	Liquid Argon	12
3.3.3	Helium	13
3.3.4	Nitrogen	14
3.3.5	Liquid Nitrogen	14
3.3.6	Hydrogen	15
3.4	Incompatible Materials in the Laboratory	15
3.5	Chemical Spills	16

Pages

3.6 Disposal Procedures for Chemical Wastes 17
3.7 Potentially Explosive Chemicals (including organic peroxide) 18
3.8 Methylene Chloride Exposure Monitoring 19

4.0 Protective Equipment Evaluation 20

4.1 Procedures for Checking Safety Showers/Eye Wash 20
4.1.1 Safety Showers 20
4.1.2 Eye Wash 21

4.2 Instruction for Replacing Portable Emergency Eye Washes 21

4.3 Local Exhaust Systems 21
4.3.1 Baseline Evaluation 22
4.3.1.1 Airflow Measurements of Hoods 22

4.4 Respiratory Protection Program 22
4.4.1 Policy Statement 22
4.4.2 Responsibilities 22
4.4.2.1 Chemical Hygiene Officer 22
4.4.2.2 Divisional/Departmental Safety Representatives 22
4.4.2.3 Employees 23
4.4.3 Respirator Selection 23
4.4.4 Training and Education 23

4.5 Program Evaluation 24

4.6 Respiratory Protective Equipment: Use and Limitations 24
4.6.1 Air Purifying Respirators 24
4.6.2 Chemical Cartridges Respirators Limitations 24

4.7 Respirator Physical Evaluation Form 26

4.8 Medical Summary Form 27

5.0 Employee Information and Training 28

5.1 Employee Information 28

5.2 Employee Training 28
5.2.1 Training Statement 28
5.2.2 Safety Data Sheets 28
5.2.3 Labeling Policy 29
5.2.4 Hazard Determination 30
5.2.5 Employee Training 30

			Pages
	5.2.6	Execution/Assigned Responsibilities	31
		5.2.6.1 Purchasing Section	31
		5.2.6.2 Departmental Safety Representatives	31
		5.2.6.3 Personnel Section	31
		5.2.6.4 Chemical Hygiene Officer	31
	5.2.7	Fire Extinguisher Training	32
		5.2.7.1 Portable Fire Extinguishers	32
	5.2.8	First Aid/CPR Training	32
		5.2.8.1 Mandatory Training	32
		5.2.8.2 29 CFR 1910.151	32
		5.2.8.3 Voluntary	33
6.0	**Medical Consultation and Medical Examinations**		**33**
	6.1	Medical Examinations	33
	6.2	Medical Consultation	33
	6.3	Information Provided to the Physician	33
	6.4	Physician Written Opinion	33
	6.5	Record Keeping	34

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

1.0 INTRODUCTION

1.1 Objective

The objective of this plan is to set forth procedure for equipment handling, personal protective equipment and work practice to protect employees from the health hazards presented by hazardous chemicals used in the laboratory and to meet the requirements set forth in Section E, Federal Register 29 Part 1910 for Occupational Safety and Standards.

1.2 Responsibility

The Chemical Hygiene Plan is designed as a tool to coordinate safety procedures at EMAX. Responsibility for chemical hygiene rests at all levels including the Chief Executive Officer who is ultimately responsible for it. Therefore, it is essential that he/she must provide continuing support for the plan.

The management must designate a Chemical Hygiene Officer who is qualified to by training or experience to provide technical guidance in the development and implementation of the provisions of the Chemical Hygiene Plan. To maintain manufacturers Safety Data Sheet to determine appropriate practice and equipment requirements. To monitor proper functioning and inspection of protective and emergency equipment and arrange for prompt repairs as needed.

Laboratory supervisors must ensure that workers understand and practice the chemical hygiene rules. Protective equipment should be made available and kept in good working order. However, individual laboratory worker must develop good personal hygiene habits and make proper use of the protective equipment. Thus, all employees must cooperate in complying with the provisions of the chemical hygiene plan.

Disciplinary action may be taken when personnel do not adhere to the Chemical Hygiene Plan. The employee will be given an initial verbal warning. Further incidents will warrant a written warning placed in the employee's personnel file. Repeated and habitual offenses will be subject to dismissal.

2.0 CONTROL MEASURES

Professional judgment is used in determining the degree of control measure required for specific contaminants. Annually, a walk through survey should be conducted by an industrial hygienist. Additional air sampling or increased ventilation if required will be conducted. Individual issues addressed at the safety meeting will be evaluated and corrective actions be taken.

2.1 Prior Approval of Hazardous Chemicals

Additional preventive measures may be required to protect employees working with particularly hazardous chemicals such as select carcinogens, reproductive toxins and chemicals producing a high degree of toxicity.

Specific control measures to be taken later will be based on the degree of toxicity, the potential of exposure in a particular procedure and the capacity of the engineering controls administrative practices or protective equipment to control exposures effectively.

One area of potential chemical exposure at EMAX is the organic prep area. Methylene Chloride is the primary chemical solvent used for most of the extraction methods. Exposure level of Methylene Chloride in the organic prep working area is monitored annually. See section 3.8 under Standard Operating Procedure.

2.1.1 Select Cancer Causing Agents

Substances proven to be carcinogenic to humans or demonstrating high carcinogenic potency to animals should be controlled extremely well. Four categories of carcinogens are addressed.

2.1.1.1 Substances regulated as a Carcinogen by OSHA

Chemical regulated as carcinogens by OSHA can be found in 29 CFR part 1910.

2.1.1.2 National Toxicology Program (NTP) Carcinogen List.

Chemicals listed as “known to be carcinogens” in the Annual Report on Carcinogens published by the National Toxicology program (NTP)

2.1.1.3 World Health Organization Carcinogenic Agents

Carcinogenic agent listed under group 1 “carcinogenic to humans” by the International Agency for Research on Cancer Monographs (IARC)

2.1.1.4 State of California Reproductive Toxin Chemical List

Chemicals listed on the State of California Safe Drinking Water and Toxic Enforcement Act of 1986 (Proposition 65) as chemicals known to cause reproductive toxicity as of 1990.

3.0 STANDARD OPERATING PROCEDURES

Standard operating procedures should be familiar to all employees.

When working with hazardous materials, it is essential to know about their physical properties and their potential health effects.

3.1 Chemicals

The term chemicals, as used in this policy, refers to hazardous substances, samples, and hazardous wastes.

3.1.1 General Rule

- Awareness is the most fundamental rule of chemical safety.
- Use common sense when working with or around any area where chemicals are being stored.
- The chemicals hazards are determined from the SDS (Safety Data Sheet) but also are now visibly displayed on the label of that any particular chemical following the globally harmonized system.

3.1.2 Acids and Bases

- General Information
- Corrosive: pH greater than 12 or less than 2.
- Corrosive chemicals will irritate or burn the skin, eyes, and respiratory tract. Severe exposure can cause permanent damage.
- Proper Handling, Use and Storage Procedures
- Add acid or base to water (A to W) never vice versa
- Always pour acid slowly to avoid splashing or superheating.
- Always make sure there is a source of water in the area when working with corrosive chemicals, in case of an emergency.
- Always flush the outside of acid or base containers with water after using.
- After using acids or bases, wipe the surface area you used it on. This will get rid of splatters and spills that may not otherwise be visible.
- All bases or base solutions should be stored in plastic. Do **not** use glass.
- All acids or acid solutions should be stored in glass. Do not use plastic.

- Store acids and bases separately and keep both away from flammables.

3.1.3 Flammable Liquids

Whenever flammable vapors are present there is both fire and an explosion hazard. It is important to understand that it is the vapor, not the liquid that can burn. Flash point is the temperature at which enough vapor is given off to form an ignitable mixture with air. Chemicals with a flash point of less than 100°F are classified as flammable.

You must be aware of the hazards of flammable liquids and you must take positive measures to eliminate the risk of injury. Flammable liquids can be managed safely if they are stored, handled, mixed and poured according to the following safety procedures.

3.1.4 Toxic Materials

A toxic chemical has four (4) routes of entry into the body:

1. Inhalation
2. Ingestion
3. injection
4. Eye or skin contact

You can protect yourselves by using the proper Personal Protective Equipment (PPE).

3.1.5 Personal Protective Equipment

Proper protective equipment shall be provided, used and maintained in a sanitary and reliable condition where there is a chemical hazard that may cause illness or injury to the employees.

- Safety glasses must be worn at all times as prescribed by the procedures of the activity being performed. ANYONE entering a designated laboratory area must wear safety glasses.

- Face shield or splash goggles must be worn when any SIGNIFICANT SPLASH HAZARD exists. (To be determined by supervisors or from past experience with splash-type accidents).

- Contact lenses, which are an additional potential hazard when working in a building where there are samples, acids, fuming agents, solvents, etc., **must not be worn** at any time by employees.

- Lab coats provide a good first level of skin protection against chemical splashes as well as protective clothing, and should be worn during all lab activities. Lab coats are not to be worn in the lunchroom, restrooms or front offices.

- Respirators-Respirators must be worn if there is a potential for exposure to air contaminants in excess of the allowable limits or if the individual is experiencing any symptoms such as headaches or irritation.

- Shoes – A closed-toe shoe is the only footwear allowed in the laboratory area.

- Sample handling-When handling unknown samples, gloves must be worn. (See glove awareness policy section 3.1.5.1)

- Corrosives - Chemical-resistant gloves must be worn. If there is a splash potential, chemical goggles and/or face shield and additional protective clothing should be worn depending on the extent of possible exposure.

- Solvents - Wear chemical-resistant gloves to prevent prolonged or repeated skin contact, such as submerging hands in solvent or handling solvent-soaked rags. If there is a splash potential, chemical goggles and/or face shield and additional protective clothing should be worn, depending on the extent of possible exposure.

3.1.5.1 Glove Awareness Policy

Sample Receiving:

1. Gloves must be worn when coolers are received and placed under the hood to check for broken sample containers.
2. Remove Ziploc bag containing the Chain of Custody from the cooler.
3. Chain of custody must be removed from the bag by someone else without gloves. (COC may be removed also by using a Kim wipe or paper towel).
4. Remove gloves to make a work copy to write all discrepancies or have another person make the copy.
5. Using gloves move cooler (s) inside sample receiving area and remove sample containers from the coolers.
6. Place every container in an orderly fashion to match the label IDs to the chain of custody later.
7. Tag the sample containers. Check tagging, label IDs, and chain of custody information are all matching.
8. Check the pH on all containers and write any discrepancies on work COC.
9. Check for discrepancies on work COC and fill out SRF1 and SRF2. (Preferably another sample receiving personnel should perform this function).
10. Once the SRFs are filled out, finish paperwork in the office area.
11. Using gloves place all sample containers in storage.

Sample Weighing:

1. When weighing samples, samples must be handled with gloves.
2. Write the sequence and/or sample IDs in the run log or prep log ahead of time.
3. When reading sample weight protect the logbook with transparency film specially designed for logging sample weight so that your gloves do not touch the logbook.
4. Use the pencils provided at weighing area attached by a fixed string. Only the tip of the pencil should be touching the logbook. If you are using your own pen, handle the pen without gloves.

Sample Preparation:

1. Handle logbooks without gloves. Write the sequence and/or sample Ids on logbook ahead of time; otherwise protect the logbook from touching the gloves.
2. Wear gloves to remove samples from the storage area.
3. Wear gloves when weighing soil samples. (Follow procedure for sample weighing above).
4. If gloves appear dirty or contaminated, discard and use new gloves. Use a Kim wipe or paper towels to open doors and proceed to the prep area.
5. Continue wearing gloves to finish the extraction process. If methylene chloride is to be used wear appropriate gloves for the procedure.
6. Remove gloves when performing administrative tasks or have another technician write in the logbook. (i.e. spiking).

Sample Analysis:

1. Use gloves when handling samples, extracts or preparing standards.
2. Write the run log sequence and/or sample Ids on logbook ahead of time; otherwise protect the logbook from touching the gloves.
3. Remove gloves when performing any administrative tasks. (Writing in logbooks or using the computer keyboard).

Sample disposal:

1. Wear gloves when handling satellite waste or sample disposal.
2. Put satellite waste on a cart and move the waste to the 90 day storage area.
3. Use kimwipe or paper towels to open doors.

4. If during the waste disposal process, gloves become dirty and/or contaminated discard them and use a new pair of gloves.
5. Dispose satellite waste or samples in appropriate 55 gallon drums.
6. Before leaving the 90 day storage area, remove gloves. Log in the waste in the logbook.
7. Put new gloves on and lock the 90 day storage area.
8. Return empty satellite containers to the lab. Use kim wipes or paper towels to open doors.
9. Discard gloves and wash your hands.

Note: If gloves appear to be dirty or contaminated, replace them to keep the level of contamination low. Do not touch door with gloves. Remove gloves or use a paper towel to open doors. Dispose paper towel in waste bin.

3.2. COMPRESSED GAS CYLINDERS

3.2.1 General

- Be extremely careful when handling compressed gas cylinders. Do not drop or expose them to extreme temperatures.
- Except when in use, the valve cap or valve protection device must be always in place.
- Be sure all cylinder valves are closed before moving them, when work is finished, and when the cylinder is empty.
- Never use the valve or valve cap to lift or move cylinders. Always use a hand truck for support. Chain cylinder to the hand truck. Never roll cylinders.
- Improperly fitting connections on cylinders should never be forced. Never tamper with safety devices of cylinder valves. Always use proper fitting on gauges suitable for the particular gas being used.
- Contents must be properly marked on all cylinders. Color coding is not a standardized means for identification.
- Never mix gases in a cylinder. Explosion, contamination, corrosion, and other hazards can result.
- Cylinder valves must not be tampered with nor should any attempt be made to repair them. If you experience trouble, the supplier should be notified promptly, indicating the type of trouble and the cylinder's serial number.

3.2.2 Inspection

- Cylinder should be checked periodically for rust, dents, or leaks, particularly around the neck of the cylinder (including the screw threads) and at the bottom.
- When there is doubt about a cylinder's structural strength or the possibility of leakage notify the supplier immediately.
- Safety valves, gauges, and regulators should be securely mounted and may not be used if they are bent or damaged by exposure to weather or by mishandling.
- The Department of Transportation requires that all cylinders be tested for strength and integrity on a periodic basis. Last testing date is stamped on each cylinder along with symbols indicating date cylinder is due to be re-tested. If you have any questions, contact the Safety Officer or Supplier.

3.2.3 Storage/Transport/Use

- Always store and use compressed gas cylinders (whether full or empty) in an upright position. Chain or otherwise secure them so they cannot be upset, fall or strike each other.
- Storage temperature should be regulated, so as not to exceed 100°F.
- All valves should be closed when not in actual use.
- Use proper regulator for the particular gas.
- Carefully and slowly open valves and adjust gas flow rates.
- Always consider cylinders to be full and handle accordingly.
- All empty cylinders must be marked "EMPTY". They must be separated from the full cylinders and promptly returned to the storage area with the valve protection caps in place. All valves must be closed.
- Discontinue using a high-pressure cylinder when the pressure reaches 500 psig, and clearly marked EMPTY; then remove for return to vendor.
- Cylinders must never be placed where they might become part of an electrical circuit.
- Always make sure to protect cylinders from sparks, flames, and contact with energized electrical equipment.
- Always store flammable gas cylinders in properly designated and safeguarded areas only.

3.3 Specific Gases

3.3.1 Argon

- Argon is the most abundant member of the rare gas family, which consists of helium, neon, argon, krypton, and xenon. All of these gases are monatomic and are characterized by their extreme chemical inactivity. Argon is colorless, odorless, and tasteless gas somewhat soluble in water (4 volumes in 100). It is normally supplied as a non-liquified gas compressed into cylinders at a pressure of approximately 200 p.s.i.g at 70ºF.

- Argon is nontoxic but can act as a simple asphyxiant by displacing the necessary amount of air to support life.

- Leaks of argon in lines and equipment setups may be detected by painting the suspected sites with water, leaks will be evident by bubble formation.

- Since argon is inert, no special materials of construction are required. However, any piping or vessels containing argon should be adequately designed to have a working pressure as specified by competent engineers, using a safety factor conforming to the A.S.M.E. (American Society of Mechanical engineers) code for pressure piping.

- Cylinders containing argon have safety devices of either the frangible disc type or frangible disc backed up with fusible metal melting at approximately 212 ° F. Cylinders pressurized 10% in excess of their marked service pressure in accordance with present D.O.T. (Department of Transportation) regulations must contain only safety devices are usually frangible disc type. These safety devices are usually an integral part of the cylinder valve, situated opposite the valve outlet.

- Argon is shipped in high pressure steel cylinders as non-flammable, compressed gas, taking a D.O.T. "Green Label". They are usually filled to the marked service pressure of the cylinder or to a maximum of 10% excess of the marked service pressure in accordance with present D.O.T. regulations.

- Argon is monatomic, chemically inactive gas. It will not react with other elements or compounds. While a few compounds of argon and other rare gases are reported to have been prepared, such researchers, and the results obtained, may be considered of scientific interest only. For all practical purposes every attempt to make argon combine to give compounds of the usual types, like treatment with oxidizing or reducing agents, has failed. The comparative stability of argon is due to the complete pairing of all electrons present and the absence of any bonding orbitants.

3.3.2 Liquid Argon

Liquid argon is tasteless, colorless, odorless, non-corrosive, nonflammable, and extremely cold. Belonging to the family of rare inert gases, argon is the most plentiful of the rare gases, making up approximately 1% of the earth's atmosphere.

It is monatomic and extremely inert, forming no known chemical compounds. Special materials of construction are not required to prevent corrosion. However, materials of construction must be selected to withstand the low temperature of liquid argon. Vessels and piping should be designed to American Society of Mechanical Engineers (ASME) specifications or the Department of Transportation (DOT) codes for the pressures and temperatures involved.

Although used more commonly in the gaseous state, argon is commonly stored and transported as a liquid, affording a more cost-effective way of providing product supply. When argon is converted to liquid form it becomes a cryogenic liquid. Cryogenic liquids are liquefied gases that have a normal boiling point below -238°F (-150°C). Liquid argon has a boiling point of -302.6°F (-185.9°C). The temperature difference between the product and the surrounding environment, even in winter, is substantial.

Keeping this surrounding heat from the product requires special equipment to store and handle cryogenic liquids. A typical system consists of the following components: a cryogenic storage tank, one or more vaporizers, a pressure control system, and all of the piping required for fill, vaporization, and supply. The cryogenic tank is constructed like a vacuum bottle. It is designed to keep heat away from the liquid that is contained in the inner vessel. Vaporizers convert the liquid argon to its gaseous state. A pressure control manifold controls the pressure at which the gas is fed to the process.

3.3.3 Helium

Helium is inert, colorless, and tasteless gas and is only slightly soluble in water (0.86 parts in 100 parts). One of the most important characteristics of helium is its extremely low density. Helium is normally supplied as a non-liquified gas compressed into cylinders at a pressure of approximately 2200 p.s.i.g. at 70°F.

A mixture of helium and oxygen finds use as a breathing gas for divers who must who must work under high pressures. Atmospheres containing helium are used for therapy and an anesthesia of patients suffering from asthma or other obstructive conditions in the respiratory passages.

Helium is nontoxic but can act as an asphyxiant by displacing the necessary amount to air to support life.

Safety Devices: Cylinders containing helium have safety

devices of the unbacked frangible disc type or frangible disc backed up with fusible metals, melting at approximately 212 °F. Cylinders pressurized 10% in excess of their marked service pressure in accordance with present D.O.T. regulations must contain only safety devices of the unbacked frangible disc type. These safety devices are an integral part of the cylinder valve, situated opposite the valve outlet.

Shipping Regulations: Helium is shipped in high pressure steel cylinders as a nonflammable compressed gas, taking a D.O.T. "Green Label". They are usually filled to the maximum of 10% in excess of the marked service pressure of the cylinder in accordance with present D.O.T. regulations.

Chemical Properties: Helium is a monatomic chemically inactive gas. It will not react with other elements or compounds under ordinary conditions.

3.3.4 Nitrogen

Nitrogen comprises approximately 79% by volume of the air. It is found chemically combined in many forms in nature. Nitrogen will not burn and will not support combustion. Nitrogen is normally available in cylinders compressed to 200 p.s.i.g.

Nitrogen is non-toxic, but can act as an asphyxiant by displacing the necessary amount of air to sustain life.

Leaks in lines and equipment may be detected by painting the suspected sites with soapy water. Leaks will be evident by the formation of bubbles.

Since nitrogen is inert, no special materials of construction are required. However, any piping or vessels containing nitrogen should be designed to have a working pressure as specified by competent engineers, using a safety factor in compliance with the A.S.M.E. code for pressure piping.

Nitrogen is packed in D.O.T. approved, high-pressure steel cylinders.

Cylinders containing nitrogen have safety devices of either the frangible disc type or frangible disc type backed with fusible metal, melting at approximately 212°F. Cylinders pressurized 10% in excess of their marked service pressure in accordance with present D.O.T. regulations must be equipped only with safety devices.

Shipping Regulations: Nitrogen is shipped in high-pressure steel cylinders as a non-flammable compressed gas, taking a D.O.T. "Green Label". The cylinders are usually filled to the marked service pressure of the cylinder or to the maximum of 10% in excess of the marked service pressure, in accordance with present D.O.T. regulations.

Nitrogen is extremely inert, except when heated to very high temperatures where it combines with metal to form nitrides. Nitrogen

combines with oxygen in an electric arc or gas fired furnace to give an unfavorable equilibrium mixture containing about 2.23% (by volume) nitric oxide. Nitrogen combines with hydrogen at elevated temperatures and pressures in the presence of catalysts to form ammonia. Nitrogen is an essential constituents of all living plant and animal matter, but cannot be utilized by the animal organism to form amino acids. This vital function is carded out only by plants.

3.3.5 Liquid Nitrogen

Liquid nitrogen is a colorless, odorless, extremely cold liquid and gas under pressure. It can cause rapid suffocation when concentrations are sufficient to reduce oxygen levels below 19.5%. Self-Contained Breathing Apparatus (SCBA) may be required.

Contact with liquid or cold vapors can cause severe frostbite. Cold vapors in the air will appear as a white fog due to condensation of moisture. While this may indicate the presence of the gas it should not be used to determine its concentration in the atmosphere. Oxygen concentrations must be monitored in the release area. All cryogenic liquids produce large volumes of gas when they vaporize. One volume of liquid nitrogen will expand to produce 696.5 equivalent volumes of gas.

3.3.6 Hydrogen

Hydrogen is a colorless gas with no odor. It is not toxic; the immediate health hazard is that it may cause thermal burns. It is flammable and may form mixtures with air that are flammable or explosive. Hydrogen may react violently if combined with oxidizers, such as air, oxygen, and halogens. Hydrogen is an asphyxiant and may displace oxygen in a workplace atmosphere. The concentrations at which flammable or explosive mixtures form are much lower than the concentration at which asphyxiation risk is significant.

Hydrogen is not toxic by any route. Asphyxia may result if the oxygen concentration is reduced to below 18% by displacement.

The only safe way to extinguish a flammable gas fire is to stop the flow of gas. If the flow cannot be stopped, allow the entire contents of the cylinder to burn. Cool the cylinder and surroundings with water from a suitable distance. Extinguishing the fire without stopping the flow of gas may permit the formation of ignitable or explosive mixtures with air. These mixtures may propagate to a source of ignition.

Excessive pressure may develop in gas cylinders exposed to fire, which may result in explosion, regardless of the cylinder's content. Cylinders with pressure relief devices (PRD's) may release their contents through such devices if the cylinder is exposed to fire. Cylinders without PRD's have no provision for controlled release and are therefore more likely to explode if exposed to fire.

Protect the cylinders from direct sunlight, precipitation, mechanical damage, and temperatures above 55°C (130°F).

Ship and store cylinders with the outlet plug and valve protective cap in place.

3.4 Incompatible Materials in the Laboratory

DO NOT MIX:

Alkali Metals: Calcium, potassium and sodium with water, carbon dioxide, carbon tetrachloride and other chlorinated hydrocarbons.

Acetylene: With copper tubing, fluorine, bromine, chlorine, iodine, silver, mercury or their compounds.

Ammonia, Anhydrous: With mercury, halogens, calcium hypochlorite or hydrogen fluoride.

Ammonia Nitrate: With acids, metal powders, flammable liquids, chlorates, nitrates, sulfur and finely divided organics or other combustibles.

Chromic Acid: With acetic acid, napthalenes, camphor, alcohol glycerine, turpentine and other flammable liquids.

Chlorine: With ammonia, acetylene, butadiene, benzene and other petroleum fractions, hydrogen, sodium carbide, turpentine and finely-divided powdered metals.

Concentrated Acids and Bases and Their Salts: Will produce large amounts of heat when mixed together with water.

Cyanides: With acids.

Hydrogen Peroxide: With copper, chromium, iron, most metals or their respective salts, flammable liquids, and other combustible materials, aniline and nitro-methane.

Hydrogen Sulfide: With nitric acid, oxidizing gases.

Hydrocarbons: With fluorine, chlorine, bromine, chromic acid or sodium peroxide.

Iodine: With acetylene or ammonia.

Mercury: With acetylene, fulmic acid, hydrogen.

Nitric Acid: With acetic, chromic and hydrocyanic acids, aniline, carbon, hydrogen sulfide, flammable liquids or gases and substances which are readily nitrate.

Perchloric Acid: With acetic anhydride, bismuth and its alloys, and organic material.

Potassium Permanganate: With glycerine, ethylene glycol and sulfuric acid.

Sulfuric Acid: With chlorates, perchlorate, permanganates; and water.

Hydrofluoric Acid: Keep in tightly closed polyethylene containers. Store in a cool, dry place with adequate ventilation separated from other chemicals. Protect from physical damage. Storage facilities should be constructed for containment and neutralization of spills. Handling and storage of HF requires special materials and technology for containers, pipes, valves, etc., which is available from suppliers. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

3.5 Chemical Spills

Alert your neighbors and the supervisor!

For **small** spills only, if there is no fire hazard and the materials are not dangerous, clean up as directed by your supervisor. For **large** spills of liquids, an absorbent material will aid in the clean-up operation. Substances such as Haz-sorb pillows and Zorb-A are good choices. Neutralize acid spills with sodium bicarbonate. Use a dustpan and brush to remove all items used to neutralize and contain the spill. Gloves should be worn. If the spill is on the floor, be sure the area of the spill is dry before leaving-this will prevent slippage by others.

If volatile, flammable or a toxic material is spilled, clearly warn everyone to turn off burners and spark producing equipment. Shut down all experimentation and leave the area. Your supervisor will direct the clean-up effort.

Spilled mercury should be immediately and painstakingly cleaned up using a trapped vacuum line attached to a tapered glass tube similar to a medicine dropper. Cover small droplets in inaccessible spots with sulfur to convert the mercury to mercury sulfide. Droplets of mercury present a large surface area and may generate dangerous concentrations of mercury vapor in the air.

Organic Halogen and Related Compounds (Methylene Chloride, Freon)
For spills less than 1 liter, clean-up should be under the direction of the Organic Lab Manager. For spills larger than 1 liter, the immediate area should be evacuated until the Laboratory Director and/or Chemical Hygiene Officer permits employees to re-enter the area.

Gas Leak: If valve is leaking because it cannot be turned off, the gas can be bubbled through a reducer and excess sodium bicarbonate solution.

Liquid or Solid: Cover with a reducer (a bisulfite will work well). Mix well and spray with water. A sulfite will require an addition of some SM - H_2 - $S04$ to promote rapid reduction. Scoop the slurry into the container and neutralize with soda ash. Wash down the drain with large amounts of water.

IF THE SPILL IS LARGE, LEAVE THE AREA AND GET PROFESSIONAL HELP!

3.6 Disposal Procedures for Chemical Wastes

No Drain Dispose: No samples or chemicals are allowed to be drain disposed.

Organic Solvents: All organic solvents including methylene chloride and Freon are to be placed in 55 gallon waste drums for organic solvent waste. Water and acidified water samples resulting from extractions **should not** be added to organic waste drums under any circumstances.

Reducing Substances: (Sulfur Dioxide) Wear rubber gloves, safety goggles, lab coat.

Heavy Metals (Lead, Mercury, Cadmium, and their compounds): Wear rubber gloves, lab coat, and in the event of a large mercury spill, use a self-contained breathing apparatus.

Recovery: Every effort should be made to recover these chemicals before they can contaminate air or water. Clean up procedures will vary depending on the type and amount of chemical involved. Contain the spill and consult your supervisor for further instructions.

These above four examples are general guidelines to help you safely deal with chemical wastes. **See your supervisor if you have any questions on proper disposal.**

3.7 Potentially Explosive Chemicals (including organic peroxide)

Certain chemicals that are known to be unstable can constitute a serious safety hazard and should be treated accordingly. These materials may deteriorate and become more susceptible from friction, from mechanical impact, from thermal shock or from spontaneous combustion. The materials can be used safely if personnel are familiar with and carefully follow instructions for use, storage and disposal.

A wide variety of chemicals fit this category. Compounds containing "phospheres" contribute to the tendency for a compound to explode. The following is a list of several common phosphores:

PHOSPHORES	EXAMPLE
Nitro	Nitromethane, Trinitrophenol (picric acid) and Trinitrotoluene (TNT)
Nitramine	Etylene nitramine
Diazo	Diazodinitrophenol
Azide	Lead azide
Peroxide	Benzoyl peroxide, diethyl ether, and tetrahydrofuran

Diethyl ether may fall into the peroxide category because the influence of air (oxygen) and light promotes oxidation to form organic peroxide. Because this

oxidation proceeds at a variable rate, depending on many factors, containers of diethyl ether must be dated when received and must be disposed of after a period of six (6) months. This chemical should be purchased in small quantities that can be consumed within six (6) months. Similar limitations apply to tetrhydrofuran and other "peroxide formers".

Ethers with high concentrations of peroxides are extremely sensitive to impact and physical shock and are capable of violent explosion. Ethers with unknown concentrations of peroxides should be handled as if they were booby-trapped. No on should "shake" a container to see how much is in it. Shock-sensitive compounds such as old ethers or old picric acid should be handled with great respect for their explosive power.

When chemicals in the above categories require a disposal, a phone request for disposal hygiene should be made directly to one of the following: (1) Chemical Hygiene Officer, (2) the Safety Officer, and (3) the Fire Marshal. Removal of the problem material will be by the members of the Laboratories' Hazardous Material Emergency Response Team or by the trained outside group of people.

If the material in question has been stored beyond its recommended safe shelf life, appropriate warning signs should be posted emphasizing that the material is not to be touched or moved. The area should be isolated while arrangements are made for proper and safe disposal. The contents may be shock sensitive and any handling by untrained professional could result in a serious accident.

3.8 Methylene Chloride Exposure Monitoring.

3.8.1 Policy.

A yearly exposure monitoring shall be performed in the sample preparation area where (CH_2Cl_2) Methylene Chloride (MC) is used. As per OSHA PEL (Permissible Exposure Limit), exposure shall not exceed 25 parts MC per million parts of air (25 ppmv) as an eight hour time-weighted average (8-hour TWA PEL) or 125 parts of MC per million parts of air (125 ppmv) averaged over a 15-minute period (STEL).

It is the responsibility of the Safety Officer/Chemical Hygiene Officer to conduct the exposure monitoring at various locations of the sample preparation area.

It is the responsibility of the Safety/Chemical Hygiene Officer to stop the operation when a critical condition is observed or found. Suspension of operation shall only be lifted upon successful resolution of the deficiencies.

It is the responsibility of the operation manager to oversee the implementation and /or enforcement of corrective action and/or recommendation for improvement.

3.8.2 Safety

Dichloromethane is an OSHA “Select Carcinogen” and is a skin and eye irritant. Exposure to high concentration of dichloromethane vapor (>500

ppm for 8h) can lead to lightheadedness, fatigue, weakness, and nausea. Contact of the compound with the eyes causes painful irritation and can lead to conjunctivitis and corneal injury if not promptly removed by washing. Dichloromethane is a mild skin irritant, and upon prolonged contact (e.g., under the cover of clothing or shoes) can cause burns after 30 to 60 min exposure. Prolonged exposure to vapors may cause cancer or exacerbate cardiac disease. Dichloromethane is not teratogenic at levels up to 4500 ppm or embryotoxic in rats and mice at levels up to 1250 ppm.

Most people cannot smell methylene chloride until it reaches a hazardous level – so don't depend on your sense of smell to warn you of overexposure. If you can smell it, your exposure is too high.

3.8.3 Instrument and Supplies

Escort Elf Pump	MSA Escort Elf Pump – P/N 497701 – S/N A2-30987
Gemini Twin Port Sampler	MSA Gemini Twin Port Sampler - 497697
Charcoal Tubes	Supelco ORBO-32 Large Charcoal Tubes 400/200mg - 20228
Battery Charger	MSA Omega Battery Charger P/N 494716 Rev.5, Rapid Charge; Trickle Charge

3.8.3 Procedure

3.8.3.1 Perform Operations Check as per operation manual.

3.8.3.2 Preset the Escort Elf Pump at 1.5 LPM for proper airflow.

3.8.3.3 Set Gemini Twin Port Sampler at 200 ml/min for each charcoal tubes. See operation manual for Twin Port Sampler on page 1-7.

3.8.3.4 Break the end of the charcoal tubes carefully and place them with the arrows going down into the Twin Port Sampler.

3.8.3.5 Place the Pump and Twin Port Sampler at the desired location and start the pump. Exposure monitoring shall be performed for 8 hour. After approximately 8 hour, stop the pump. The pump will display time in minutes of its operation.

3.8.3.6 Record the time on a chain of custody with the 200 ml/min flow rate. Cap both tubes carefully. Give the tubes and the chain of custody to sample receiving for login and analysis of the Charcoal Tubes.

3.8.4 Quality Control

The permissible exposure limit is 25 part per million part of air. In the event that the PEL is exceeded, the chemical Hygiene officer and management will investigate the cause of the high exposure. The engineering and administrative control will be checked and re-evaluated. Any corrective actions will be discussed with personnel and documented.

3.8.5 Supplementary notes:

3.8.5.1 Definition of Terms:

Critical Condition: Any results above the OSHA PEL.

4.0 PROTECTIVE EQUIPMENT EVALUATION

The evaluation of protective equipment is addressed in the following:

4.1 Procedures for Checking Safety Showers/Eye Wash

4.1.1 Safety Showers

Step 1 Find a 5-gallon bucket.

Step 2 Place bucket and funnel device under safety shower.

Step 3 Pull chain or push rod on safety shower.

Step 4 Let shower run for approximately 30 seconds or until the bucket is full. The bucket should have 1 gallon or greater of water after 30 seconds flush.

Step 5 Release chain or rod on safety shower.

Step 6 Empty bucket.

Step 7 Continue flushing until water is clean, then return the bucket to proper place. The water temperature should be in a comfortable range (60-75°F).

Step 8 Wipe up any water spilled on the floor.

4.1.2 Eye Wash

Step 1 Start flow of water.

Step 2 Continue flushing until water is clean.

Step 3 Observe flow to make sure spray is even and meets in center.

Step 4 Water temperature should be in a comfortable range (60-75°F).

The Frequency of checking safety showers/eye wash stations is monthly.

4.2 Instruction for Replacing Portable Bottled Emergency Eye Washes

Portable bottled eye washes are used only when a plumbed system is not possible.

Step 1 Remove expired emergency eye wash containers from holders in each area.

Step 2 Replace with new purchased bottled eye washes.

4.3 Local Exhaust Systems.

Ventilation systems such as fume hoods, canopy hoods and other local exhaust system have many uses. These ventilation systems are installed to control exposure to airborne contaminants or to control other internal conditions. To ensure that the ventilation systems are operating properly, they need to be evaluated through the measurement of airflow patterns, velocities and volume. All ventilation systems installed to control exposures to airborne contaminants should have baseline and annual evaluations. These evaluations will be performed in accordance with the following procedures:

4.3.1 Baseline Evaluation

4.3.1.1 Airflow Measurements of Hoods

A **baseline** measurement of airflow patterns, velocities and volume will be made with a Velometer or other similar instrument. Bi-weekly measurements of the airflow patterns, velocities and volumes will be made utilizing the same instrument used during the baseline evaluation. The new measurements will be documented on a Hood Flow Chart. Procedure for the Fume Hood Inspection can be found in EMAX-QC09 SOP.

4.4. Respiratory Protection Program

4.4.1 Policy Statement

The Respiratory Protection Program was established in order to coordinate the use and maintenance of all respiratory protective equipment.

The use of respiratory protective equipment is only necessary under certain conditions in order to: (1) reduce the potential for employee exposure to harmful levels of toxic chemicals and (2) allow employees to enter and work safely in hazardous work environments, such as confined space entry or hazardous waste sites.

The requirements set forth in this policy ensure compliance with the OSHA Respiratory Protection Standard 29 CFR 1910.134.

Evaluation has shown no need for respirators for routing 126 procedures. Engineering controls provide proper chemical control.

4.4.2 Responsibilities

4.4.2.1 Chemical Hygiene Officer

- Evaluate operations to determine if respiratory protection is required.

- Ensure proper respirator medical exam is provided.
- Supervise the respirator selection process. Ensure the purchase of NIOSH-approved respirators and replacement parts.
- Conduct respirator training and fit testing.
- Evaluate effectiveness of the program.
- Maintain employee respirator records

4.4.2.2 Divisional/Departmental Safety Representatives

- Ensure that employees wear respirators as required.
- Issue respirators.
- Inspect emergency use respirators monthly.
- Maintain records of emergency use respirator inspections.

4.4.2.3 Employees

- Use the respirators assigned to him/her in accordance with instructions and training.
- Clean, disinfect, and inspect his/her respirator.
- Report respirator malfunctions to Divisional/Departmental Safety Representatives.

4.4.3 Respirator Selection

Any operation which exposes employees to harmful dusts, fogs, fumes, mists, gases, smokes, sprays or vapors will be evaluated by the Chemical Hygiene Officer. If respirators are required, the Chemical Hygiene Officer will select a NIOSH-approved respirator based on the hazard(s) to which the employee is exposed. Essential elements to be considered in making the respirator selection include:
The Air Purifying Respirator or Chemical Cartridge Respirator is used at EMAX. A combination of organic vapor and acid fume cartridges are used during activities where the employees may be most exposed without the used of the lab engineering controls. (Waste disposal).

4.4.4 Training and Education

Every employee who may have to wear a respirator must be instructed in the proper selection, use and maintenance of the respirator. Both the employee and the supervisor will receive this training. The training will be repeated at least annually.

Procedure:

- Inspect the respirator before each use for cleanliness, proper assembly and absence of any damage.

- Position narrow portion of respirator on your nose bridge and place cradle suspension system on your head so that the top headstrap rests across the top of your head.

- Hook the bottom headband behind your neck and position the facepiece for comfort.

- All the headbands are adjustable. Hold respirator in one hand and pull the straps toward the rear to tighten. To loosen, pull forward on the plastic yoke through which the strap is threaded.

- Check the seal of your respirator by conducting a positive and negative pressure seal check. A seal check should be conducted every time the facepiece is put on and periodically while wearing it.

- To conduct a positive pressure seal check, cover the opening in the exhalation valve guard with the palm of your hand and exhale gently. If an air leak is detected, reposition the facepiece and/or readjust the headband straps. Repeat the check until an effective seal is obtained.

- To conduct a negative pressure seal check, place the palms of your hands over the openings in the filter covers. Inhale enough to collapse the facepiece slightly and hold your breath for five seconds. If the face piece does not remain collapsed, reposition the facepiece and/or readjust the headband straps. Repeat the check until an effective seal is obtained.

- Select a testing location having exhaust ventilation sufficient to avoid general contamination of the area by the irritant smoke, which is used to test the effectiveness of the respirator.

- The employee will be advised that the smoke can be irritating to the eyes and instruct the subject to keep his/her eyes closed while the test is performed. Proceed with the fit test using the instructions provided in the fit test kit using irritant smoke.

- After the use of the respirator, the respirator must be cleaned with ar alcohol wipe pad and placed in the seal plastic bag provided.

4.5 Program Evaluation

The Chemical Hygiene Officer must evaluate the effectiveness of the respiratory protection program. This on-going evaluation process should include:

- Periodic review of feasible engineering, administrative, or work practice control measures that could be initiated to eliminate or reduce employee exposure to air contaminants.

- Random inspections to assure that employees have been trained and are wearing the correct respirator, and that the respirators are properly maintained and stored.

- Periodic checks to assure that the record keeping requirements are being fulfilled. Record and date the above inspections/evaluations.

4.6 Respiratory Protective Equipment: Use and Limitations

4.6.1 Air Purifying Respirators

4.6.2 Chemical Cartridges Respirators Limitations

DO NOT USE chemical cartridge respirator's for the following materials:

1. Acroline
2. Aniline
3. Arsine
4. Bromine
5. Carbon monoxide
6. Dimethylaniline
7. Dimethyl sulfate
8. Hydrogen cyanide
9. Hydrogen fluoride
10. Hydrogen selenide
11. Hydrogen sulfide
12. Methanol
13. Methyl bromide
14. Methyl chloride
15. Methyl biphenyl isocyanate
16. Nickel carbonyl
17. Nitro compounds
    - Nitrobenzene
    - Nitrogen oxides
    - Nitroglycerine
    - Nitromethane
18. Ozone
19. Phosgene
20. Phosphine
21. Phosphorous trichloride
22. Stibine
23. Sulfur chloride
24. Toluene diisocyanate
25. Vinyl chloride

Note: The above list is far from complete and is offered only as a guide to proper evaluation of the many contaminants found.

4.7 Respirator Physical Evaluation Form

NAME: _____AGE: _____BIRTHDATE:

HEIGHT: _____WEIGHT:

Do you have or have you ever had any of the following?

	Yes	No
a. Diabetes	_____	_____
b. Seizures or convulsions	_____	_____
c. Alcohol dependency or abuse	_____	_____
d. Punctured ear drums	_____	_____
e. Skin disorders (dermatitis/allergies)	_____	_____
f. Trouble smelling	_____	_____
g. Lung problem such as asthma, emphysema, TB, x-ray evidence of pneumoconiosis, reduced lung function, etc.	_____	_____
h. Heart disease	_____	_____
i. Stroke	_____	_____
j. High blood pressure	_____	_____
k. Anemia (low blood/low hemoglobin)	_____	_____
l. Surgery to head/face-other than dental	_____	_____
m. Had trouble breathing when wearing a respirator	_____	_____
n. Claustrophobia when wearing a respirator	_____	_____
o. Any medical condition, which you feel could be a problem when wearing a respirator.	_____	_____
p. Significant weight gain (since last filling out this form)	_____	_____

If you answer yes to any of the above, please explain your answer on the reverse side of this sheet. Include any treatment and results.

List on the reverse side of this sheet any prescription medications you take on a regular basis.

The above is, to the best of my knowledge, a truthful statement concerning my present health.

_____ _____

Signature Date

4.8 Medical Summary Form

Name: _____ Social Security No.:_____

Date of Physical Exam: _____

MEDICAL SUMMARY

	COMMENTS	**NORMAL**	
		YES	NO
A	Laboratory Tests		
	1. Pulmonary Function Tests:	_____	_____
	2. Chest X-Ray:	_____	_____
	3. Vision Screening:	_____	_____
	4. Hematology Survey:	_____	_____
	5. Urinalysis:	_____	_____
	6. Heavy Metal Screen:	_____	_____
	7. Blood Chemistry Screen:	_____	_____
	8. Audiogram:	_____	_____

B. Examiners Summary of Ability to Wear a Respirator
Self-Contained Breathing Apparatus and Negative Pressure Respirators

C. Examiners Summary of Physical/Medical Summary

D. Any Additional Comments/Recommendations

_____M.D. _____
Signature Date

I understand that this form will be released to EMAX Laboratories, Incorporated and I will receive a copy. I also understand that EMAX Laboratories, Incorporated retains the right to access my medical record as needed.

_____ _____
Signature Date

5.0 Employee Information and Training

5.1 Employee Information

The Occupational Safety and Health permissible exposure limits (PEL) are available from the Chemical Hygiene Officer. Signs and symptoms associated with overexposure to hazardous chemicals used in the laboratory are discussed on the Safety Data Sheets, as well as product labels and known reference material. Additional reference material is available by contacting the Corporate Safety Officer. Safety Data Sheets are available upon request and a copy will be provided upon request.

5.2 Employee Training

5.2.1 Training Statement

EMAX Laboratories, Incorporated is firmly committed in providing each of its employees a safe and healthy work environment. It is important that employees in these situations are aware of the identity and/or toxic or other hazardous properties of the chemical, since an informed employee is more likely to be a careful employee.

The success of our training depends to a great extent upon the cooperation of every employee. Employees should be alert to the potential hazards of all the chemicals in the work area, consult the Safety Data Sheet for specifics concerning the hazardous chemicals with which they work, follow the appropriate work practices that have been established to protect their health and safety.
Active employee participation in our training program will result in the continued prevention of chemical-related illnesses and injuries.

5.2.2 Safety Data Sheets

A "chemical inventory" will be maintained by the Chemical Hygiene Officer. This inventory will list each hazardous substances used. The inventory will be updated when new substances are procured.

A safety data sheet (SDS) containing the information required by the Federal Hazard Communication Standard will be kept for each substance listed on the "chemical inventory". The SDS will be the most current one supplied by the chemical manufacturer, importer or distributor.

The SDS's will be filed in the Safety Data Sheet Notebook, alphabetically by the name identified in the chemical inventory. The original SDS's will be filed in a Master SDS notebook.

No hazardous material will be used in the workplace unless an SDS has been obtained and is on file. For new chemicals, an SDS should be requested at the time of purchase.

A new health information becomes available about any hazardous material on the inventory, a revised Safety Data Sheet will be placed in the SDS file. Each Divisional/Departmental Safety Representatives will review revised SDS's for their division/department. The employee who handles or might be exposed to the material will be notified if there are any changes in work procedures or personal protecti equipment requirements.

Safety Data Sheets will be made readily available to all employees on any work shift, as well as to representatives of State or the Federal Occupational Safety and Health and to the employee's physician. The SDS Library is maintained in the office of the Health and Safety Officer.

5.2.3 Labeling Policy

Employees will accept no hazardous chemicals for use unless labeled with at least the following information:

Identity of the hazardous chemical (trade or chemical name)
Appropriate hazard warnings
Names and address of the chemical manufacturer

If the hazardous chemical is regulated by OSHA in a substance specific health standard, the label used will be in accordance with the requirements of that standard (see Section 6.1 for list).

In certain situations involving stationary process containers, the label may be replaced by a sign or a SDS.

Any portable container of hazardous chemicals will be labeled with the appropriate information from the shipping label. Employees with questions concerning the appropriate information to use on a label, should check with their divisional/departmental Safety Representatives.

Bottles of reagents will be labeled as follows:

Reagent name
Test name
Preparation Date and Initials
Expiration Date

The label information obtained from "Standard Methods" when the reagents are being prepared. The reagent must also be entered into the "Reagent Logbook".

The identity of the material that appears on the manufacturer's label or the in-house label will be the same name used to identify the material on the "chemical inventory" and the SDS for that substance.

All labels will be legible and prominently displayed on the container.

5.2.4 Hazard Determination

EMAX Laboratories, Incorporated will rely on the chemical manufacturer's hazard evaluation for all chemicals purchased.

The Chemical Hygiene Officer will review SDS's on new products to determine appropriate work practices and protective equipment requirements. (See section 3 for further criteria).

5.2.5 Employee Training

All employees who may be routinely exposed to hazardous substances or harmful physical agents will be provided with training.

Training must be provided to an employee prior to initial assignment to a workplace where the employee may be routinely exposed to a hazardous substance or harmful physical agent.

Divisional/Departmental Safety Representative will be responsible for coordinating new employee training sessions with the Personnel Section.

If a new chemical is introduced into a work area, each affected employee will be appropriately trained prior to using the new chemical. This training will be in the form of a SDS review at Departmental meetings.

Records must be kept of each training session. The record must include the date of the session, the type of session and an attendance list. These records will be retained for five years and made readily available, upon request, for review by employees and representatives of State or Federal Occupational Safety and Health Administrations.

Before any non-routine task is performed that could involve exposure to hazardous chemicals the employee's supervisor will carefully review all potential hazards of the task with the work area(s) that the contractor's employees will be working in. The SDS for the relevant chemicals will be available to the contractor upon request.

As part of the contractual arrangement between any outside contractor and the contractor must list all hazardous chemicals that are to be used by his employees in the course of their work

on company property, so that employees may receive the necessary information and training concerning the potential hazards of the substances to which they may be exposed.

Furthermore, prior to beginning work on Company property, the contractor must certify that his/her employees have been trained. As necessary, the CHO will identify particular hazards associated with the work area(s) that the contractors employees will be working in. The SDS for the relevant chemicals will be available to the contractor upon request.

5.2.6 Execution/Assigned Responsibilities

The execution and assigned responsibilities for the Chemical Hygiene Plan shall be as described in this section. Each department will be responsible for assigning specific individuals to ensure that the program is executed as required. The individuals or sections will perform the tasks identified as follows:

5.2.6.1 Purchasing Section

- Maintain the chemical inventory.
- Ensure the availability of the SDS for each substance on the chemical inventory.
- Request SDS's from manufacturer when not available.
- Maintain Master SDS Notebooks.

5.2.6.2 Departmental Safety Representative

- Maintain work Area SDS Notebook.
- Review new SDS's at Departmental meetings.
- Coordinate new employee training
- Ensure that all container's for hazardous substances in their departments are properly labeled.

5.2.6.3 Personnel Section

- Maintain training record.
- Coordinate and schedule new employee and annual training sessions with the Chemical Hygiene Officer to ensure that all employees requiring the training are trained on an annual basis.
- Ensure that contractual agreements with outside contractors address hazardous substance use.
- Inform Bench employees of the contractor hazards.
- Inform contractor's of applicable chemical hazards.

5.2.6.4 Chemical Hygiene Officer

- Conducts of coordinates training.
- Provides technical assistance as required.

- Develops training program.

5.2.7 Fire Extinguisher Training

All personnel shall receive fire extinguisher training on an ANNUAL basis. The training shall include the general principles of fire extinguisher use, the classes of fires and the appropriate fire extinguisher for each class and the operation of the various types of fire extinguishers.

The annual training will be coordinated by the Chemical Hygiene Officer. Documentation of the training will be recorded in each employees training record by the Departmental Safety Officer. Fire extinguisher training is mandatory and is required by 29 CFR 1910.157.

5.2.7.1 Portable Fire Extinguishers

- Where the employer has provided portable fire extinguishers for employee use in the workplace, the employee shall also provide an educational program to familiarize employees with the general principles of fire extinguisher use and the hazards involved with incipient stage fire fighting.
- The employer shall provide the education required in the above paragraph upon initial employment and at least annually thereafter.
- The employer shall provide employees who have been designated to use fire-fighting equipment as part of an emergency action plan with training in the use of the appropriate equipment.
- The employer shall provide the training required in the above paragraph upon the initial assignment to the designated group of employees and at least annually thereafter.

5.2.8 First Aid/CPR Training

5.2.8.1 Mandatory Training

First Aid and CPR training is required for any personnel who wear respiratory protective equipment during confined space entries or who conduct field operations where an infirmary, clinic or hospital is not near proximity to the worksite.

5.2.8.2 29 CFR 1910.151

In the absence of an infirmary, clinic or hospital in near proximity to the workplace, which is used for the treatment of all injured employees, a person or persons shall be adequately trained to render first aid. First aid

supplies approved by the consulting physician shall be readily available.

5.2.8.3 Voluntary

It is desired to have as many people trained in First Aid and CPR as possible. For those personnel who are interested in this training, EMAX Laboratories, Incorporated will reimburse any employee the cost of obtaining First Aid and/or CPR training; however, the training must be obtained during non-working hours.

6.0 Medical Consultation and Medical Examinations

6.1 Medical Examinations

Where exposure monitoring reveals an exposure level **routinely** above the action level for an OSHA regulated substance for which there are medical surveillance requirements, medical surveillance shall be established for the affected employee as prescribed by the particular standard.

6.2 Medical Consultation

Whenever an event takes place in work areas such as a spill, leak, explosion, or other occurrence resulting in the likelihood of a significant exposure to a hazardous chemical, the employee shall be provided the opportunity for a medical consultation. Such consultation shall be for the purpose of determining the need for medical examinations and consultations shall be performed by a licensed physician and shall be provided without cost to the employee, without loss of pay and at a reasonable time and place. Whenever an employee develops signs and symptoms associated with a hazardous chemical to which the employee may have been exposed in the laboratory, the employee shall be provided an opportunity to receive an appropriate medical examination.

6.3 Information Provided to the Physician

EMAX Laboratories, Incorporated shall provide the following information to the physician:

1. The identity of the hazardous chemical(s) to which the employee may have been exposed; and
2. A description of the signs and symptoms of exposure that the employee is experiencing, if any.

6.4 Physician Written Opinion

EMAX Laboratories, Incorporated must obtain a written opinion from the examining physician which includes:

1. Any recommendation for further medical follow-up
2. The results of the medical examination and any associated tests.

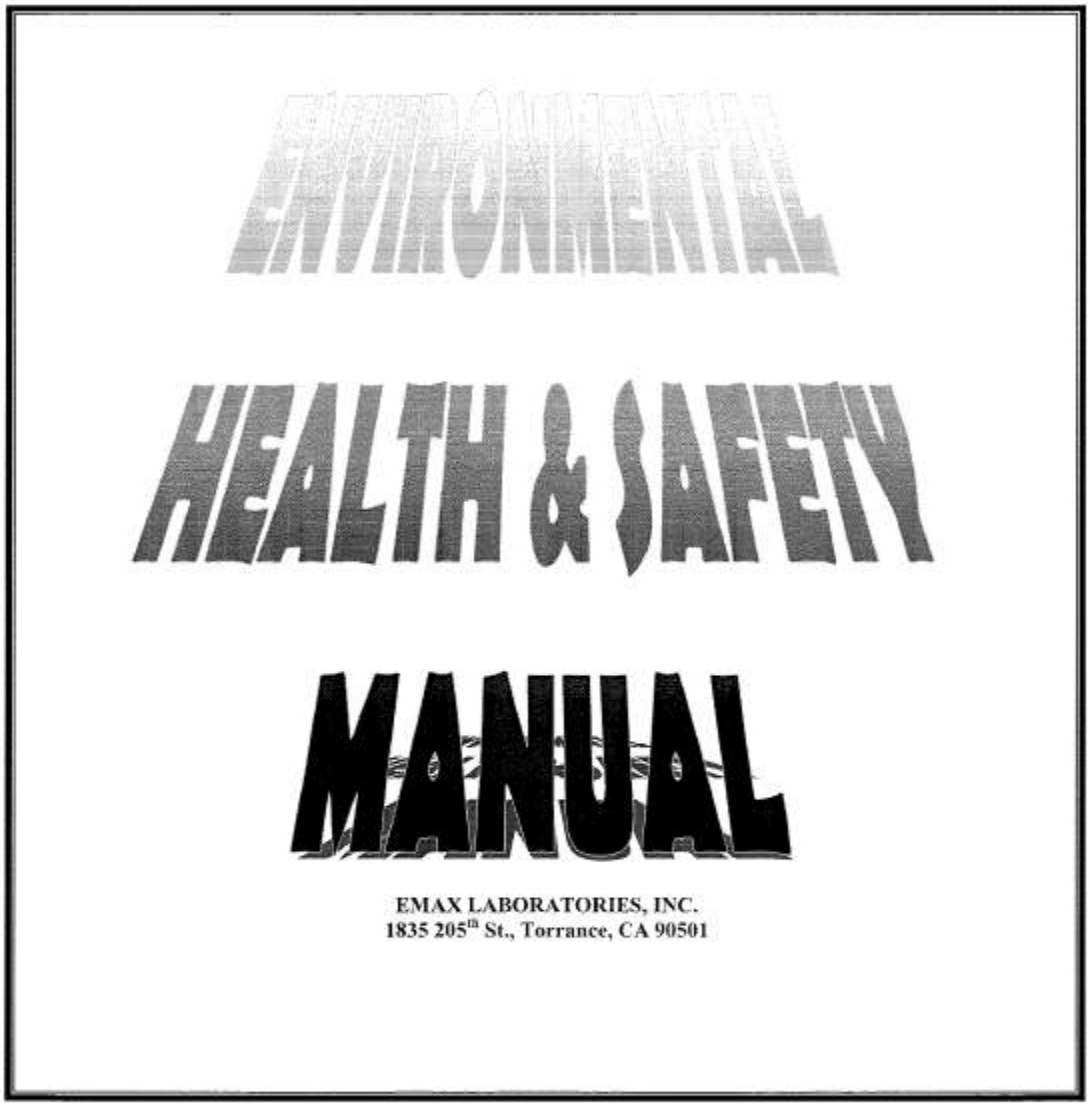
3. Any medical condition which may be revealed in the course of the examination which may place the employee at increased risk as a result of exposure to a hazardous chemical.
4. A statement that the employee has been informed by the physician and any medical condition that may require further examination and treatment.

The written opinion shall not reveal specific findings of diagnosis unrelated to occupational exposure.

6.5 Record Keeping

EMAX Laboratories, Incorporated shall establish and maintain for each employee an accurate record of any measurements taken to monitor employee exposures and any medical consultation and examinations including tests or written physical opinions. These records must be preserved and maintained for at least the duration of employment plus 30 year.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices



Approved By	Richard Beauvil, Safety Officer	8/26/19 Date
Approved By	Caspar Pang, President	8/26/19 Date

Document ID	**EMAX-HSM-05**
Revision No.	5
Date Released	08/26/19
Issue No.	**EMAX-HSM-05-**
Issued To	

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

TABLE OF CONTENTS

TABLE OF CONTENTS 2

ENVIRONMENTAL HEALTH & SAFETY POLICY 4

HEALTH AND SAFETY PROGRAM STRUCTURE 4

1.0 RESPONSIBILITIES 5

1.1. Health and Safety Officer 5
1.2. Radiation Safety Officer 5
1.3. Safety Committee 6
1.4. Department Safety Coordinator 6
1.5. Supervisors 7
1.6. All Employees 7

2.0 HEALTH AND SAFETY RULES & REGULATIONS 8

2.1. Work Ethics 8
2.2. Protective Gear 8
2.3. Food & Beverages 8
2.4. Chemicals 8
2.5. Facility 8
2.6. Ensuring Compliance 9
2.7 Embryo/Fetus Policy 9

3.0 HEALTH AND SAFETY ORIENTATION 10

4.0 EMPLOYEE HEALTH AND SAFETY TRAINING 11

4.1. Initial Training 11
4.2. Training on Specific Hazards 11
4.3. Safety Videos 12
4.4. Safety Datat Sheets 12
4.5. Equipment Operating Manuals 12

5.0 INJURY & ILLNESS PREVENTION PROGRAM (IIPP) 15

5.1. Identifying Workplace Hazards 15
5.2. Communicating Workplace Hazards 15
5.3. Correcting Workplace Hazards 15
5.4. Investigating Injuries And Illnesses 16

6.0 EMERGENCY PROCEDURES 18

6.1. Emergency Fire Procedure 18
6.1.1. Fighting a Fire 18
6.1.2. Types of Fire Extinguishers Used in the Laboratory 18
6.1.3. Using a Fire Extinguisher 18
6.1.4. Fire Safety Tips 19
6.2. Emergency Procedures For Chemical Spillage 19
6.2.1. Organic acids (limited to C, H and O composition) 19
6.2.2. Inorganic acids 19
6.2.3. Alkalis 19
6.2.4. Mercury Spills 20
6.3. Electrical Safety Guidelines 20
6.3.1. General Electrical Safety 20
6.3.2. Extension Cords 20

6.3.3. Electrical Panels 20
6.3.4. Electrical Emergency Response 21

7.0 HEALTH AND SAFETY PROGRAM EVALUATION 22

7.1. General Inspection 22
7.2. Self-Inspection 22

8.0 RECORD KEEPING 22

8.1. Documents Maintained by Health and Safety Office 22

9.0 FORMS 23

Report of Unsafe Condition or Hazard. 24
Hazard Correction Report 25
Occupational Accident, Injury and Illness Investigation Report 26
New Employee Safety Training Record 27
Health and Safety Orientation Form 26
Health and Safety Orientation Tour 27
Safety Training Attendance Record 28
Safety Committee Meeting Documentation 29
Laboratory Inspection Form 30
General Inspection Form 35
Emergency Evacuation Route 38
Location of Safety Material and Equipment 39

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

Environmental Health & Safety Policy

At EMAX the health and safety of our employees comes first. Management is committed to doing everything possible to prevent injuries and to maintain a healthy environment.

- All supervisors are responsible for ensuring that employees are trained in approved work procedures to obtain optimal output without accidents or injuries and to ensure that employees follow safe work methods by adhering to safety regulations.
- All personnel are required to support the Health and Safety program and make safety and health a part of their daily routine and to ensure that they are following safe work methods and relevant regulations;
- All personnel will be held accountable for implementing this program; and all relevant laws and regulations are incorporated in our program as minimum standards

Health and Safety Structure Program Structure

The Health and Safety Manual describes management's commitment to promote health and safety for all employees in the workplace. Any deviation and/or departure from policies established in this manual shall require a duly approved addendum or new revision. The manual is reviewed annually and revisions are issued as appropriate to keep up to date. All revisions to the manual are recorded in the revision history. Obsolete copies of the manual are kept for at least one (1) year. The program sections are described as follows:

1.0 The Health and Safety officer and safety committee are responsible for the formulation and implementation of the program. The Health and Safety Committee is comprised of members who are active participants in the development, implementation, and monitoring of all phases of the health and safety program.

2.0 The Health and Safety Program describes the safety rules and regulations specific to health safety concerns in the workplace, it's enforcement and the consequences when the rules are not followed.

3.0 Health and Safety Orientation describes the orientation process when an employee joins the organization. Specifically included are the policies, rules and the line of authority in the administration.

4.0 Training describes the implementation of health and safety policies into specific job practices and to raise awareness and skill levels to an acceptable standard.

5.0 The Injury & Illness Prevention Program establishes a framework for identifying and correcting workplace hazards within the company, and describes the process of identifying existing hazards to recommend appropriate corrective action. It describes obtaining medical care and/or first aid when necessary.

6.0 The Emergency Procedures describe the procedures for chemical spills, fire, and work injuries.

7.0 The Health and Safety Evaluation describes the process of departmental internal inspection to create environmental health and safety awareness within each department. It provides guidelines for performing regular workplace inspections.

8.0 Record Keeping describes essential record generation, maintenance and the procedure of archiving.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

1.0 RESPONSIBILITIES

Department Supervisors have primary authority and responsibility to ensure departmental implementation of the Injury and Illness Prevention Program (IIPP) and to ensure the health and safety of the employees. This is accomplished by communicating emphasis on health and safety, analyzing work procedures for hazard identification along with correction, ensuring regular workplace inspections, providing health and safety training and encouraging prompt employee reporting of health and safety concerns without fear of reprisal.

1.1. Health and Safety Officer

Responsibilities:

- To develop and implement the safety program procedure in the laboratory.
- To ensure all health and safety regulations are observed.
- To ensure all issues of concern are related to the laboratory Director.
- To train new employees.
- To develop, administer and update the Health and Safety manual, the Chemical Hygiene Plan, and the Right-to-Know manual.
- To maintain Safety Data Sheets (SDS) and to provide training in their use.
- To monitor the procurement, use and disposal of chemicals in the laboratory.

Qualifications:

- Education: Minimum of Bachelor's degree in chemistry or any science/engineering discipline or equivalent, with 40 hour training on Hazardous Waste Management.

- Experience: Minimum of one-year experience in administering health and safety regulations.

1.2. Radiation Safety Officer (RSO)

Responsibilities:

- To develop and implement the Radiation Protection Program.
- To direct the use of radioactive material for its intended purpose in a manner that protects the health and minimizes the danger to life or property.
- To ensure that personnel radiation exposure be maintained As-Low-As-Reasonably-Achievable (ALARA).
- To train new employees in safe work practices.

Qualifications:

- Education: Minimum of Bachelor's degree in chemistry, any science/engineering discipline or equivalent. Qualified Officer must have completed Radiation Safety School and have received the RSO certification.

- Experience: Minimum of one(1) year applied experience dealing with radiation principles and practices in an analytical laboratory or equivalent.

1.3. Safety Committee

The Safety Committee shall have a representative from each department. The Safety Committee has the ongoing responsibility to maintain and update this Injury and Illness Prevention Program (IIPP), to assess

departmental compliance with applicable regulations and policies, to evaluate reports of unsafe condition, and to coordinate any necessary corrective actions. The Safety Committee is to meet on a yearly basis or as needed. Currently, the department's Safety Committee consists of:

The Safety Committee membership may rotate periodically.

Unsafe conditions that cannot be immediately corrected by an employee and/or his/her supervisor should be reported to the Department Safety Coordinator or any Safety Committee member by filling out a "Report of Unsafe Condition or Hazard" form.

The Safety Committee will be responsible for reporting unsafe conditions and injury on a timely manner. The Safety Committee will:

- Review the results of workplace inspections to identify any necessary safety procedures or programs.
- Track specific corrective actions.
- Review supervisors' investigations of accidents and injuries to ensure that all causes have been identified and corrected.
- Where appropriate, submit suggestions to management for the prevention of future incidents.
- Review alleged hazardous conditions brought to the attention of any committee member, determine necessary corrective actions, and assign responsible parties and correction deadlines
- When determined necessary by the Committee, the Committee may conduct its own investigation of accidents and/or alleged hazards to assist in establishing corrective actions
- Submit recommendations to assist department management in the evaluation of employee safety suggestions

The Safety Committee shall prepare and make available to all department personnel written minutes of issues discussed at the meetings. The Committee meeting minutes must be documented. These minutes are posted at the lunchroom and shall be mentioned on QA meetings.

1.4. Department Safety Coordinator

The Safety Coordinator has responsibility for:

- Ensuring that the Safety Committee is aware of all accidents which have occurred, and all hazards which have been observed since the last meeting
- Working with the Building Coordinator to address facility-related safety concerns
- Assisting in the coordination of required health and safety training
- Serving as liaison with HEALTH AND SAFETY and other campus safety resources on issues the department cannot resolve
- Maintaining copies of Safety Committee minutes and other safety-related records
- The Safety Coordinator may seek assistance from other members of the department as necessary to meet these responsibilities.

1.5. Supervisors

Supervisors play a key role in the implementation of the department's IIPP. (For the purpose of this template, the term "supervisor" includes any employee who oversees the work of others.) They are responsible for:

- Communicating to their staff with appropriate emphasis on health and safety
- Ensuring periodic, documented inspection of workspaces under their authority

- Promptly correcting identified hazards
- Modeling and enforcing safe and healthful work practices
- Providing appropriate safety training and personal protective equipment
- Implementing measures to eliminate or control workplace hazards
- Stopping any employee's work that poses an imminent hazard to either the employee or any other individual
- Encouraging employees to report health and safety issues to the Safety Committee without fear of reprisal

1.6. **All Employees**

It is the responsibility of all staff to comply with all applicable health and safety regulations, EMAX HEALTH AND SAFETY policies, and established work practices. This includes but is not limited to:

- Observing health and safety-related signs, posters, warning signals and directions
- Reviewing the building emergency plan and assembly area
- Learning about the potential hazards of assigned tasks and work areas
- Taking part in appropriate health and safety training
- Following all safe operating procedures and precautions
- Using proper personal protective equipment
- Warning coworkers about defective equipment and other hazards
- Reporting unsafe conditions immediately to a supervisor, and stopping work if an imminent hazard is presented
- Participating in workplace safety inspections

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

2.0 HEALTH AND SAFETY RULES & REGULATIONS

2.1. Work Ethics

2.1.1. All new employees must undergo Health and Safety orientation prior to commencement of work.

2.1.2. All employees shall undergo safety training applicable to the activities they perform prior to performance of work.

2.2. Protective Gears

2.2.1. A closed-toe shoe is the only footwear that can be used while working in the laboratories.

2.2.2. Short pants and skirts are not allowed in the laboratories.

2.2.3. Lab coats shall be worn in the laboratories while performing activities involving instrumentation, samples or chemicals and must be removed when leaving the lab. Lab coats are not to be worn when going to the lunchroom, restrooms or the front offices.

2.2.4. Safety glasses shall be worn at all times as prescribed by the safety procedures of the activity being performed. Safety glasses shall be worn by administrative staff in areas of the lab, where hazards (physical or chemical) are present. Contact lenses are not allowed in the laboratories.

2.2.5. Face shields shall be worn at all times when handling corrosive chemicals e.g. concentrated acids or concentrated bases.

2.2.6. Gloves shall be worn at all times when handling samples, chemicals and as prescribed by the safety procedures of the activity being performed. Gloves shall be removed while performing administrative tasks. (Keyboards, logbooks, etc.). Gloves shall not be worn in the lunchroom, restrooms and front offices.

2.2.7. Respiratory protective equipment shall be worn when working with known toxic chemicals in confined spaces.

2.2.8. Earplugs shall be worn when working with a pulse sonicator or other equipment that produces the same noise level of $\geq$140 decibels.

2.3. Food & Beverages

2.3.1. The lunchroom is the only place where food can be stored for a very short periods and consumed. Overnight storage of perishable is not permitted.

2.3.2. No eating, drinking or chewing gum in the laboratories.

2.4. Chemicals

2.4.1. All chemicals shall be labeled properly.

2.4.2. All chemicals should be contained in a chemical carrier while transporting.

2.4.3. Incompatible chemicals must be stored separately.

2.5. Facility

2.5.1. Proper ventilation must be maintained in every workspace.

2.5.2. Cleanliness must be observed at all times.

2.5.3. Wet floors must be flagged with caution signs.

2.5.4. Fire extinguishers must be properly located, inspected, and maintained

2.5.5. First Aid kits must be properly located, inspected, and supplied.

2.5.6. Emergency eyewash and shower stations must be inspected.

2.6. **Ensuring Compliance**

2.6.1. All personnel have the responsibility for complying with safe and healthful work practices including applicable regulations, Health and Safety policy, and safety procedures. Overall performance in maintenance of a safe and healthful work environment should be recognized by the supervisor and noted in performance evaluations. Employees will not be discriminated against for work-related injuries, and injuries will not be listed in performance evaluations, unless the injuries were a result of an unsafe act on the part of the employee.

2.6.2. Standard progressive disciplinary measures in accordance with the applicable personnel policy will result when employees fail to comply with applicable regulations of the Health and Safety policy, and/or safety procedures. Staff members will be disciplined for unsafe practices in accordance with the EMAX Code of Conduct contained in the Human Resources Manual. All personnel will be given instruction and an opportunity to correct unsafe behavior. Repeated failure to comply or willful and intentional non-compliance may result in disciplinary measures up to and including termination.

2.7. **Embryo/Fetus Policy**

2.7.1. EMAX policies shall ensure that employees that are pregnant are protected from hazardous chemicals and radioactive materials. EMAX shall provide right to know training for all employees on Embryo/Fetus policy. Employees who are pregnant shall declare their pregnancy to EMAX human resources for their protection and the protection of the unborn child. EMAX shall discuss potential chemicals and radioactive exposure for employees working in the lab and options for the reminder of their work during the pregnancy.

2.7.2. Exposure of the unborn child shall not exceed 500 millirem for the entire gestation period. Individuals are encouraged to notify human resources and the RSO regarding "declared pregnancies".

3.0 HEALTH AND SAFETY ORIENTATION

Health and safety education should start with employee orientation when an employee joins the organization or is transferred to a new job. At a minimum Health and Safety orientation should include the following:

3.1. Health and Safety rules and regulations

3.2. Reasons for each health and safety rule

3.3. Emergency procedures

3.4. Location of first aid stations

3.5. Reporting of injuries, unsafe conditions and acts

3.6. Use of personal protective equipment

3.7. Right to refuse hazardous work

3.8. Hazards, including those outside own work area

A new employee can be expected to absorb only a certain amount of information in the first few days. A brochure outlining the points covered in the orientation sessions is useful as a handout to employees. It also serves as a checklist for the person conducting the orientation. A mentor system is a useful follow-up to the initial orientation. This allows for on-the-job reinforcement of the information presented to the new employee. This process promotes the safety awareness of the experienced workers who are the "mentors".

New, inexperienced or transferred employees should be encouraged to ask questions at any time when doubt exists as to correct procedures.

The supervisor or one of the analysts working in the same department will be the new employee's mentor for the first few months. During that period, the new employee will be trained according to EMAX.QA05.

4.0 EMPLOYEE HEALTH AND SAFETY TRAINING

Employee safety training is conducted during the employee's normal working hours on EMAX time. Safety training may be presented by a knowledgeable supervisor, other department personnel or by representatives from other relevant sources. Regardless of the instructor, all safety training must be documented using the "Safety Training Attendance Record" or an equivalent record. By law, this documentation must be retained by the department for at least five years.

4.1. Initial Training

The objective of training is to ease the implementation of health and safety policies into specific job practices and to raise awareness and skill levels to an acceptable standard. While all employees can benefit from health and safety training, special attention should be given to the training of supervisors, trainers, and workers.

When an employee is being trained on new assignments all safety features prescribed in conducting the procedure involved becomes an integral part of the training. The trainer is directly responsible for health and safety matters that are incorporated into the procedure.

Occasions when employee training may be required are:

- commencement of employment
- reassignment or transfer to a new job
- introduction of new equipment, processes, or procedures
- inadequate performance

Training will also be provided on how to report unsafe conditions, how to access the Safety Committee, and where to obtain information on workplace safety and health issues.

4.2. Training on Specific Hazards

Supervisors are required to be trained on the hazards to which the employees under their immediate control may be exposed. This training aids a supervisor in understanding and enforcing proper protective measures.

All supervisors must ensure that the personnel they supervise receive appropriate training on the specific hazards of work they perform, and the proper precautions for protection against those hazards. Training is particularly important for new employees and whenever a new hazard is introduced into the workplace. Such hazards may include new equipment, hazardous materials, or procedures. Health and Safety training is also required when employees are given new job assignments on which they have not previously been trained and whenever a supervisor is made aware of a new or previously unrecognized hazard.

Specific topics which may be appropriate to department personnel include but are not limited to the following:

- Understanding Safety Data Sheet (SDS).
- Fire prevention techniques and fire extinguisher use.
- Obtaining emergency medical assistance and first aid.
- Disaster preparedness and response, including building evacuation procedures.
- Health and safety for computer users.
- Back care, body mechanics, and proper lifting techniques .
- Hazard communication, including training on SDSs, chemical hazards and container labeling.
- Proper Laboratory housekeeping.

- Chemical spill reporting procedures.

All training are documented in the safety training record.

4.3. Safety Videos

A list of workplace safety videos can be obtained by contacting health and safety officer. Videos are available on a wide range of topics, including hazard communication, chemical safety, and various physical hazards. Videos should be used to supplement, not replace, face-to-face safety instruction, so that trainees have an opportunity to ask questions of a knowledgeable instructor.

4.4. Safety Data Sheets

Safety Data Sheets (SDSs) provide information on the potential hazards of products or chemicals. Hard copies of SDSs for the chemicals used in the department are available in 3-ring binders located at the sample receiving area. If an SDS is found to be missing, a new one can be obtained by faxing a written request to the manufacturer. A copy of this request should be kept until the SDS arrives. In addition, SDS can be obtained in the Health and Safety browser. Understanding the use of SDS is also detailed in the EMAX browser and in the Chemical Hygiene plan.

4.5. Equipment Operating Manuals

All equipment is to be operated in accordance with the manufacturer's instructions, as specified in the equipment's operating manual. Copies of operating manuals should be kept with each piece of equipment in the department. Persons who are unfamiliar with the operation of a piece of equipment and its potential hazards must read the operating manual before using the equipment and demonstrate equipment proficiency to their supervisors.

4.6. Globally Harmonized System (GHS) Training

GHS is an international approach to hazard communication, providing agreed criteria for classification of chemical hazards, and a standardized approach to label elements and safety data sheets. The GHS was negotiated in a multi-year process by hazard communication experts from many different countries, international organizations, and stakeholder groups. It is based on major existing systems around the world, including OSHA's Hazard Communication Standard and the chemical classification and labeling systems of other US agencies.

The definitions of hazard have been changed to provide specific criteria for classification of health and physical hazards, as well as classification of mixtures. These specific criteria will help to ensure that evaluations of hazardous effects are consistent across manufacturers, and that labels and safety data sheets are more accurate as a result.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

4.6.1. Labeling

OSHA is requiring that employees are trained on the new label elements (i.e, pictograms, hazard statements, precautionary statements, and signal words)

4.6.2. Safety Data Sheets (SDS)

- The Hazard Communication Standard (HCS) requires chemical manufacturers, distributors, or importers to provide Safety Data Sheets (SDSs) (formerly known as Material Safety Data Sheets or MSDSs) to communicate the hazards of hazardous chemical products. As of June 1, 2015, the HCS will require new SDSs to be in a uniform format, and include the section numbers, the headings, and associated information under the headings below:
- **Section 1, Identification** includes product identifier; manufacturer or distributor name, address, phone number; emergency phone number; recommended use; restrictions on use.
- **Section 2, Hazard(s) identification** includes all hazards regarding the chemical; required label elements.
- **Section 3, Composition/information on ingredients** includes information on chemical ingredients; trade secret claims.
- **Section 4, First-aid measures** includes important symptoms/ effects, acute, delayed; required treatment.
- **Section 5, Fire-fighting measures** lists suitable extinguishing techniques, equipment; chemical hazards from fire.
- **Section 6, Accidental release measures** lists emergency procedures; protective equipment; proper methods of containment and cleanup.
- **Section 7, Handling and storage** lists precautions for safe handling and storage, including incompatibilities.

- **Section 8, Exposure controls/personal protection** lists OSHA's Permissible Exposure Limits (PELs); Threshold Limit Values (TLVs); appropriate engineering controls; personal protective equipment (PPE).
- **Section 9, Physical and chemical properties** lists the chemical's characteristics.
- **Section 10, Stability and reactivity** lists chemical stability and possibility of hazardous reactions.
- **Section 11, Toxicological information** includes routes of exposure; related symptoms, acute and chronic effects; numerical measures of toxicity.
- Section 12, Ecological information*
- Section 13, Disposal considerations*
- Section 14, Transport information*
- Section 15, Regulatory information*
- **Section 16, Other information,** includes the date of preparation or last revision.

5.0 INJURY & ILLNESS PREVENTION PROGRAM (IIPP)

5.1. Identifying Workplace Hazards

Regular, periodic workplace safety inspections must be conducted throughout the department by the Safety Officer or his representative. By law, the first of these inspections must take place when the IIPP is first adopted. The inspections should be documented, and copies of this documentation must be retained by the Health and Safety officer for at least one year. These regular inspections will be supplemented with additional inspections whenever new substances, processes, procedures, or equipment introduced into the workplace represent a new occupational safety and health hazard or whenever supervisors are made aware of a new or previously unrecognized hazard.

Supervisors are responsible for identification and correction of hazards that their staff face and should ensure that work areas they exercise control over are inspected at least annually. Supervisors should check for safe work practices with each visit to the workplace and should provide immediate verbal feedback where hazards are observed.

The "Report of Unsafe Condition" Form should be filled out when a referral is made to the Safety Committee as a result of a condition discovered during an inspection for which the responsible supervisor could not determine an immediate remedy. The "Report of Unsafe Condition" form can also be downloaded from the Health and Safety Browser, filled out and turned to the Safety Officer. No signature is necessary.

5.2. Communicating Workplace Hazards

Supervisors are responsible for communicating with workers about safety and health issues. All department personnel are encouraged to communicate safety concerns to their supervisor without fear of reprisal.

The Safety Committee is another resource for communication regarding health and safety issues for department employees. Each employee has a representative on the committee that will inform him or her of hazard corrections and committee activities. Additionally, Safety Committee minutes and other safety-related items are posted in the lunchroom bulletin board. Employees will also be informed about safety matters by distribution of written memoranda, or by attendance of safety meetings. Occasionally, the Safety Committee may also sponsor seminars or speakers or coordinate other means to communicate with employees regarding health and safety matters.

Supervisors are responsible for ensuring that employees are supplied access to hazard information pertinent to their work assignments. Information concerning the health and safety hazards of tasks performed by department staff is available from a number of sources. These sources include, but are not limited to, Safety Data Sheets (SDSs, see below), equipment operating manuals, the Department Safety Coordinator, HEALTH AND SAFETY, container labels and work area postings.

5.3. Correcting Workplace Hazards

Hazards discovered either as a result of a scheduled periodic inspection or during normal operations must be corrected by the supervisor in control of the work area, or by cooperation between the department in control of the work area and the supervisor of the employees working in that area. Supervisors of affected employees are expected to correct unsafe conditions as quickly as possible after discovery of a hazard, based on the severity of the hazard.

Specific procedures that can be used to correct hazards include but are not limited to the following:

- Tagging unsafe equipment "Do Not Use Until Repaired," and providing a list of alternatives for employees to use until the item is repaired.
- Stopping unsafe work practices and providing retraining on proper procedures before work resumes.

- Reinforcing and explaining the need for proper personal protective equipment and ensuring its availability and use.
- Barricading areas that have chemical spills or other hazards and reporting the hazardous conditions to a supervisor or Building Coordinator.

Supervisors should use the "Hazard Correction Report" to document corrective actions, including projected and actual completion dates. If necessary, supervisors can seek assistance in developing appropriate corrective actions by submitting a "Report of Unsafe Condition" to the Safety Committee. If the Safety Committee requires assistance from Health and Safety Officer, or Operations Manager these resources should be contacted immediately.

If an imminent hazard exists, work in the area shall cease, and the appropriate supervisor must be contacted immediately. If the hazard cannot be immediately corrected without endangering employees or property, all personnel need to be removed from the area except for those qualified and necessary to correct the condition. These qualified individuals will be equipped with necessary safeguards before addressing the situation.

5.4. Investigating Injuries And Illnesses

5.4.1. Injury Reporting

Employees who are injured at work must report the injury immediately to their supervisor. If immediate emergency medical treatment beyond first aid is needed, **dial 9-911**. The injured party will be taken to the appropriate hospital or medical center. If non-emergency medical treatment for work-related injuries or illnesses is needed, obtain treatment authorization form and go to **Kaiser Permanente On-the-Job South Bay Medical Offices 18600 S. Figueroa Street, Suite 120 Gardena, CA 90248. Phone: 310-527-5600.**

The supervisor of the injured employee must work with the department's safety coordinator to ensure that the "Employer's Report of Occupational Injury or Illness" and a "Workers' Compensation Claim Form" are completed properly and submitted to the Human Resources Department.

If the injured employee saw a physician, the supervisor shall obtain a medical release form before allowing the employee to return to work. The health care provider may stipulate work tasks that must be avoided or work conditions that must be modified before the employee resumes his or her full duties.

5.4.2. Injury Investigation

The injured employee's supervisor is responsible for performing an investigation to determine and correct the cause(s) of the incident. Specific procedures that can be used to investigate workplace accidents and hazardous substance exposures include:

- Interview the injured personnel and witnesses
- Examine the injured employee's workstation for causative factors
- Review the established procedures to ensure they are adequate and were followed by the employee.
- Review training records of injured employees.
- Determine all contributing causes to the accident.
- Take corrective actions to reduce potential for similar accident/exposure from reoccurring.
- Record all findings and actions taken involving the injury.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

The supervisor's findings and corrective actions should be documented and presented to the Safety Committee using the "Occupational Accident, Injury or Illness Investigation Report". If the supervisor is unable to determine the cause(s) and appropriate corrective actions, other resources should be sought. Available resources include the department's Safety Committee and the Safety Officer.

The Safety Committee will review each accident or injury report to ensure that the investigation was thorough and that all corrective actions are completed. Investigations and/or corrective actions that are found to be incomplete will be routed back to the supervisor for further follow-up, with specific recommendations noted by the committee. The Department Safety Coordinator will bring corrective actions that are not implemented in a reasonable period of time to the attention of the Safety Officer and/or Operation Manager.

6.0 EMERGENCY PROCEDURES

Emergency procedures are plans for dealing with emergencies such as fires, explosions, major releases of hazardous materials, violent occurrences, or natural hazards. When such events occur, the urgent need for rapid decisions, shortage of time, lack of resources, and trained personnel can lead to major confusion. The objective of the plan is to prevent or minimize fatalities, injuries, and damage. The organization and procedures for handling these sudden and unexpected situations must be clearly defined.

6.1. Emergency Fire Procedures

- Sound the alarm. Activate the fire alarm as soon as you find or suspect a fire.
- Exit Immediately. Leave the building. As you leave, warn other occupants by knocking on doors and shouting. Refer to Figure 1 for Emergency Evacuation Route.
- In case the stairs are needed, go to the nearest stairway or exit. Stairway doors--if closed--will keep out fire and smoke and protect you until you are outside.
- If heat, fire or smoke blocks the nearest exit, stay low and go to another exit.
- Stay out of the building until given the OK to return by fire officials.
- Call 911. Report the emergency from a safe location. Dial 9-911 from an EMAX phone or 911 from a cellular phone if available. Provide the dispatcher with as much information as possible.

6.1.1. Fighting a Fire

If the fire is very small, and you know how to use a fire extinguisher safely, you may attempt to put out the fire. Make sure the fire department has been called first! Be sure you have the right fire extinguisher for the type of fire you are attempting to fight. Using the wrong type of extinguisher may make the fire worse. Do not let the fire get between you and your exit route. Stay between the fire and the door. If one extinguisher does not put out the fire, get out immediately, closing the door behind you.

6.1.2. Types of Fire Extinguishers Used in the Laboratory

Fire extinguishers are categorized by the type of fires they put out:

- Type A--Ordinary combustibles such as wood, paper, cloth, rubber, and plastics.
- Type B--Flammables liquids such as gasoline, oils, paints, and grease.
- Type C--Electrical fire such as burning wires, switches, machinery, and appliances.

Fire extinguishers will have a label indicating what type of fire they can extinguish. Extinguishers found in the laboratory are ABC extinguishers and can fight all three kinds of fires. Using the wrong extinguisher can be dangerous. For example, using a Type A extinguisher on a Type B fire (burning liquid) can cause the liquid to dissipate, spreading the fire. If you're not sure about either the type of fire or the type of extinguisher, wait for the fire department.

6.1.3. Using a Fire Extinguisher

If you know you have the right extinguisher and the fire is small enough to fight safely, remember the PASS acronym, to use your extinguisher effectively:

- **Pull** the safety pin at the top of the extinguisher.
- **Aim** the nozzle, horn, or hose at the base of the flames.
- **Squeeze** the handle of the extinguisher.
- **Sweep** the nozzle from side to side until the fire goes out.

6.1.4. **Fire Safety Tips**

- Know your exits. There is a fire exit path in every department. Be familiar with it.
- Keep your exit paths clear.
- Know where the nearest fire alarm and fire extinguisher are.
- Remember - extension cords are for temporary power only.
- Participate in fire drills.
- Observe no smoking postings.

6.2. **Emergency Procedures For Chemical Spillage**

6.2.1. **Organic Solvent**

- Eliminate all sources of ignition and instruct others to keep at a safe distance, well away from the spillage area.
- Open windows if possible and close laboratory doors on the way out. Aim to ventilate the area but isolate the material.
- Decide whether to control the spillage on site, or to evacuate the building by sounding the fire alarm and calling in the local fire brigade.
- Wear a laboratory coat or protective organic solvent proof overall, organic solvent proof rubber gloves, eye and/or face protection and air purifying respirator.
- Dispose spill clean up according to CHP section 3.5 – 3.6.

6.2.2. **Inorganic acids**

- Eliminate all sources of ignition and instruct others to keep at a safe distance, well away from the spillage area.
- Open windows if possible and close laboratory doors on the way out. Aim to ventilate the area but isolate the material.
- Decide whether to control the spillage on site, or to evacuate the building by sounding the fire alarm and calling in the local fire brigade.
- Wear a laboratory coat or protective acid-proof overall, acid-proof rubber gloves, eye and/or face protection and air purifying respirator.
- Dispose spill clean up according to CHP Section 3.5 – 3.6.

6.2.3. **Alkalis - Note that these compounds may react violently with water.**

- Eliminate all sources of ignition and instruct others to keep at a safe distance, well away from the spillage area.
- Open windows if possible and close laboratory doors on the way out. Aim to ventilate the area but isolate the material.
- Decide whether to control the spillage on site, or to evacuate the building by sounding the fire alarm and calling in the local fire brigade.
- Wear a laboratory coat, protective gloves, eye and/or face protection and air purifying respirator.
- Dispose spill clean up according to CHP section 3.5 – 3.6.

6.2.4. Mercury Spills

Mercury spills should be cleaned up immediately and treated as any other hazardous chemical waste.

6.3 Electrical Safety Guidelines

The danger of injury through electric shock is possible whenever electrical power is present. When a person's body completes a circuit and thus connects a power source with the ground, an electrical burn or injury is imminent. Most fatal injuries result from high voltage exposure; however, people can sustain severe injuries from low voltage power if it has a high current flow. In addition, overloaded circuits and poorly maintained electrical equipment and connections can lead to fires. Electrical fires are also capable of causing injuries and usually result in significant damage to facilities.

6.3.1 General Electrical Safety

Electrical safety is important in every laboratory and office. Follow these guidelines for general electrical safety:

- Be familiar with the electrical hazards in your laboratory or office.
- Be familiar with electrical emergency response procedures.
- Unplug electrical equipment before repairing or servicing.
- DO NOT work on electrical equipment unless you are qualified to do so. This includes changing electrical plugs on equipment.
- Inspect cords and plugs for defects such as frayed wiring, loose connections or cracked insulation. Repair or replace items, which exhibit these defects.
- Ensure that all outlets are firmly mounted and in good condition.
- Report all electrical problems (tripped breakers, broken switches and flickering lights) to the Safety Officer or the Facility Manager.
- Keep electrical equipment and appliances away from water.
- Avoid overloading circuits--if possible only plug a single appliance into an outlet.
- If multiple appliances are necessary, use an approved UL listed power strip that has a surge protector and a circuit breaker.
- Ensure that all electrical equipment have grounded plugs (3-prong for 110/208V single phase and 4-prong for any 3-phase system).
- Keep electric cords away from areas where they may be pinched and areas where they may pose a tripping or fire hazard (i.e. door ways, walkways, under carpet, etc.).

6.3.2 Extension Cords

Extension cords should only be used on a temporary basis and are not to be used in place of permanent wiring. If work requires using equipment or appliances in areas where there are not enough outlets, a request for additional installation of outlets from the department supervisor as needed.

Ensure that all extension cords are correct by size and rated for the equipment in use. The diameter of the extension cord should be the same or greater than the cord of the equipment. If you are unsure about the rating of an extension cord, contact the Safety Officer or the Facility Manager for advice.

6.3.3 Electrical Panels

Electrical panels or breaker boxes require special safety considerations, including the following:

- Know the location of the electrical panel in your work area.
- Do not block access to the panel with furniture or equipment. You must maintain a 36 " clearance in front of the panel to provide unobstructed access.
- Never tape circuit switches to keep a breaker from tripping.
- Ensure that breakers are accurately labeled within panel boxes.
- Ensure that the panel door is securely attached.
- Report tripped breakers to Facility Manager.

6.3.4 Electrical Emergency Response

All employees should be familiar with the following guidelines in case of electrical injury:

- If the victim is still in contact with the electric current, immediately turn off the power source.
- If you cannot disconnect the power source, try to separate or pry the victim away from the power source by using a non-conductive item such as a piece of dry wood or book.
- **Never touch a victim still in contact with the power source; you could electrocute yourself.**
- After removing the person from the power source, call 9-911 to summon medical help and administer first aid or CPR if you are trained to do so.

7.0 HEALTH AND SAFETY PROGRAM EVALUATION

7.1. General Inspection

A quarterly Health and Safety general audit shall be conducted to measure the effectiveness of the HEALTH AND SAFETY program. The audit uses a checklist in which each area of concern is checked for appropriateness and adequacy for a safe and healthy environment. Records, observations, interviews, and questionnaires are used to evaluate performance for each sub-element. The audit team includes representation from the joint health and safety committee, must receive appropriate training in audit procedures. The audit identifies weaknesses in the health and safety program as well as its strength. A procedure is also established to ensure prompt follow-up on deficiencies. This procedure must include provision for target dates for remedial action and checks to confirm completion.

7.2. Self-Inspection

All laboratories are required to perform and document self-inspections on a quarterly basis. The self-inspection form will help document laboratory safety inspections and will assist laboratory personnel in identifying and correcting many common unsafe practices and conditions. The unsafe practices and conditions identified are prohibited by state laws or EMAX policies, or are not generally accepted safe laboratory practices. Findings shall be documented in the Laboratory Self-Inspection form.
A designated individual in each department must inspect the laboratory using this form and answer each question by checking Yes (Satisfactory), No (Needs Correction), or N/A (Not Applicable) if the question does not apply to the department. After completing the self-inspection form, results must be shared with the Supervisor, and the individual in the department.
Correct each identified deficiency as soon as possible and document correction on the form.
Inform the Operations Manager if assistance is needed in correcting conditions identified during the self-inspection, or if there are questions or concerns about laboratory safety, whether or not they pertain to this self-inspection.
Submit the original self-inspection form to the Health and Safety Officer for proper record keeping.
This checklist does not address specific activities involving radioactive materials or radiation-producing machines, which are separately inspected by the RSO or his designee.

8.0 RECORD KEEPING

8.1. Documents Maintained by Health and Safety Officer

By law, certain documents related to the HEALTH AND SAFETY must be kept by the safety officer and maintained

in accordance with contractual and regulatory requirements. These records include:

- Records of scheduled and periodic workplace inspections, including the persons conducting the inspection, any identified unsafe conditions or work practices, and corrective actions taken.
- Employee safety training records, including the names of all attendees and instructors, the training date, and material covered.
- Reports of Unsafe Conditions or Hazards.
- Safety Committee Meeting Documentation.
- Hazard Correction Reports.
- Accident, Injury or Illness Investigation Reports.

9.0 Forms

9.1. Report Of Unsafe Condition Or Hazard Form
9.2. Hazard Correction Report Form
9.3. Occupational Accident, Injury Or Illness Investigation Report Form
9.4. Safety Training Attendance Record Form
9.5. New Employee Safety Training Record Form
9.6. Health and Safety Orientation Form
9.7. Health and Safety Orientation Tour
9.8. Safety Committee Meeting Documentation Form
9.9. Laboratory Self-Inspection Form
9.10. General Self-Inspection Form
9.11. Emergency Evacuation Route
9.12. Location of Safety Material and Equipment

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

REPORT OF UNSAFE CONDITION OR HAZARD

I. Unsafe Condition or Hazard	
Name: (optional)	
Title:	Department
Location of Hazard:	
Date and time the condition or hazard was observed:	
Description of unsafe condition or hazard:	
What changes would you recommend to correct the condition or hazard?	
Employee Signature: (optional)	Date
II. Management/Safety Committee Investigation	
Name of person investigating unsafe condition or hazard:	
Results of investigation (What was found? Was condition unsafe or a hazard?): (Attach additional sheets if necessary.)	
Proposed action to be taken to correct hazard or unsafe condition:	
Signature of Investigating Party:	Date:

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

HAZARD CORRECTION REPORT

Department:_____

This form should be used in conjunction with the "Report of Unsafe Condition" as appropriate, to track the correction of identified hazards.

All hazards should be corrected as soon as possible based on the severity of the hazard. If a serious, imminent hazard cannot be immediately corrected, remove personnel from the area and restrict access until the hazard can be corrected.

Supervisor/Safety Coordinator Name:_____ Telephone Ext.:_____

_____ _____

Supervisor/Safety Coordinator Signature Date

Description and Location of Unsafe Condition	Date Discovered	Required Action and Responsible Party	Completion Date	
			Projected	Actual

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

OCCUPATIONAL ACCIDENT, INJURY OR ILLNESS INVESTIGATION REPORT

Department:		
Supervisor's Name		
Person(s) involved:		
Location:	Time:	Date:
Task being performed when accident occurred:		

NOTE: This form is intended to serve only as a local record of the investigation conducted within the department. Should an injury or illness occur, required forms must be submitted to the Human Resources Department. "Hazard Correction Report" must be completed in conjunction with any accident, injury or illness.

Describe the accident, illness, or injury and the probable root cause(s) of the incident. Include the nature of the injury or illness, any eyewitness accounts, and any property damage, which may have occurred. Be sure to include the names and phone numbers of any witnesses. Attach a separate sheet if necessary.

Describe what corrective actions need to be taken to ensure this type of incident does not recur. Also, include the name(s) and phone number(s) of those who will ensure that these corrective actions are done in a timely manner.

Signature of Supervisor Conducting Investigation Date

Signature of Department Safety Coordinator Date

(Do not sign until a thorough review of the incident by the Safety Committee is complete and corrective actions are in place.)

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

NEW EMPLOYEE SAFETY TRAINING RECORD

Department:

Instructor/Supervisor:

This completed form should be retained in the individual's personnel file as evidence of initial training required under the Injury and Illness Prevention Program. Follow-up training covering more specific topics can also be documented by completing the space provided in item III below.

Employee Name:

☐ New Hire ☐ Transfer ☐ Other:

Date of Hire / New Assignment:

I, _____, hereby certify that this employee has
(Instructor)

been trained on the following: (Check appropriate boxes.)

I. ☐ Initial Training on Department's IIPP, Including: Date:

- My right to ask any question, or report any safety hazards, either directly or anonymously without any fear of reprisal.
- The location of departmental safety bulletins and required safety postings (i.e., summary of occupational injuries and illnesses, and Safety and Health Protection Poster).
- Disciplinary procedures that may be used to ensure compliance with safe work practices.
- Reporting safety concerns.
- Accessing the department safety committee.
- Reporting occupational injuries and illnesses.

II. ☐ Hazard Communication Training Date:

- The potential occupational hazards in the work area associated with my job assignment.
- The safe work practices and personal protective equipment required for my job title.
- The location and availability of SDSs.
- The hazards of any chemicals to which I may be exposed, and my right to the information contained on Safety Data Sheets (SDSs) for those chemicals.

III. ☐ Other Safety Training Date:

Describe Subject Covered: (Attach documentation.)

I understand the above items and agree to comply with safe work practices in my work area.

Employee Signature: Date:

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

HEALTH AND SAFETY ORIENTATION FORM

EMPLOYEE NAME _____

DEPARTMENT _____

TITLE _____

HIRE DATE _____

1. SAFETY TOUR

   Date Completed _____ Employee Initials _____ Safety Rep's Initials _____

2. SAFETY AND HEALTH MANUAL (Reviewed marked Sections)

_____ Section 1	_____ Section 5.1	_____ Section 6
_____ Section 2	_____ Section 5.2	_____ Section 7
_____ Section 3	_____ Section 5.3	_____ Section 8
Section 4	_____ Section 5.4	_____ Section 9

   Date Reviewed _____ Employees Initials _____

3. PERSONAL PROTECTIVE EQUIPMENT

_____ Safety Glasses	_____ Ear Plugs
_____ Rubber Gloves	_____ Respirator
_____ Laboratory Coat	_____ Other (Specify)

   Date Issued _____ Employees Initials _____

4. SAFETY AND HEALTH TRAINING REQUIRED

_____ Right To Know	_____ Confined Space Entry
_____ Respirator Protection	_____ Hazardous Waste Operation
_____ Asbestos	_____ Manual Review (Indicate Applicable Sections)

5. PHYSICALS REQUIRED

_____ Asbestos	_____ Hazardous Waste Operation
_____ Respirator	_____ Other (Specify)

Employees Signature _____ Safety Officer's Signature _____

Date _____ Date _____

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

HEALTH AND SAFETY ORIENTATION TOUR for_____

1. Building Tour: Layout of office areas and Laboratories.
2. Location of Health and Safety Manual: Review Chapter Headings.
3. First Aid Station: Mandatory Injury Reporting.
4. Safety Glasses Policy:
   a. Arrange for temporary safety glasses.
   b. Initiate paperwork for ordering safety glasses.
5. Location of lab coats and policy on use.
6. Safety features of each room/areas.
   a. Fire extinguishers.
   b. Emergency eyewash stations.
   c. Emergency showers.
   d. Emergency exits.
   e. Telephones: paging system 59; emergency 9-911.
   f. Chemical storage areas.
   g. Gas cylinders.
   h. Sample storage areas.
7. SDS Library: Located in the Safety Officer's room and online under SOP and internet.
8. Orientation to specific work station/area.
   a. Introduction to co-workers.
   b. Specific chemicals, handling procedures.
   c. Equipment operation.
   d. Waste disposal procedures.

Signature specifies that all items have been covered and the employee has been given the opportunity to ask questions.

Supervisor giving tour:

_____
Signature

_____
Date

New employee:

_____
Signature

_____
Date

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

SAFETY TRAINING ATTENDANCE RECORD

Topic of Training Session: _____
(attach a copy of the training session curriculum)

Instructor(s):	Location:	Date:	Time:	Length:

We are legally required to maintain records regarding our safety training activities. Please provide the information indicated below to document your attendance. Thank you.

Name *(Please Print)*	Department	Signature

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

SAFETY COMMITTEE MEETING DOCUMENTATION

Agenda	
Attendees	
Date/Time	
Issue Discussed	
Required Action and Schedule	
Responsible Party	
Issue Discussed	
Required Action and Schedule	
Responsible Party	
Issue Discussed	
Required Action and Schedule	
Responsible Party	
Issue Discussed	
Required Action and Schedule	
Responsible Party	
Issue Discussed	
Required Action and Schedule	
Responsible Party	
Issue Discussed	
Required Action and Schedule	
Responsible Party	
Issue Discussed	
Required Action and Schedule	
Responsible Party	

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

LABORATORY INSPECTION FORM

This form is used to ensure compliance with Cal/OSHA regulations that require documented periodic inspections of all work areas as part of an effective IIPP. However, completion of this self-inspection checklist and correction of any findings noted herein does not ensure that Cal/OSHA will not issue citations during an inspection. In addition, HEALTH AND SAFETY representatives will be periodically verifying that self-inspections have been documented as part of the Environmental Health & Safety Assessment program.

Lab Location	Date of Inspection
Print Supervisor's Name	Supervisor's Signature
Print Inspector's Name	Inspector's Signature

GENERAL HAZARDS

1. Are aisles, exits, and adjoining hallways maintained free of obstructions that would hinder emergency exiting?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)

Corrective Action: Remove obstructions from aisles, exits, and adjoining hallways. Contact Safety Officer if help is needed cleaning adjoining hallways.

Completion Date: _____.

2. Is there at least 18 inches (47 cm) of vertical clearance maintained between all stored items and the ceiling-mounted fire sprinklers? (If there are no sprinklers, measure to the ceiling itself.)

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)

Corrective Action: Remove stored items that do not meet the above criteria.
Completion Date: _____.

3. Is furniture and equipment over 4 feet tall bolted to the wall or otherwise secured?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)

Corrective Action: Contact your Safety Officer for assistance in installing seismic restraints.
Completion Date: _____.

4. Are laboratory requirements for the use of personal protective equipment (such as gloves and goggles) established and enforced?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)

Corrective Action: Establish written requirements for personal protective equipment. For work with hazardous chemicals, incorporate these in your Chemical Hygiene Plan.

Completion Date: _____.

5. Are sharps waste containers available for disposing of needles, blades, and other sharps, and have all laboratory personnel been instructed on proper sharps disposal?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)

Corrective Action: Purchase a sharps container if needed. Train all laboratory personnel to avoid bending, cutting, or re-capping syringe needles.
Completion Date: _____.

EMERGENCY EQUIPMENT

6. Are all eyewash and emergency shower stations free of obstructions which would prevent quick access by someone temporarily blinded by a chemical splash?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Remove all obstructions from emergency eyewashes and showers.
Completion Date: _____.

7. Are the emergency eyewashes for your laboratory tested (flushed) monthly, with the tests documented?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Assign a laboratory employee to test all emergency eyewashes monthly. Document each test in a logbook or on an attached tag. Inform the Facility Manager if repairs are needed.
Completion Date: _____.

FLAMMABLE MATERIALS

8. Does the total quantity of flammable liquids stored in the laboratory comply with each of the following?

- The number of one (1) gallon glass containers stored outside storage cabinets must not exceed ten (10).
- The number of one (1) gallon safety cans in use outside storage cabinets must not exceed ten (10). The number of two (2) gallon safety cans must not exceed five (5).
- Total quantity of flammable liquids stored in the laboratory must be less than 30 gallons.

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Reduce the quantity of stored flammable liquids to meet the above criteria. Consult with the Safety Officer if assistance is needed.
Completion Date: _____.

9. Are the doors on all flammables storage cabinets self-closing?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Consult with the Department Supervisor for assistance in ordering and installing self-closing mechanisms.
Completion Date: _____.

10. Are all refrigerators and freezers in the laboratory labeled with "NO FOOD"?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Consult with the Department Supervisor for assistance in determining if refrigerators are suitable for flammables storage, and to obtain labels.
Completion Date: _____.

HAZARDOUS CHEMICALS

11. Does your laboratory have a new revised or reviewed Chemical Hygiene Plan that has been completed or updated within the last year?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)

Corrective Action: Contact your Safety Officer if you need a Chemical Hygiene Plan template. Assign a Chemical Hygiene Officer for the laboratory and have them read, fill out, and follow the guidance in the template. Post the Plan by the telephone.
Completion Date: _____.

12. Has documented training on the Chemical Hygiene Plan been provided to all laboratory workers?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Ensure that all workers have read and signed the Plan. Consult with the Safety Officer with any questions.

Completion Date: _____.

13. Has a chemical inventory of all hazardous materials in the laboratory been completed or revised within the last year and forwarded to HEALTH AND SAFETY? Date of last Chemical Inventory _____.

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Complete and update the Chemical Inventory. . Consult with the Safety Officer if you need assistance in preparing an inventory.

Completion Date: _____.

14. Are food and beverages prohibited from laboratory work areas?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Remove food and beverage items and reinforce this rule.

Completion Date: _____.

CHEMICAL STORAGE

15. Are all laboratory countertops, floors, and equipment maintained free of spilled hazardous materials and removable chemical residues?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Clean up any spilled material. . Consult with the Safety Officer for assistance if needed.

Completion Date: _____.

16. Are chemical fume hoods kept uncluttered so that air flows properly (e.g. storage minimized and adequate usable work area provided)? Are chemical fume hood sashes in good condition, and used at the proper setting?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Train laboratory occupants to minimize hood clutter and place sashes to maintain good airflow and provide splash protection. Inform Facilities Manager for repairs.

Completion Date: _____.

17. Are all sinks labeled with "No Hazardous Chemicals" stickers, and are all laboratory personnel aware of the EMAX Drain Disposal Guidelines?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Consult with the Safety Officer for additional stickers.

Completion Date: _____.

18. Are all chemical containers kept closed when not in use, to avoid the evaporation of volatile materials?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Train all laboratory occupants to keep chemical containers closed when not in use.
Completion Date: _____.

19. Are all chemical containers clearly labeled to identify their contents and in good condition (not corroded or leaking)?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Label all chemical containers, replacing those that are corroded or leaking.

Completion Date: _____.

20. Are stored chemicals segregated by hazard class (acids separate from bases, oxidizers separate from flammables, etc.)?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Segregate chemicals by hazard class. Consult with the Safety Officer for assistance with chemical segregation.

Completion Date: _____.

21. Are one gallon (4-liter) and larger containers of hazardous chemicals stored in secondary containment?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Provide secondary containment such as chemically-resistant tubs or coated bottles.

Completion Date: _____.

22. Are corrosive chemicals stored below eye level to prevent accidental injury?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Relocate corrosives to a location below eye level.

Completion Date: _____.

23. Are peroxide formers (such as diethyl ether) stored away from light or heat, and labeled with the date they were opened and their expiration date?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Label all peroxide formers with the opening date and expiration date. These chemicals may become explosive after prolonged storage. If any of these chemicals are present and have not been used for a long time, do not handle them. Consult with the Safety Officer and ask for assistance.

Completion Date: _____.

ELECTRICAL EQUIPMENT

24. Are extension cords and power strips in good condition (e.g., no breaks or exposed wiring), used only as temporary wiring (<30 days), and not connected in series?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)

Corrective Action: Dispose of or repair all electrical cords that are not in good condition. Remove all daisy chained or permanent use power strips and extension cords. Inform the Facilities Manager for installation of permanent wiring.
Completion Date: _____.

25. Is high voltage equipment clearly labeled, properly guarded, and is its use restricted to only trained personnel?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Label all high voltage equipment with appropriate warnings. Restrict use of this equipment to properly trained personnel.
Completion Date: _____.

OTHER EQUIPMENT

26. Are all compressed gas cylinders adequately secured with non-combustible restraints to keep the cylinder(s) from falling during an earthquake? (If chains are used, two should be present for each cylinder.) Also, are all compressed gas cylinders capped when not in use?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Inform Facilities Manger for assistance in installing gas cylinder restraints. Bench clamps are not allowed. In addition, train laboratory occupants to cap compressed gas cylinders when not in use.
Completion Date: _____.

27. Are all refrigerators and microwave ovens properly labeled either "food only" or "no food"?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Label all refrigerators and microwave ovens as "food only" or "no food".
Completion Date: _____.

OTHER HAZARDS

28. List any other hazardous conditions in need of correction that are not mentioned in this checklist. Assign and document correction of each item listed below.

1. _____

2. _____

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

GENERAL INSPECTION FORM

Office Location	Date of Inspection
Print Supervisor's Name	Supervisor's Signature
Print Inspector's Name	Inspector's Signature

1. Is the Cal/OSHA poster "Safety and Health Protection on the Job" displayed in the building in an area accessible to all employees?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Contact Health and Safety Officer to obtain posters.
Completion Date: _____.

2. Is documentation of safety training, workplace self-inspections, and hazard corrections maintained and accessible where indicated in your department's IIPP?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Confirm location listed in the IIPP and ensure that records are stored there.
Completion Date: _____.

3. Have employees in the area been trained on the applicable Building Emergency Plan (BEP)?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)

Corrective Action: Contact the Safety Officer to obtain the BEP, or contact your Building Coordinator if a BEP is not available.

Completion Date: _____.

4. Are evacuation diagrams posted?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Contact the Department Safety Coordinator, Building Coordinator, or HEALTH AND SAFETY for assistance in preparing diagrams as required by the BEP.

Completion Date: _____.

5. Are fire alarm pull boxes clearly identifiable and unobstructed?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Clear area of obstructions.
Completion Date: _____.

6. Are fire hose stations and/or portable extinguishers clearly identifiable and unobstructed?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Label fire-fighting equipment and clear area of obstructions.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

Completion Date: _____.

7. Are fire extinguishers tagged with current annual inspections?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Call 800-841-9696 to arrange for a fire extinguisher inspection by H&P Fire Extinguisher Co. Ensure that the extinguisher is properly tagged after the inspection.

Completion Date: _____.

8. Do self-closing devices and door latches on fire-rated doors work freely? (Doorstops are not permitted.)

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Contact your Safety Officer to arrange for door repairs.
Completion Date: _____.

9. Are there at least 18 inches (47 cm) of vertical clearance maintained between all stored items and any ceiling equipped with fire sprinklers?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Remove stored items that do not meet the above criteria.

Completion Date: _____.

10. Are electrical panels accessible and circuit breakers clearly identified?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Label circuit breakers as to their function, and clear area in front of electrical panels by 36 inches.

Completion Date: _____.

11. Are aisles, exits, and adjoining hallways maintained free of obstructions so that the area can be easily evacuated or accessed in case of an emergency?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Remove obstructions from aisles, exits, and adjoining hallways. Contact Safety Officer if help is needed cleaning adjoining hallways.

Completion Date: _____.

12. Are electrical equipment (e.g., copiers and computers) grounded? (Ensure that the grounding prong has not been removed, and that 3-prong to 2-prong adapters are not used.)

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Contact your supervisor or Safety Officer to arrange for installation of appropriate outlets and plugs.

Completion Date: _____.

13. Are extension cords in good condition (e.g., no breaks or exposed wiring), used only as temporary wiring (less than 30 days), and not connected in series?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

Corrective Action: Do not connect extension cords in series. Dispose of or repair all electrical cords that are not in good condition, and replace those in use more than 30 days with permanent wiring.

Completion Date: _____.

14. Is broken, unguarded, or otherwise dangerous equipment or furniture present? (Example: A paper cutter without a guard to keep fingers away from the blade.)

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Contact your supervisor or Safety Officer to arrange for removal or repair of equipment or furniture.

Completion Date: _____.

15. Are floor surfaces kept dry and/or made slip-resistant?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Work with your supervisor or Safety Officer or Safety Committee to address this issue.

Completion Date: _____.

16. Is furniture and equipment over 4 feet tall braced to prevent tipping in an earthquake?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Contact your supervisor or Safety Officer for assistance in installing seismic restraints, or remove all of items in question.

Completion Date: _____.

17. Are all work areas adequately illuminated?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Contact your supervisor or Safety Officer for assistance in obtaining additional lighting.

Completion Date: _____.

18. Have computer workstations been ergonomically evaluated for all employees who spend 4 or more hours at their computer each day?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Contact your supervisor or Safety Officer to have a trained workstation evaluator assess the workstation. If you department does not have an evaluator, contact the Safety Officer.

Completion Date: _____.

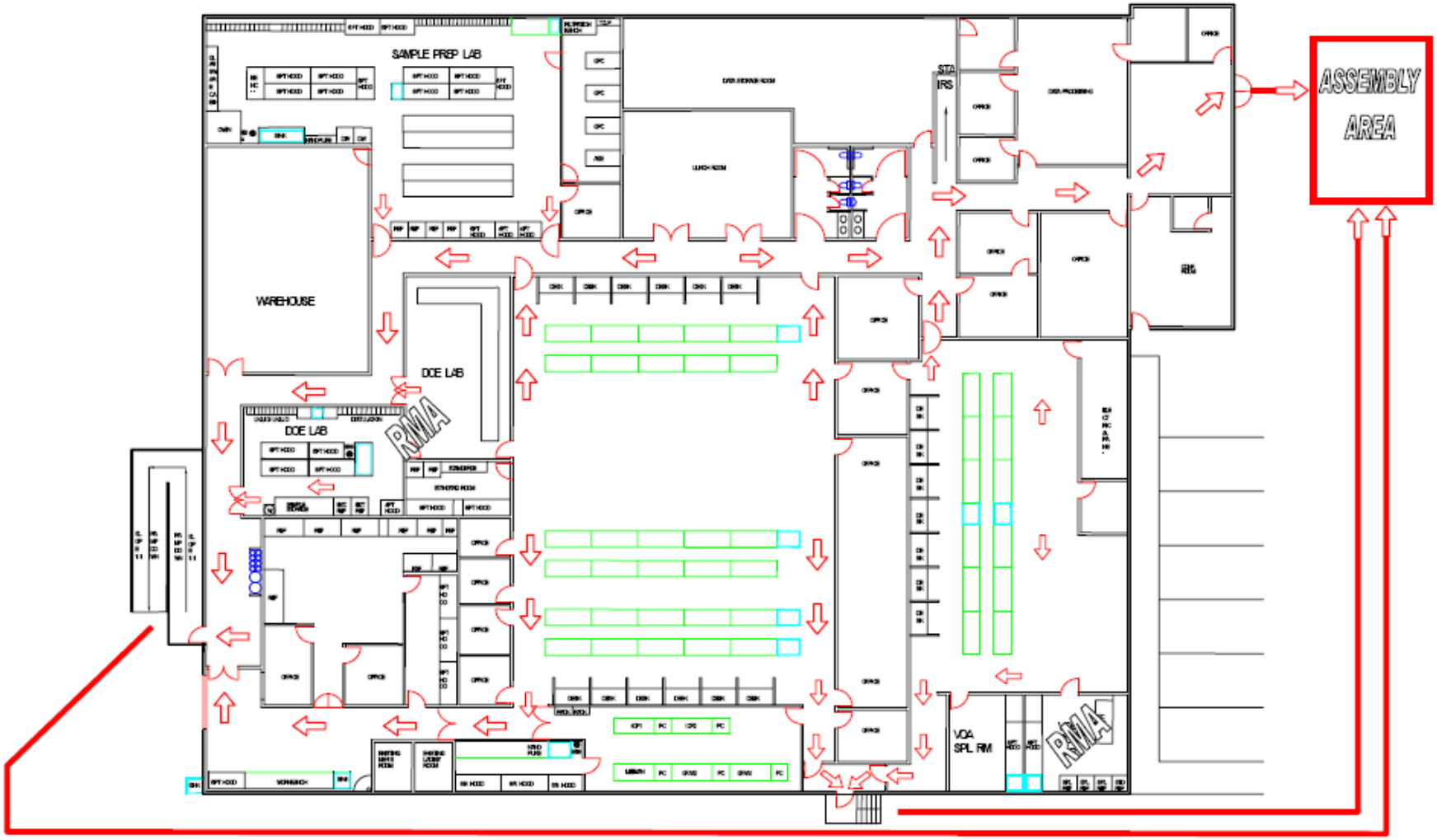
19. Are emergency back up lights in proper working conditions?

☐ Yes (Satisfactory) ☐ No (Needs Correction) ☐ N/A (Not Applicable)
Corrective Action: Contact your supervisor or Safety Officer to have a trained workstation evaluator assess the workstation. If you department does not have an evaluator, contact the Safety Officer.

Completion Date: _____.

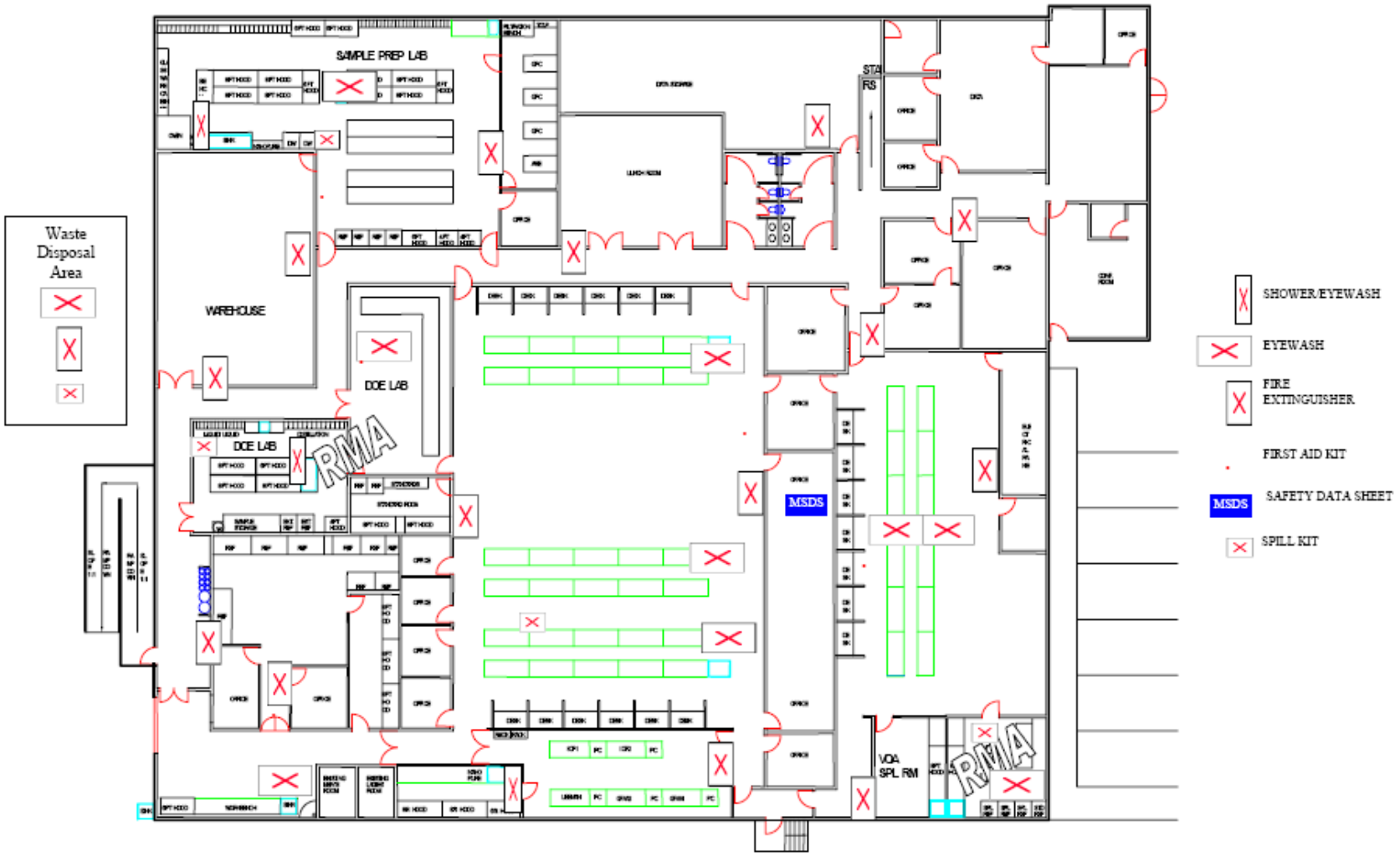
EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

EMERGENCY EVACUATION ROUTE



EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

LOCATION OF SAFETY MATERIAL AND EQUIPMENT



RMA – Radioactive Material Area

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

Approved By	Richard Beauvil, Safety Officer	8/26/19 Date
Approved By	Caspar Pang, President	5/26/2019 Date

Document ID	EMAX-RTK-05
Revision No.	5
Date Released	8/26/19
Issue No.	EMAX-RTK-05-
Issued To	

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

RIGHT-TO-KNOW/HAZARD COMMUNICATION MANUAL

Table of Contents

		Pages
1.0	Right To Know Law/Hazard Communication Standard	2
2.0	Hazardous Substances Overview	3
2.1	Introduction	3
2.2	Hazard Recognition	3
2.3	Hazard Control	4
3.0	Safety Aspects of Hazardous Substances	5
4.0	Basic Precautions	6
5.0	Emergency Procedures	7
6.0	How To Read A Safety Data Sheet (SDS)	8
7.0	The Labeling System	10
7.1	The Labeling System	10
8.0	Corrosives/irritants	11
9.0	Gases	12
10.0	Mercury	13
11.0	Paints, Inks, and Dyes	14
11.1	Hazards	14
11.2	Precautions/Control Measures	15
12.0	Particles	16
13.0	Solvents/Oils	18
13.1	Health Effects	18
13.2	Precautions/Control Measures	19
14.0	Heat	20
15.0	Noise	22
16.0	Glossary	23
17.0	Understanding a Safety Data Sheet	26

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

1.0 RIGHT TO KNOW LAW/HAZARD COMMUNICATION STANDARD

The information and training presented in this program is designed to inform you about the potential work-related hazards in order to promote a safe and healthy work environment for all employees.

As a result of this training, each employee should know how to recognize and help control hazards present in the work environment. You should know what questions to ask, whom to ask, where to find important health and safety information, and how to apply the information to your own jobs, work areas, and work practices.

The following is a summary of the provisions contained in the Right to Know Law:

1. Annual training must be provided to all employees who are assigned to jobs in which they may be routinely exposed to hazardous substances (chemicals) and harmful physical agents (noise, heat).

2. Written information about hazardous substances and harmful physical agents must be made available.

3. All containers of hazardous substances, other than "immediate use" containers, must be labeled.

4. Records must be kept of all training sessions held.

Be sure and retain your training manual for future reference.

2.0 HAZARDOUS SUBSTANCES OVERVIEW

2.1 Introduction

Hazardous substances are present in every work environment and in every home. As you know, many substances are used every day without causing harm. But, exposure to too much of any substance or misuse of a substance may be harmful.

2.2 Hazard Recognition

This is the first step in protecting yourself. Hazard is the probability that a substance will harm you. Hazard depends on how the substance is used, and how you come in contact with it. Toxic properties relate to the ability of a substance to produce injury or harm once it reaches the body.

Some substances are also safety hazards if they can easily Catch fire, explode or react with other substances. Whenever they are in use or stored, there is a greater chance of an accident occurring.

The frequency and duration of exposure to chemicals also affects the hazard potential. You may work with a chemical every day for eight hours in your job. At home, you may use the same product very infrequently for only a short period of time.

It is important to remember that the amount of hazard presented by a substance is dependent upon:

1. the substance itself;
2. the amount that you are exposed to (concentration); and
3. how long you are exposed (time).

Remember: Hazard is not the same as toxicity. Toxic substances can be handled in a safe manner, if the proper precautions are taken and the substance is treated with a measure of respect.

The major **ROUTES OF EXPOSURE** of hazardous substances into the body are:

1. skin and eye contact/absorption;
2. inhalation (breathing); and
3. ingestion (swallowing)

Note: Exposures can occur through more than one route with the same chemical.

Exposure Limits: PELs (Permissible Exposure Limits - OSHA) and TLVs (Threshold Limit values - guidelines) are established. They are set at a level that is safe for the majority of persons working with the substance 8 hours a day, 40 hours a week, for a working lifetime.

2.3 Hazard Control

When you use chemicals in your home or at work, you can protect yourself by taking some simple steps. Read the labels and pay attention to warnings and recommendations. If you use chemicals with sufficient information, common sense, and respect, you can protect yourself and others.

Controlling hazardous substances in the workplace is especially important, since the risks associated with using or producing chemicals are usually greater at work than at home. This increased risk is the result of exposure to a greater number and higher concentrations of chemicals, for a longer period of time in the workplace, as compared to the home.

Employers are responsible for providing a safe and healthy work environment. This includes providing effective methods to control potentially hazardous exposures. Employees are responsible for following the proper procedures of handling and using hazardous substances and harmful physical agents.

3.0 SAFETY ASPECTS OF HAZARDOUS SUBSTANCES

Hazardous substances in the workplace can also be safety hazards, depending on certain properties that determine how likely they are to burn, explode, or react with other materials. It is important to understand these properties so that you can safely use, store, transport, and dispose of hazardous materials.

FLAMMABLE MATERIALS include any solid, liquid, vapor or gas that ignite at temperatures below 100°F, and burn quickly. (Xylene)

COMBUSTIBLE MATERIALS include any solid that is difficult to ignite and that it burns relatively slowly; or liquids that have a flashpoint above 100°F. (Mineral spirits)

EXPLOSIONS occur when flammable vapors or gases (such as hydrogen) or combustible dusts (such as grain) accumulate in a poorly ventilated area and are set on fire by a source of ignition.

OXIDIZING AGENTS give off oxygen at room temperature or upon slight heating. Contamination with dirt, other chemicals, moisture, etc. may start a chemical reaction generating heat and other gases. Some oxidizers may have a corrosive effect on body tissues. (Hydrogen peroxide)

REACTIVE CHEMICALS are substances that will easily react with other chemicals. The reaction can result in the production or absorption of heat, or a fire.

STABLE CHEMICALS have the ability to remain unchanged under expected and normal conditions of storage and use. Some materials such as explosives are **UNSTABLE.**

INCOMPATIBLE MATERIALS should never be mixed. Direct contact with one another could cause a dangerous reaction. (acids and bases)

4.0 BASIC PRECAUTIONS

You can protect your health and safety on the job and at home; and you can prevent accidental exposures to hazardous materials by observing these basic precautions:

1. Always use common sense and safe work practices.
2. Know what chemicals you are handling and working with and the potential hazards.
3. Use personal protective equipment and clothing when appropriate.
4. Observe all safety procedures - they are for your protection.
5. Do not eat, smoke, or drink while you are working with hazardous materials.
6. Where applicable, know the location of the eye wash fountains and safety showers in your work area in case of an emergency.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

5.0 EMERGENCY PROCEDURES

In the event of an emergency involving contact with or overexposure to a hazardous substance:

IMMEDIATELY NOTIFY YOUR SUPERVISOR AND SUMMON MEDICAL ASSISTANCE

Be prepared to identify the chemical involved in the emergency.

BASIC FIRST AID PROCEDURES (while someone else is summoning medical assistance):

1. Chemical Contact - Eyes:

   IMMEDIATELY flush eyes with large amount of water for at least 15 minutes, while holding the eyes open for complete cleaning.

2. Chemical Contact - Skin:

   IMMEDIATELY wash the area with water for at least 15 minutes; do not scrub. Remove contaminated clothing.

3. Chemical Contact - Ingestion:

   If a person accidentally swallows a chemical agent, **IMMEDIATELY** seek emergency medical help and/or call the local poison information center (911) or a physician.

4. Chemical Contact - Inhalation:

   If a person is overcome by breathing airborne chemicals **IMMEDIATELY** move the person to fresh air. Restore breathing if necessary.

In ALL cases, seek medical attention and report the incident.

6.0 HOW TO READ A SAFETY DATA SHEET (SDS)

The following is a short description of the information contained on a Safety Data Sheet (SDS), and how to read it and use this information.
SDS's are accessed by the product trade name which can be found on the container label. To promote consistent presentation of information, OSHA recommends that SDS follow the16-section established by the Globally Harmonized System (GHS).

Section 1, Identification includes product identifier; manufacturer or distributor name, address, phone number; emergency phone number; recommended use; restrictions on use.

Section 2, Hazard(s) identification includes all hazards regarding the chemical; required label elements.

Section 3, Composition/information on ingredients includes information on chemical ingredients; trade secret claims.

Section 4, First-aid measures includes important symptoms/ effects, acute, delayed; required treatment.

Section 5, Fire-fighting measures lists suitable extinguishing techniques, equipment; chemical hazards from fire.

Section 6, Accidental release measures lists emergency procedures; protective equipment; proper methods of containment and cleanup.

Section 7, Handling and storage lists precautions for safe handling and storage, including incompatibilities.

Section 8, Exposure controls/personal protection lists OSHA's Permissible Exposure Limits (PELs); Threshold Limit Values (TLVs); appropriate engineering controls; personal protective equipment (PPE).

Section 9, Physical and chemical properties lists the chemical's characteristics.

Section 10, Stability and reactivity lists chemical stability and possibility of hazardous reactions.

Section 11, Toxicological information includes routes of exposure; related symptoms, acute and chronic effects; numerical measures of toxicity.

Section 12, Ecological information*, Assists you in evaluating the effect a chemical may have if it's released to the environment.

Section 13, Disposal considerations*, Provides proper disposal information for environmental professionals or individuals responsible for waste management activities.

Section 14, Transport information*, Provides shipping classification information for the employer, distributor, emergency responders, and transport/shipping departments.

Section 15, Regulatory information*, Provides regulatory information for employers and regulatory compliance personnel.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

Section 16, Other information, includes the date of preparation or last revision.

*Note: Since other Agencies regulate this information, OSHA will not be enforcing Sections 12 through 15(29 CFR 1910.1200(g)(2)).

Employers must ensure that SDSs are readily accessible to employees.
See Appendix D of 1910.1200 for a detailed description of SDS contents.
For more information: www.osha.gov

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

7.0 LABELING

Read all labels, on all containers

1. before you move them
2. before you open them
3. before you use the contents

Use of an unlabeled container is acceptable only when that container is intended for immediate use!

The label is your key to identifying the chemicals you work with.

7.1 The Labeling System

A. Manufacturer's Labels

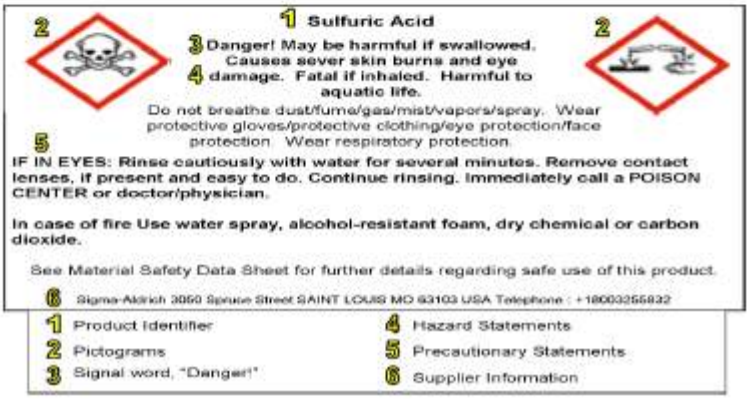
These labels are found on the original shipping containers. Read these labels carefully.

All containers of hazardous chemicals must be labeled with the following information:

The GHS standard requires that there be six label elements:

- Product identifier or ingredient disclosure; (Chemical Name, code number, or batch number)
- Signal Word; (Danger and Warning)
- Pictograms: (Very specific)
- Hazard statement; (Nature of the chemical and the degree of hazard)
- Precautionary statement; and (recommended measures to minimize or prevent adverse effect from exposure)
- Supplier identification. (Name, address, phone number)

Label Must be cross-referenced with the SDSs and the chemical inventory entry. Primary and Secondary containers must have the new label.



EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

B. D.O.T. Labels.

These are picture labels with a word designation. This type of label gives you at-a-glance information about the properties of the material (for example, flammable or corrosive).

C. Waste Management's Labels

Labels will be placed on all containers (other than immediate use*) that hazardous materials are transferred into. These labels will contain the product's trade name and a hazard warning. (A copy of the original shipping container label can be used.)

The **TRADE NAME** should be used to obtain further information about a particular chemical. Safety Data Sheets will be listed alphabetically by Trade Name.

D. Immediate Use labels

An "Immediate Use" container is a container into which substances are transferred from labeled containers and is used by the employee who performs the transfer.

8.0 CORROSIVES/IRRITANTS

These chemicals can irritate, burn or destroy living tissue such as skin and mucous membranes. Contact with corrosives such as sodium hydroxide generally produce severe damage, such as burns. Irritants cause tissue inflammation as opposed to tissue destruction. Corrosives/irritants include acids and bases (alkalies, caustics) as well as other chemicals. The health effect depends on the chemical, the solution strength, and the length of time it is left in contact with the skin.

Exposure to irritants and corrosives usually occurs when they are spilled or splashed on the skin or in the eyes. When airborne, through heating or spraying, certain chemicals may be irritating to your nose and throat when inhaled. Some corrosives/irritants are in powder form.

Protective clothing that should be worn includes:

1. chemical-resistant gloves (length dependent on type of operation)
2. chemical goggles or faceshield, during pouring or mixing if there is a chance of splashing
3. apron or other garment dependent on the extent of exposure (splash 3. potential)
4. chemical-resistant shoes, as necessary.

CORROSIVE/IRRITANT CHEMICALS include: General Purpose Cleaner (1 57), PW901, Power Wash, sodium hydroxide (caustic soda), ammonia, soda ash, sulfuric acid (battery acid), boiler compounds, water treatment chemicals, cleaning chemicals, bleach, muriatic acid (hydrochloric acid), nitric acid, acetic acid, and disinfectants and germicides

Remember: When mixing acid or bases and water, always add the acid or base to water. Never directly mix acids and bases, since a violent reaction can occur upon mixing.

Refer to the Safety Data Sheet and the product label for important health and safety information on each specific substance.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

9.0 GASES

A material is called a gas if its typical physical state at normal temperature and pressure. Substances that are normally a solid or liquid at room temperature can change their physical state to a gas, upon heating, burning or decreasing pressure.

Most gases are colorless at low concentrations and cannot be seen. Some gases (i.e. ammonia and chlorine) can be detected by their odor and irritating effects at certain concentrations. Gases such as carbon monoxide, have no odor or immediately irritating effect on the body. In general, gases can be inhaled directly into the lungs, where they are either absorbed into the blood or exhaled. Some gases will immediately irritate the respiratory system.

Gases are used directly in some work processes, or they can occur as by-products from work processes or operations, such as carbon monoxide from fuel-operated fork lift trucks. Gases may also be contained in pressurized cylinders such as compressed gases (acetylene, oxygen and propane).

ASPHYXIANTS: These gases interfere with the body's ability to utilize oxygen. Simple asphyxiants are gases that in a normal atmosphere are safe, but in confined or poorly ventilated spaces can accumulate. This displaces the normal amount of oxygen we depend upon to sustain life. Methane, argon, helium, and nitrogen are examples of simple asphyxiants. Chemical asphyxiants, interfere with the transportation and use of oxygen by the tissues in the body. Overexposure to chemical asphyxiants such as carbon monoxide and hydrogen cyanide can also result in suffocation. Initial symptoms include: headache, dizziness, and nausea. Symptoms will usually subside upon ceasing exposure to the asphyxiant gas.

COMPRESSED GASES: Containers of compressed gas should be handled very carefully. Cylinders should be stored and secured in an upright position in a well-protected and ventilated area. Cylinders should always be capped during use and transport. Oxygen and chlorine cylinders should be stored separately from fuel gas cylinders. If a leak occurs due to a faulty valve or piping, stop the gas flow, if it can be done safely. If not, evacuate the area, and get assistance. Other commonly used compressed gases include propane (flammable), nitrogen, ammonia, argon and acetylene.

VEHICLE EXHAUST EMISSIONS: Vehicle exhaust emissions consist of carbon monoxide and other gases, particulate (including lead) and aldehydes. Carbon monoxide is produced in higher concentrations from the combustion of gasoline, as compared to diesel fuel. Diesel fuel tends to produce more of the irritating particulate and gases.

The most common irritants that can be found in vehicle exhaust emissions include oxides of nitrogen and aldehydes. In high concentrations, they may irritate the nose and respiratory tract.

10.0 MERCURY

Mercury, a heavy metal, has the unique property of being a liquid at room temperature. Mercury is highly toxic and exposure should be kept to a minimum. Emotional and psychic disturbances, as well as digestive disorders and liver damage are characteristic of chronic mercury poisoning. Symptoms include: headache, dizziness, Irritability, tremors of the hand, head, lips, or jaw, salivation, inflammation of the gums, nausea, vomiting and diarrhea.

Inhalation is the main route of exposure, but mercury can also be absorbed through skin contact or ingestion.

1. Good housekeeping is essential. All spills must be cleaned up immediately and thoroughly. Employees must be trained on proper spill clean-up procedures. To prevent mercury build-up in cracks and crevices due to spills, laboratory work surfaces should be smooth.

2. Mercury, including waste material, must be properly stored in airtight, nonmetallic containers.

3. Adequate laboratory ventilation will minimize exposure to mercury vapors.

4. Proper work procedures, use of personal protective equipment as needed, and good personal hygiene is very important when working with mercury.

11.0 PAINTS, INKS AND DYES

HAZARD POTENTIAL is dependent upon the following:

- Method of Application:
  hand
  mechanical dipping
  spraying

- Type of Paint, Ink or Dye:
  oil base
  water base
  polyurethane
  epoxy

- Control Measures Present
  local exhaust
  personal protective equipment
  spray booths

- Type of pigment or base in the paint, ink or dye.
  lead chromate
  titanium dioxide
  Cadmium
  aniline

- Type of Solvents
  used in the paint, ink or dye
  used in the department
  Examples: xylene, toluene, and naptha

11.1 Hazards

1. Dermatitis - Dermatitis may occur from either excessive skin contact with solvents or occasionally from the paint, ink or dye itself.

2. Solvent Vapor Inhalation - General symptoms include dizziness, headache, nausea, and eye and nose irritation. See the section on solvents for further information.

3. Inhalation of Paint and Ink Pigments - Some paints and inks contain toxic heavy metals such as lead, zinc chromates and cadmium. Long term health effects from overexposure may include lung damage from chromate and cadmium compounds. And possibly damage to the bloodforming, nervous and reproductive systems from lead.

4. Sensitization - Urethane and epoxy paints cause respiratory and skin sensitization; and allergic reaction. The allergy develops over a period of time of exposure to the vapors or the liquid.

5. Aniline compounds can be absorbed through the skin and overexposure

may result in cyanosis. Chronic overexposure may result in liver or kidney damage.

11.2 Precautions/Control Measures

1. Wear protective clothing (i.e., gloves, barrier cream or coveralls) as required or as needed to prevent prolonged skin contact with paints. Avoid skin contact with aniline-based dyes.

2. Wear a respirator as needed. Make sure the appropriate cartridges are used; organic vapor cartridges to reduce exposure to the solvent vapors. May also be used in combination with a paint pre-filter.

3. Do not remove paint from your skin using solvents. Use a waterless skin cleaner or soap and water.

4. Good personal hygiene is very important.

12.0 PARTICLES

Some chemicals are present as particles in the air or on surfaces. Particle types include: dust, fume, smoke, mist, and fog.

Dusts, fumes and smoke consists of solid particles. Dusts are produced by sanding, grinding, crushing, and handling powdered materials, etc. Fumes are produced when metals are melted during welding and foundry operations. Smoke usually is the result of heating or burning a material.

Mists and fogs consists of liquid particles. Mists are liquid droplets that are produced by breaking up a liquid such as in spraying. Fogs are formed when humid air cools and the water vapor condenses.

Particles are easily released into the air and can settle onto exposed work surfaces. If the particles remain in the air, you can inhale them. Depending on their size, they may get trapped in your nose, or if they are small enough, they can reach your lungs.

If solid particles settle out of the air, like dust in your home, they can contaminate the floor and work surfaces. If the particles are not cleaned up, they can be made airborne again. If you touch surfaces that are contaminated with toxic dusts, you could transfer the particles from your hands or clothing to your mouth.

Exposure to particles can result in a variety of symptoms ranging from irritation to lung damage; again depending on the concentration, length of exposure and the toxicity of the material.

ASBESTOS: Inhalation of excess asbestos fibers can result in asbestosis. Symptoms of asbestosis include chest pain, breathing difficulty, dry sound during Breathing, and weight loss. Lung cancer and mesotheliorna (cancer of the chest lining) may also result from excessive asbestos inhalation. Smoking enhances the lung cancer causing properties of asbestos. Special procedures must be followed when working with asbestos to prevent inhalation of the fibers.

BRAZING AND SOLDERING: When brazing or soldering, the exposure depends on the base metal, the filler metal composition, the temperature and the type of flux. Thermal decomposition products from fluxes may be irritating to the eyes, nose and throat. There is also a potential for exposure to lead, silver and cadmium and other metal fumes. These fumes have the potential to cause respiratory irritation, as well as long term health effects such as lung damage or neurological disorders, with chronic overexposure. Some fluxes are also skin irritants. *Metal Fume Fever - Overexposure to metal fumes (commonly zinc or copper) may produce this effect. The symptoms are similar to the flu and usually occur a few hours after the exposure.

LEAD: Sources of lead include paint pigment, automobile exhaust, gasoline additives, and welding and soldering. Lead poisoning may occur through inhalation or accidental ingestion. The symptoms of chronic overexposure include: loss of appetite, anemia, and muscle and joint pain. The liver, kidneys, and reproductive systems may also be affected.

METAL DUST AND FUMES: Iron oxide is the major metal fume generated from

heating mild steel. Iron/Iron oxide and Aluminum/Aluminum oxide, as well as other metal dusts, may cause respiratory irritation. Metal work on parts containing toxic metals such as Nickel, Chromium, Copper, Cadmium or Beryllium increase the health hazard potential. Follow the safety procedures established for welding or soldering. Read and follow all warning labels. Use all required protective equipment. (See section on welding, if applicable).

NUISANCE DUST: This includes plastic, dirt, 'house dust', etc. At high concentrations, dusts may cause respiratory irritation in some individuals. In general, nuisance dusts are so named because they do not have a toxic effect on the body.

WOOD DUST: At high concentrations, wood dust can act as a respiratory and eye irritant. Certain woods, including pine, beech and mahogany can cause an allergenic response (asthma and dermatitis) in sensitized individuals. Chronic overexposure to certain hardwood dusts is suspected of causing nasal cancer.

13.0 SOLVENTS/OILS

SOLVENTS are a class of liquids that are used to dissolve certain other solid materials (clean, degrease, thin). They will be found in paints, inks, dyes, thinners, cleaners, resins and adhesives. Petroleum based oils are also included in this section due to their similar characteristics. Solvents and oils used at EMAX Laboratories, Inc. include some of the following:

a. Acetates (cellosolve acetate, butyl acetate, ethyl acetate)
b. Alcohols (isopropyl alcohol, butyl alcohol, ethyl alcohol)
c. Aldehydes (formaldehyde, acetaldehyde)
d. Aliphatic hydrocarbons (hexane, nitropropane)
e. Aromatic hydrocarbons (styrene, xylene, phenol, toluene)
f. Chlorinated hydrocarbons (trichloroethane, trichloroethylene, methylene chloride, perchloroethylene, chloroform, carbon tetrachloride)
g. Esters (butyl cellosolve, methyl cellosolve, ethoxy ethanol)
h. Ethers (diethyl ether)
i. Fluorinated hydrocarbons (freons, trichlorotrifluoromethane)
j. Ketones (methyl ethyl ketone, methyl isobutyl ketone, acetone)
k. Mixtures (stoddard solvent, petroleum distillates, naphtha, mineral spirits, kerosene, turpentine, gasoline, laquer thinner, oils)

Properties of Solvents:

a. Many solvents are flammable.
b. In general, solvents evaporate quickly.
c. With heating, evaporation rate is quicker.
d. Upon exposure to flames, chlorinated hydrocarbons can decompose generating hydrogen chloride and phosgene.

13.1 Health Effects

1. **SKIN DISORDERS.** Repeated skin contact can cause a rash or irritation (dermatitis), as well as dry out the skin to the point of cracking and bleeding. Solvents should not be used to wash or clean the skin. Some solvents can be absorbed both through contact, as well as open skin. Particular attention should be paid to contact with these materials. This information will be noted on the Safety Data Sheet. Oils tend to block skin pores upon prolonged exposure and may cause inflammation (folliculitis).

2. **EYE INJURY:** Solvent vapors can irritate the eyes. Directly splashing the liquid into eyes may cause serious burns, and possible permanent damage.

3. **INHALATION:** Solvents act as central nervous system depressants. General symptoms include: headache, dizziness, nausea. Generally, when the exposure is stopped, the reactions will disappear. Extreme overexposure situations could lead to unconsciousness. Solvent vapors can also act as respiratory irritants. Chronic overexposure to some solvents, such as trichloroethylene and carbon tetrachloride, may cause permanent injury to the liver or kidneys, or other internal systems.

Certain 'cellosolve' products such as cellosolve acetate, cellosolve (ethoxy ethanol), and methyl cellosolve (methoxy ethanol) may cause reproductive disorders with chronic overexposure. Chloroform, carbon tetrachloride and formaldehyde are suspect carcinogens.

Inhalation of oil would occur as oil mist from splashing or as vapors/mist (similar to the above) upon heating. Oil mist/vapors can be irritating.

13.2 Precautions/Control Measures

1. Wear solvent resistant gloves to prevent repeated or prolonged skin contact.

2. In operations where the liquid could splash into the eyes, chemical goggles or a face shield should be worn. In case of eye contact, flush the eyes with water for at least 15 minutes, while holding the eyelids open.

3. In operations where the liquid could splash onto the skin, appropriate protective clothing, such as a solvent-resistant apron, should be worn.

4. If extensive skin contact should occur, flush the area thoroughly for at least 15 minutes.

5. Personal hygiene is very important, Wash thoroughly. Apply hand cream as needed to prevent drying. Do not wash up with solvents, use soap and water or a waterless skin cleaner.

6. In situations where a respirator is required, be sure the proper respirator is worn. It should protect you against organic vapors for solvents, and dusts/mists for oil mist.

7. In Case of a solvent spill or leak, avoid contact with the material, wear an organic vapor respirator to prevent overexposure during cleanup operations. Attempt to stop the spread of the material. Remove all ignition sources.

14.0 HEAT

Heat is another physical agent to which you can be exposed. Your body internally produces heat as a result of metabolic activity. When the body is exposed to excessive external heat, the natural response is sweating: the body's method for cooling down.
The amount of sweat loss depends on the environmental conditions, the length of exposure, and the degree of activity.

The effects of overexposure to heat (heat stress) can range from a mild reaction, such as heat exhaustion, to a very serious reaction such as heat stroke. Heat stroke is a serious medical condition that must be treated immediately and correctly, or it can result in death or permanent damage. Emergency medical assistance should be called for immediately.

Preventing Heat Stress at Work and at Home:

1. Gradual exposure over a period of time allows the body to get used to higher environmental temperatures. This process is called acclimatization.

2. During periods of extreme heat and humidity, adjusting the work-rest cycle may be necessary. For example, increasing the frequency of rest breaks.

3. Fans are helpful in indoor situations, except in extreme heat.

4. Drink plenty of fluids throughout the day, not just during breaks. Do not depend on thirst to signal that the body needs fluids.

5. Replace salt lost by heavy sweating by: (a) lightly salting food at meals; (b) eating a salty snack; or (c) drinking electrolyte replacement fluids (such as Gatorade) in small quantities.

6. Avoid the use of salt tablets. Too much salt without adequate water may cause nausea and vomiting.

7. The most strenuous tasks should be performed during cooler parts of the day, when possible.

Health Problems Associated with Excessive Heat

1. Heat Cramps are painful muscle spasms that occur when an individual sweats profusely, drinks large quantities of water, but fails to replace the body salts lost during the sweating process. The low salt concentration in the muscle results in cramps. Muscles used in performing work (in particular the arms, legs, and abdomen) are usually the most susceptible.

   Treatment: Move victim to a cool area. Apply manual pressure to the spasm. Give salt water or other sodium replacement fluid. Caution: Persons with heart problems or those on a low sodium diet must consult a physician regarding what to do under these conditions.

2. Heat Exhaustion is caused by dehydration due to excessive sweating. It results in general weakness, dizziness, nausea, and possibly fainting. The victim continues

sweating. The skin is clammy and moist; the complexion is pale or flushed; and the body temperature remains normal. Persons who are unacclimatized, physically unfit, or obese are more prone to experience heat exhaustion.

Treatment: Seek medical assistance. Move victim to a cool area. Lower the head. Loosen any restrictive clothing. Give water if victim is conscious.

3. Heatstroke is the most serious health problem from exposure to excessive heat. Sweating stops, leaving the body with no means to get rid of excess heat. A heatstroke victim's skin is hot, dry, and usually red or spotted. The body temperature rises to dangerous levels of 105 degrees or higher. The person is mentally confused, irritable and may complain of feeling chilled. Heatstroke can result in convulsions, coma, and possibly death.

   Treatment: Seek medical assistance. Move victim to a cool area. Elevate the legs. If the victim is unconscious, maintain an open airway. Remove as much clothing as possible. Soak the person with cold water. Use a fan or other method to assist in cooling down the body. Do not give anything by mouth. Prompt and Proper treatment is essential!

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

15.0 NOISE

Noise is defined as unwanted sound. Exposure to loud noise can result in different types of hearing loss. Temporary hearing loss can result from exposure to loud noise for a short period of time. Your ears usually recover from this type of loss if given adequate time. With chronic exposure to excessive noise levels, your ears may lose their ability to recover from the hearing loss, and it gradually becomes permanent.

Safe exposure levels to noise have been established for the workplace by OSHA. Continuous exposure below 85 dBA, day after day, should not result in permanent hearing loss for the majority of people.

The OSHA Hearing Conservation Amendment requires that a hearing conservation program be initiated if the exposure levels exceed 90 dBA over 8 hours, the OSHA Noise Standard requires that engineering or administrative controls be utilized where and when feasible.

If you work in an area where the noise levels exceed those permitted by OSHA, you will be provided with hearing protection and periodic hearing tests. Signs must be posted in areas where the noise levels (employee exposure) exceed the OSHA limits, warning employees of the potential hazards, and informing them that hearing protection is required.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

16.0 GLOSSARY

Action Level	The level of exposure for a hazardous substance or agent at which certain provisions of the standards or guidelines must be initiated.
Acute	A toxic effect or reaction that occurs over a short period of time.
Breathing Zone Sample	An air sample collected in the breathing area of a worker to measure his/her exposure to airborne contaminants.
Carcinogen	Any substance that is capable of causing the development of cancer in living tissue. Example: asbestos
Chronic	Describes continued and repeated exposure, generally over a long period of time, to hazardous materials or harmful physical agents and the health effects that result from this type of exposure.
Combustible	Any solid that is difficult to ignite and that burns relatively slow; or liquids that have a flashpoint above 100°F. Example: wood
Corrosive	Any solid, liquid, or gas that bums, irritates, or destroys body tissues, usually the skin. When swallowed, the lungs and stomach will be affected. Example: strong acids (nitric acid) and strong bases (sodium hydroxide)
Dermatitis	Inflammation of the skin.
Dose	A term used to express the amount of hazardous substance or harmful physical agent absorbed by a body organ or an individual. (Concentration and duration).
Flammable	Any solid, liquid, vapor, or gas that ignites easily and bums rapidly; liquids that have a flashpoint below 100°F.
Gas	A material that us a gas under normal pressure and room temperature. Example: chlorine
Latent Period	The time which elapses between exposure to a chemical or physical agent and the appearance of symptoms.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

Safety Data Sheets	Any data sheet which contains information pertaining to the physical, chemical, and hazardous properties of a substance or mixture.
Mg/M^3	Milligram (mg) of substance per cubic meter (m) of air. Unit of measurement of dusts and other particles.
Mutagen	A substance capable of causing abnormal changes in living cells. All carcinogens are mutagens, but not all mutagens are carcinogens. Example: ionizing radiation.
NIOSH	National Institute for Occupational Safety and Health (research)
OSHA	Occupational Safety and Health Administration (research)
PEL	Permissible Exposure Limit. Legally enforceable limit set by OSHA.. Safe exposure level. (See page 3 of this manual)
PPM	Parts per million (ppm) of the substance per million parts of air. Unit of measurement for gases and vapors.
Reproductive Toxic Agent	Substances which interfere with the normal production of sperm or ovum or produce abnormalities in the embryo or fetus.
Respirator	A device worn over the nose and mouth to filter out hazardous substances from an individual's breathing air.
Sensitizer	Hazardous materials that have the ability to produce an allergic reaction in exposed workers.
Susceptibility	The varied responses of different persons to the same hazardous material. The usual factors affecting the response include age, sex, and general health.
Synergistic	Occurs when the combined effect of hazardous materials acting together on the body is greater than the sum of the effect of each agent acting alone. Example: asbestos and cigarette smoking produce a synergistic effect on the lungs.
Teratogen	A substance capable of producing a severe abnormality in an embryo or fetus. Examples: methylmercury, thalidomide.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

Toxicity	The ability of a hazardous substance to produce a harmful effect on a living organism.
TLV	Threshold Limit Value. Guideline exposure limits. (See page 3 in this manual)
Vapor	Vapors are formed by the evaporation of a substance which is normally a liquid at room temperature.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

17.0 UNDERSTANDING A SAFETY DATA SHEET

The following is a description of the information contained in a Safety Data Sheet (SDS); and how to read it and use this information. SDS can be accessed by the product Trade Name which can be found on the container label.

Section 1 gives details of the company issuing the data sheet....
SECTION I: Identification:
Trade name: Benzene
Manufacturer/Supplier:
Alfa Aesar, A Johnson Matthey Company Johnson Matthey Catalog Company, Inc. 30 Bond Street Ward Hill, ...

.... and, often, emergency call-out information. Emergency information: During normal hours the Health, Safety and Environmental Department. After normal hours call .....

The second section summarizes the major hazards associated with use of the chemical. The R and S codes in this section are followed by explanatory text.
SECTION II: Hazards identification
Hazard description:
T Toxic F Highly flammable
Information pertaining to particular dangers for man and environment
R 45 Can cause cancer - Group I (extremely hazardous)
R 11 Highly flammable.
R 48/23/24/25 Toxic: danger of serious damage to health by prolonged exposure through inhalation, in contact with skin and if swallowed.

The third section identifies the material, and gives the CAS and other registry numbers.
SECTION III: Composition, Information on Ingredients:
Benzene (CAS# 71-43-3); 100%
Identification number(s):
EINECS Number: 200-753-7
EU Number: 601-020-00-8

The fourth section outlines first aid measures.
SECTION IV: First aid measures
After inhalation: Supply fresh air. If required, provide artificial respiration. Keep patient warm. Seek immediate medical advice. After skin contact .....

Section 5 covers fire fighting and protective equipment.
SECTION V: Fire fighting measures
Suitable extinguishing agents Carbon dioxide, extinguishing powder or water spray. Fight larger fires .....

Section 6 outlines the procedures to be followed in case of accidental release of the chemical, including methods to be used to clean up spills. Note that these measures are

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

unlikely to be sufficiently detailed if the chemical is particularly hazardous, and local procedures should be drawn up to supplement what is given in the SDS sheet.
SECTION VI: Accidental release measures
Person-related safety precautions: Wear protective equipment.....
Measures for environmental protection..... Do not allow material to be released to the environment without proper governmental permits. Measures for cleaning/collecting: Absorb with liquid-binding material (sand, diatomite, acid binders, universal binders, sawdust). Dispose contaminated material as waste according to item 13 .....

Section 7 is self-explanatory. This is an important section, sometimes overlooked by those using chemicals in the laboratory. It contains information about the possible formation of peroxides in storage, flammability, explosive risks, etc. Pay particular attention to the possible need for flammable storage cabinets, explosion-proof fridges, and also the need to avoid storage near incompatible chemicals.
SECTION VII: Handling and storage
Information for safe handling:
Keep container tightly sealed. Store in cool, dry place in tightly closed containers. Ensure good ventilation at the workplace. Information about protection against explosions and fires: Keep ignition sources away. Protect against electrostatic charges. Fumes can combine with air to form an explosive mixture.

Storage
Requirements to be met by storerooms and receptacles: Store in a cool location. Store away from oxidizing agents .....

Section 8 provides information on regulatory standards for exposure, in other words, the maximum permitted concentration of the material in the environment to which you are allowed to be exposed. It also usually contains information on suitable types of PPE (personal protective equipment)
SECTION VIII: Exposure controls and personal protection
Additional information about design of technical systems: Properly operating chemical fume hood designed for hazardous chemicals and having an average face velocity of at least 100 feet per minute. Components with limit values that require monitoring at the workplace: Benzene mg/m3 ml/m3 ACGIH TLV short term 1.6 0.5 ACGIH TLV long term 8 2.5 B VME 1,6 0,5 .....

Personal protective equipment General protective and hygienic measures The usual precautionary measures for handling chemicals should be followed. Keep away from foodstuffs, beverages and feed. Remove all soiled and contaminated clothing immediately..... Wash hands before breaks and at the end of work. Breathing equipment:.....
Protection of hands: Impervious gloves
Eye protection: Safety glasses, Full face protection .....

Section 9 is self-explanatory
SECTION IX: Physical and chemical properties:
Form: Liquid
Color: Colorless
Odor: Aromatic

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

Change in condition
Melting point/Melting range: 5.51 °C
Boiling point/Boiling range: 80.1 °C
.....

The next section is also largely self-explanatory.
SECTION X: Stability and reactivity
Thermal decomposition / conditions to be avoided: Decomposition will not occur if used and stored according to specifications.
Materials to be avoided: Oxidizing agents
Dangerous reactions No dangerous reactions known
Dangerous products of decomposition: Carbon monoxide and carbon dioxide

---

Section 11 outlines the risks to which you may be exposed when using the chemical. It is therefore a section of crucial importance!
SECTION XI: Toxicological information
Acute toxicity: (The acute toxicity gives an indication of the kind of quantities of the chemical which may cause immediate damage to health if swallowed, inhaled or absorbed through the skin.)

LD/Lc50 values that are relevant for classification: If you have never heard of LD50s, look in the glossary on this site.

Oral: LD50: 3306 mg/kg (rat)
Dermal: LD50: 48 mg/kg (mus)
Inhalative: LC50/7H: 10.000 ppm/7H (rat)

(There follows a section which gives, often in some detail, an indication of the health effects which may be attributable to this chemical. This section should be read particularly carefully, since the range of health effects may be broad, and may include carcinogenic or sensitizer effects.)

Primary irritant effect:
on the skin: Irritant to skin and mucous membranes.
on the eye: Irritating effect.
Sensitization: No sensitizing effects known. (Chemical sensitisation, for example by platinum compounds, is a potentially debilitating problem. Pay particular attention to any information which may suggest that the chemical is a sensitiser.)

Subacute to chronic toxicity: (Here we find details of the possible long-term effects of exposure to the chemical.)

Benzene has a strong irritating effect, producing erythema and burning. Edema and blistering is possible in more severe cases. Absorption through the skin may cause the same symptoms as inhalation or ingestion. These include gastrointestinal irritation, low blood pressure, headache, blurred vision, nausea, vomiting, dizziness, loss of balance and coordination, confusion, unconsciousness, coma, respiratory failure and death. Blood, liver and kidney damage is possible. Benzene is a recognized leukemogen and an experimental mutagen and teratogen.

EMAX ORGANIZATION & MANAGEMENT
B-5 Health & Safety Practices

Additional toxicological information:
To the best of our knowledge the acute and chronic toxicity of this substance is not fully known. (For this chemical there now follow important comments regarding the carcinogenicity. The acronyms such as IARC refer to regulatory or health agencies.)

EPA-A: human carcinogen: sufficient evidence from epidemiologic studies to support a causal association between exposure and cancer.

IARC-2A: Probably carcinogenic to humans: limited human evidence; sufficient evidence in experimental animals

NTP-2: Reasonably anticipated to be a carcinogen: limited evidence from studies in humans or sufficient evidence from studies in experimental animals.

ACGIH A2: Suspected human carcinogen: Agent is carcinogenic in experimental animals at dose levels, by route(s) of administration, at site(s), of histologic type(s), or by mechanism(s) considered relevant to worker exposure. Available epidemiologic studies are conflicting or insufficient to confirm an increased risk of cancer in exposed humans.

Section 12 is largely self-explanatory
SECTION XII: Ecological information:
General notes: Do not allow material to be released to the environment without proper governmental permits.

Section 13, which deals with disposal, is often not sufficiently detailed for you to be able to undertake disposal yourself. If you need to dispose of the chemical after use, ensure that you know how to do this safely.
SECTION XIII: Disposal considerations
Consult state, local or national regulations for proper disposal.

Section 14 gives transport information, generally as a list of codes indicating the dangers associated with the chemical (flammable, radioactive, very toxic, etc) and the type of transport which may be used. There are usually UN hazard codes given in this section. A guide is these is available here.
SECTION XIV: Transport information
DOT regulations:
Hazard class: 3 Identification number: UN1114 Packing group: II .....

Section 15 lists the hazard codes (see glossary if you are not familiar with these) which indicate the principle hazards associated with the chemical and the precautions which should be taken when working with it.
SECTION XV: Regulations
Hazard symbols:
T Toxic F Highly flammable

Risk phrases:
45 Can cause cancer - Group I (extremely hazardous)
11 Highly flammable.
48/23/24/25 Toxic: danger of serious damage to health by prolonged exposure through

inhalation, in contact with skin and if swallowed.
A full list of these risk phrases is given here.

Safety phrases:
20 When using do not eat or drink.
28 After contact with skin, wash immediately with plenty of ... .
36/37/39 Wear suitable protective clothing, gloves and eye/face protection.
45 In case of accident or if you feel unwell, seek medical advice immediately.
A full list of safety phrases is available here.

National regulations (This may include a variety of country-specific detail) All components of this product are listed in the U.S. Environmental Protection Agency Toxic Substances Control Act Chemical Substance Inventory .....
This product contains a chemical known to the state of California to cause cancer or reproductive toxicity.
This product contains benzene and is subject to the reporting requirements of section 313 of the Emergency Planning and Community Right to Know Act of 1986 and 40CFR372.

Finally, a section of an additional information, such as the name of the person preparing the data sheet, a list of references from which data have been drawn, disclaimers, etc.
SECTION XVI: Other information:
Employers should use this information only as a supplement to other information gathered by them, and should make independent judgement of suitability of this information to ensure proper use and protect the health and safety of employees ....
Contact:

c) Surrogates

Recovery of surrogates on a given sample is a gauge of accuracy of target analytes detected on a given sample. Recoveries shall be evaluated against project surrogate control limits otherwise, in-house limits shall be used. When recovery exceeded surrogate control limits, project prescribed corrective action shall be applied. When re-analysis is necessary, it shall be performed unless prohibiting factors are present (e.g., re-analysis will cause damage to instrument or chromatography obviously display qualitative and quantitative quandary).

1.7.5. Sample Handling

a) Thermal Preservation

Samples are generally received on coolers cooled by ice unless thermal preservation is not required. Temperature is checked and documented in accordance to EMAX-SM02 SOP for Sample Receiving.

b) Chemical Preservation

Samples that are chemically preserved are also checked and documented during sample receipt with the exception of the samples that are not amenable to be opened or sub-sampled prior to analysis (e.g., samples for volatile analyses, oil and grease). Details for this procedure are also described in EMAX-SM02.

c) Holding Time

Sample holding time is checked against the COC sampling date and time. Maximum holding time for analyses are listed in EMAX-SM02.

For a test with recommended maximum holding time measured in hours, the holding time is tracked by the hour. For a test with recommended maximum holding time measured in days, the holding time is tracked by the day. For a test with a recommended maximum holding time measured in months, the holding time shall be tracked by the month. One month is defined as 30 days.

c) Start Time Determination

For extraction, the moment that extraction solvent touches the sub-sample determines the start time. For analysis, the moment that the extract (or sample) is introduced into the analytical instrument determines the start time.

LABORATORY QUALITY ASSURANCE MANUAL

(LQAM)

Rev. 31

September 9, 2019

Quality Assurance Manager: Melanie Levesque

The Laboratory Quality Assurance Manual is effective as of the date of the signature of the Quality Assurance Manager

EUROFINS AIR TOXICS,LLC
180 BLUE RAVINE ROAD, SUITE B
FOLSOM, CA 95630
(800) 985-5955
eurofinsus.com

LABORATORY QUALITY ASSURANCE MANUAL

Approvals

Robert Mitzel, President — Date: 9/5/2019

Heidi Hayes, Technical Director — Date: 9-9-2019

Sepideh Saeed, Laboratory Director — Date: 09-09-2019

Melanie Levesque, Quality Assurance Manager — Date: 09/09/2019

REVISION LOG

Revision: 26	**Effective Date: 03-05-2014**	
Section	**Justification**	**Changes**
Cover page; Appendices including Method Manuals, Organizational Chart, terms and definitions, and references.	Appendices were updated to correspond to the most current SOPs, methods, DoD, and TNI definitions.	General formatting and spelling errors corrected throughout the entire document.
Revision: 27	**Effective Date: 03-23-2015**	
Section	**Justification**	**Changes**
Sections 1.1, 6.3, 8.1, 11.4.1, Appendix A; Sections 2.1, 2.6.1, 6.2; Appendix C and D.	Materials testing using environmental chambers department was closed; management team was reorganized.	Remove references to materials testing using environmental chambers; remove information relating to the VP of materials testing; Update the accreditation list and organization chart to current.
Revision: 28	**Effective Date: 05-10-2016**	
Section	**Justification**	**Changes**
Sections 1.1, 2.4, 2.5, 2.6.2, 2.7.2, 2.11, 3.1, 5.5, 6.3, 6.4.1.1, 8.1, 8.2, 10.3, 11.2, and Appendix F	Update to the most current SOPs, methods, Corporate procedures, and DoD definitions.	Update to current accreditation programs, changed references of Group Leaders to Supervisors, updated management report to yearly, updated IT management to Corporate IT, added annual ethics training and Lighthouse Services for anonymously reporting data integrity concerns, added D4 for document control, removed product samples reference and unit, updated to current scope of testing, added manual data entry into LIMS procedure, and removed

		facility map from Appendix F.
Revision: 29	**Effective Date: 08-22-2017**	
Section	**Justification**	**Changes**
Sections 1.3, 2.1, 2.6.1, 2.6.3, 2.7.2, 6.3, 6.4.1.2, 6.4.1.3, 6.4.1.5, 6.4.1.6, 7.1, 8.2, 9.1.1, 10.8, 11.2, Appendices A, D, and E.	Update to the most current Corporate procedures SOPs, and NELAP criteria.	Update to current Corporate Ethics Policy statement and required yearly form signature, updated IT department to Corporate IT as well as data system back-up timelines, and archiving procedure to Corporate IT procedures, updated procurement procedure to include the new PtP system, updated management review procedure to reflect the new Corporate form, added procedure for absence of Technical Director that surpasses 15 and 30 consecutive days, added procedure for client notification prior to data records destruction as well as the procedure for lab closure and change in ownership, updated Board of Directors to Eurofins Environment Testing, added procedure for maintaining employee list, updated instrument count, and removed methods (TO-11A and ASTM D-5504) no longer provided.
Revision: 30	**Effective Date: 08-30-2018**	
Section	**Justification**	**Changes**
Sections 2.6.2, 2.11, 3.1, 5.1, 5.4, 6.3, 7.1, 8.2, 10.3, 10.8,	Updated to current laboratory procedures, SOPs, and	Updated to include in-house IT, included procedure for

11.4.1, 13.3, Appendices A, C, D, E, and F.	information.	notifying clients of data issues, updated square footage of building and office space, updated information sent with media shipments, added sorbent tube media for tracking, updated instrumentation count, updated procurement procedure to include the new COUPA system, updated to include new methods, updated in which documents calculations are kept, removed CD-ROMS for record storage, added methods TO-3, TO-14A, TO-15 and 325B to the PT studies, added reference to subcontracting SOP, added new definitions for MADEP APH and TOF, included new certifications for the state of AK and FL, updated org. chart, document references and company name change throughout.
Revision: 31	**Effective Date: 09-09-2019**	
Section	**Justification**	**Changes**
2.1, 2.11 8th bullet point, 6.3, 8.2, 9.4, 11.4.1 last bullet point, 11.4.2, Appendices A to F.	Updated to current laboratory procedures, SOPs, information, and DoD requirements.	Updated ISO 17025 to the correct reference of ISO/IEC 17025:2017 throughout the LQAM, added Group Leaders as part of management, referenced key managerial personnel to org. chart in Appendix D, included AB to notify for out of compliance results and QA-type issues, updated current equipment

		information, added EO SOP 134 to analytical test methods table, updated ISO/IEC statement on measurement uncertainty, updated QA status report to QA Management Review report and verbiage, removed definitions that are no longer applicable, updated SOP references, added NH to NELAP certifications table, included current org. chart, added carbopack B to 325B tables, removed BTEX from TO-3 method information and tables, removed references to ASE and Soxhlet for TO-13A extractions, updated various RLs for methods TO-14/TO-15 Low Level, SIM and TO-17 Passives SE, updated Table 2 for TO-17 VI, updated.

TABLE OF CONTENTS

1. INTRODUCTION

1.1 Our Unique Promise of Value 10
1.2 Mission Statement 10
1.3 Quality Policy 10
1.4 Statement of Values 12
1.5 Certifications, Accreditations, and Registrations 12

2. ORGANIZATION AND PERSONNEL

2.1 Organizational Structure 13
2.2 Management Responsibilities 14
2.3 Overview of the Quality Assurance Program 14
2.4 Quality Assurance Responsibilities 15
2.5 Communication of Quality Issues to Management 15
2.6 Personnel Qualifications and Responsibilities 16
2.7 Training 18
2.8 Employee Safety 20
2.9 Client Services/Project Management Responsibilities 21
2.10 Confidentiality 21
2.11 Operational Integrity 21

3. BUILDINGS AND FACILITIES

3.1 Facility 23
3.2 Security 23

4. DOCUMENT CONTROL

4.1 Controlled Documents Used at Eurofins Air Toxics 24
4.2 Document Approval, Issue, Control, and Maintenance 25
4.3 Laboratory Logbooks and Forms 25
4.4 Archival and Storage of Documents 26

5. SAMPLE HANDLING

5.1 SAMPLE COLLECTION 26
5.2 SAMPLE RECEIPT AND ENTRY 26
5.3 SAMPLE IDENTIFICATION AND TRACKING 27
5.4 SAMPLE STORAGE 28
5.5 SAMPLE RETURN/DISPOSAL 28
5.6 CHAIN OF CUSTODY 28

6. TECHNICAL REQUIREMENTS – TRACEABILITY OF MEASUREMENTS

6.1 REAGENTS AND SOLVENTS 29
6.2 CALIBRATION STANDARDS 30
6.3 EQUIPMENT AND INSTRUMENTATION 30
6.4 COMPUTERIZED SYSTEMS AND COMPUTER SOFTWARE 33

7. PURCHASING EQUIPMENT AND SUPPLIES

7.1 PROCUREMENT 36
7.2 SUPPLIER EVALUATION 36

8. ANALYTICAL METHODS

8.1 SCOPE OF TESTING 37
8.2 ANALYTICAL TEST METHODS 38
8.3 METHOD VALIDATION 39
8.4 PROCEDURAL DEVIATIONS 39

9. INTERNAL QUALITY CONTROL CHECKS

9.1 LABORATORY QUALITY CONTROL SAMPLES AND ACCEPTANCE CRITERIA 40
9.2 QUALITY CONTROL SAMPLE FREQUENCY AND CORRECTIVE ACTION 42
9.3 QUALITY CONTROL CHARTS 43
9.4 MEASUREMENT UNCERTAINTY 43

10. ASSURING QUALITY OF TEST RESULTS

10.1 Data Management 43
10.2 Data Documentation 44
10.3 Data Calculations 45
10.4 Reporting Limits 45
10.5 Data Review 46
10.6 Data Qualification 46
10.7 Data Reporting 46
10.8 Data Storage, Security, and Archival 47

11. AUDITS AND INSPECTIONS

11.1 Internal Quality Assurance Audits 48
11.2 Management Review System 49
11.3 Client Audits and Agency Inspections 50
11.4 Proficiency Testing Program 50

12. CORRECTIVE AND PREVENTIVE ACTION

12.1 Laboratory Investigations and Corrective Action 53

13. SERVICE TO CLIENTS

13.1 Review of Work Requests, Tenders, and Contracts 53
13.2 Timely Delivery 54
13.3 Subcontracting 54

APPENDICES

Appendix A – Terms and Definitions
Appendix B – Procedure Cross Reference List
Appendix C – Certification and Accreditation
Appendix D – Organizational Charts
Appendix E – Analytical Methods
Appendix F – References

1. INTRODUCTION

The purpose of the Laboratory Quality Assurance Manual is to provide a framework to outline the quality systems at Eurofins Air Toxics, LLC.

1.1 Our Unique Promise of Value

Eurofins Air Toxics is the global leader in the NELAC Institute (TNI) National Environmental Laboratory Accreditation Program (NELAP) for accredited vapor-phase environmental analytical laboratory services, and is also DoD-ELAP accredited for environmental air and emissions testing for key methods.

Eurofins Air Toxics supports public and private sectors, including engineering and consulting firms, manufacturers, industry, government, retailers and others by offering a wide variety of certified air methods. Eurofins Air Toxics provides unmatched quality, capacity, and technical expertise to deliver an outstanding service experience to clients worldwide.

1.2 Mission Statement

Eurofins Air Toxics, LLC is an analytical and environmental laboratory specializing in the analysis of vapor-phase contaminants and air quality parameters. Our business is guided by four key principles:

1) Providing unmatched data integrity
2) Establishing long-term relationships
3) Delivering quality client service
4) Exceeding client expectations

1.3 Quality Policy

The Executive Management Group recognizes quality as a key element of the laboratory's standard of service. This group supports the laboratory's commitment to quality as defined by NELAP and ISO/IEC 17025:2017.

The Quality Policy Statement gives employees clear requirements for producing analytical data that is scientifically valid, legally defensible, accurate, impartial, and of known and documented quality, through strict adherence to the Quality Policy Statement. The Corporate Quality Assurance Director wrote the Quality Policy Statement with final approval from the President of Eurofins Environment Testing . The policy cannot be revised without the Corporate Quality Assurance Director and Quality Assurance Officer's approvals. Employees are trained on

the components of the Quality Policy Statement during their orientation. All employees sign the statement as agreement to implement the policy in all aspects of their work. The statement is as follows:

Quality Policy Statement

We strive to provide the highest quality data achievable by:

- Reading and understanding all of the quality documents applicable to each position and implementing the process in our work.

- Following all recordkeeping requirements; describing clearly and accurately all activities performed; recording "real time" as the task is carried out; understanding that it is never acceptable to "back date" entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.

- Ensuring data integrity through the completeness, consistency, and accuracy of the data generated. Complete, consistent, and accurate data should be attributable, legible, contemporaneously recorded, original or a true copy, and accurate (ALCOA). This applies to manual paper documentation and electronic records.

- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification/validation, analysis, and reporting; establishing an audit trail (the who, what, when, and why) that identifies date, time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.

- Emphasizing a total quality management process which provides accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.

- Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.

- Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action;

documenting these activities completely, including the reasons for the decisions made.

- Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concerns on quality issues to management.

We each take personal responsibility to provide this quality product while meeting the company's high standards of integrity and ethics, understanding that improprieties, such as failure to conduct the required test, manipulation of test procedures or data, or inaccurate documentation will not be tolerated. Intentional misrepresentation of the activities performed is considered fraud and is grounds for termination.

1.4 Statement of Values

At Eurofins Air Toxics, we strive to be the BEST in everything that we do. Our very existence is based on our continued ability to provide innovative, dependable, and cost-effective environmental services to our clients. We CARE about our clients as well as our co-workers and manage our daily activities to build relationships based on mutual TRUST, HONESTY, and RESPECT. We are LEADERS in our field and accept the risks associated with building new frontiers in our professional lives. Our strength comes from our TEAMS for through them we can achieve our goals.

1.5 Certifications, Accreditations, and Registration

Accreditation/Certification is the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications and/or standards. It is the one generally accepted method by which a laboratory such as ours can demonstrate its capability of generating acceptable, professional, quality test results in those areas in which it claims competence. To this end, we have actively sought accreditation by organizations offering it in areas relevant to our technical expertise. We strive to ensure that the facility, equipment, procedures, records, and methods used by Eurofins Air Toxics laboratory in the testing of environmental samples are in compliance with the requirements of these standards.

Appendix C lists accreditations held by Eurofins Air Toxics, LLC in support of environmental work. Current copies of all scopes of accreditation are kept on file in the Quality Assurance Department.

2. ORGANIZATION AND PERSONNEL

2.1 Organizational Structure

Eurofins Air Toxics' management organization includes six core areas: Operations, Information Technology (IT), Client Services, Research, Sales and Marketing, and Finance and Administration. The management staff includes executives, directors, managers, and supervisors (group leaders). Each operating area is led by a manager and/or a supervisor (group leader). In the absence of a member of the laboratory and operational management team, deputies are appointed as follows:

Position	Deputy
President	Laboratory Director or appointee
Technical Director	Quality Assurance Manager or appointee
Quality Assurance Manager	Technical Director or appointee
Laboratory Director	Technical Director or appointee
Supervisors	Laboratory Director

If the Technical Director is absent for more than fifteen consecutive calendar days, the Quality Assurance Manager or designee meeting the qualifications of the Technical Director will temporarily fulfill the role. If the absence is greater than thirty-five consecutive calendar days, the Quality Assurance Manager will notify the primary accreditation body in writing.

Eurofins Air Toxics' management and executive (President)are committed to following and assuring compliance with the TNI Standard as defined in this Laboratory Quality Assurance Manual (LQAM). Each manager is responsible for implementing and maintaining systems as they affect their teams and for participating in their respective role in the management systems as outlined in the LQAM.

Additional key managerial personnel are presented in an Organizational Chart in Appendix D of this manual. This organizational structure is created in a way to avoid any potential for conflicts of interest or undue pressure that might influence the technical judgment of analytical personnel.

2.2 Management Responsibilities

The management team consists of supervisors (group leaders), managers, and directors, and positions above those. The following is a list of management responsibilities:

- Personnel hiring and training
- Supervision of personnel
- Ensuring quality of data produced
- Resources allocation
- Directing daily work operations, including scheduling of work
- Maintaining awareness of technical development and regulatory requirements
- Assessing laboratory capacity and workload
- Contributing to the continuous improvement of the laboratory operation
- Providing resources to ensure a safe work environment
- Providing resources to ensure a work environment free of undue pressures
- Communicating problems and concerns to executive management to enlist a higher level of support for corrections and continuous improvement, ensuring compliance with the requirements of NELAP and ISO/IEC 17025:2017
- Ensuring that corrective actions are carried out in an appropriate and agreed upon time frame

The Quality Assurance Manager ensures that the laboratory's policies and objectives for quality of testing services are documented in this quality manual. The Quality Assurance Manager must assure that the manual is communicated to, and understood and implemented by all personnel concerned.

2.3 Overview of the Quality Assurance Program

The Quality Assurance (QA) Department is responsible for developing planned activities the purpose of which is to provide assurance to all levels of management that a quality program is in place within the laboratory, and that it is functioning in an effective manner that is consistent with the requirements of NELAP and ISO/IEC 17025:2017. Although Eurofins Air Toxics is a wholly owned subsidiary of Eurofins Scientific, the Quality Assurance and quality systems described in this manual are specific to Eurofins Air Toxics.

2.3.1 Quality Assurance Manager

The Quality Assurance Manager ensures that the quality system is followed at all times. The QA Manager reports directly to the President in order to maintain independence from business operating units and facilitate communications regarding quality-related issues. The QA Manager has no direct supervisory responsibility for the generation of technical data to avoid any conflict of interest in administrating the QA program. The QA Manager has the final authority to stop work that compromises the laboratory's integrity or data quality. The situation must be investigated and appropriate corrective action must be put in place before the QA Manager will authorize the resumption of work. The specific duties of the QA Manager are communicated in job description format.

2.4 Quality Assurance Responsibilities

The Quality Assurance team is responsible for implementing and maintaining Quality Assurance procedures throughout the laboratory. This is accomplished via coordination and dissemination of internal and external assessment information, review of Standard Operating Procedures (SOPs) to document variances taken to published methods, monitoring of the Quality Assurance Manual to ensure consistency with actual practices, maintenance of an ongoing Corrective Action Program with yearly reports to the senior management team, a leadership role in employee training, data review, and other quality control-related programs.

The QA team is free from any commercial, financial, or production pressures when making assessments or decisions regarding the quality of work produced or effectiveness of the quality systems.

2.5 Communication of Quality Issues to Management

Communication between the Quality Assurance (QA) team and other management teams occurs on a regular basis (typically via monthly status meetings). Information regarding outstanding corrective action items, upcoming assessments, assessment results, and/or general observations are discussed and documented via a database of agenda notes. The QA databases along with the Laboratory Information Management System (LIMS) database are used to compile a Yearly Quality Assurance Status Report, which is distributed to the senior management team for review.

2.6 Personnel Qualifications and Responsibilities

Full resumes and specific position descriptions for all personnel are located in Corporate Human Resources (HR) Department files. In addition, the Supervisors (Group Leaders) have copies of position descriptions for their staff.

2.6.1 Executive Team

President: Provides leadership that ensures the founding mission and core values of the company are put into practice. The President leads programs relating to the development of long-range strategy, quality systems, financial infrastructure and sales. The President also provides day-to-day leadership and management of programs for overseeing the processes and resources necessary for establishing long-range service objectives, plans, and policies in cooperation with Eurofins Environment Testing. The President is responsible for the measurement and effectiveness of both internal and external processes by providing accurate and timely feedback on the operating condition of the company. In addition, the President directs the definition and operation of the laboratory production by fostering a success-oriented and accountable environment within the company.

Technical Director: The Technical Director is responsible for developing products and solutions to meet client and industry needs, and also oversees the validation process of current and new products to ensure quality objectives are met and documented as defined.

Laboratory Director: Responsible for managing the operations of the laboratory, profit/loss relating to operations, laboratory efficiency improvement in software and instrument automation, and serves as the primary interface between finance, HR, IT, project management, and sales/marketing. The Laboratory Director has the overall responsibility of ensuring customer satisfaction goals are met while elevating the skill and training of key technical staff as well as assuring that state-of-the-art instrumentation and capital assets are in place to meet global customer needs.

2.6.2 Management Team:

Laboratory management and personnel are free from any commercial, financial, or production pressures when making technical judgments or decisions regarding the quality of work produced.

Corporate and In-House Information Technology (IT) Team: Oversees all aspects of software engineering and development, database administration, and network administration. The Corporate and in-house IT Team is instrumental in designing and implementing model work-flow processes, defining user requirements, and proposing software design and implementation to satisfy long-term company business goals. This role provides established policies and procedures to ensure continuous database and server environment integrity and reliability.

Quality Assurance Manager: Responsible for overseeing the quality systems in the laboratory. Key to the Quality Assurance role is a focus on continuous improvement through effective monitoring of systems and evaluation of non-compliance and corrective actions. To support the quality systems, the Quality Assurance Manager leads the internal and external audit programs, negotiates audit resolution, and oversees the effectiveness of the Corrective Action Report (CAR) program. The QA Manager is tasked with providing timely feedback to front-line managers and bench staff regarding quality programs and also a big-picture assessment to senior management. Additionally, the QA Manager ensures required documentation and certifications are current and accurate, including regulatory accreditations, the LQAM, and SOPs.

Supervisors (Group Leaders): Responsible for day-to-day operations of the laboratory or specific departments. The Supervisors oversee technical operations, sample analysis, data entry, report generation, provision of resources, and other related areas. In addition, they are responsible for employee management and review. Supervisors report directly to the Laboratory Director. Managerial decisions are made by the Laboratory Director in their absence.

2.6.3 Laboratory Staff and Responsibilities

It is the primary responsibility of laboratory staff to produce quality data within the framework of each individual method and within the parameters of the laboratory's quality control guidelines. It is also the responsibility of staff to identify existing problems or inefficiencies, and to improve the processes of the laboratory whenever possible. Duties for these personnel typically include:

- Sample preparations
- Performance of analytical tests
- Calibrations, operation, and maintenance of instruments

- Standard and reagent preparation
- Sample storage
- Data entry
- Data package preparation

Eurofins Air Toxics maintains the confidentiality of the laboratory staff. A list of employee names, initials, and signatures is kept in the QA Department. The list is updated annually and is available for on-site review upon request.

2.7 Training

The experience and training received by personnel is of great importance to Eurofins Air Toxics' clients and regulatory agencies. Accurate training documentation is the responsibility of both employees and their supervisors. On a routine basis, the supervisor reviews and signs training documentation to verify that it is complete and current.

Each laboratory analyst being trained to perform a new analysis is required to perform an initial Demonstration of Capability (DOC) and meet the requirements for accuracy and precision before working independently on the test methods. Typically this is accomplished by the successful analysis of at least four aliquots of a laboratory quality control sample. However, there are certain tests that are not required by the mandated test method or regulation to perform the above procedure (e.g., PM10). In this case, the analyst's proficiency demonstration is satisfied by documentation of having read, understood, and agreed to follow the SOP, specific department or method forms and procedures, and observation by scientist or senior analyst.

Management personnel are responsible for planning ongoing professional growth and development activities for an employee through on-the-job training and/or internal and external training courses so that an employee can maintain a current skill set to match job responsibilities.

An annual performance review based on job accountabilities, objective measures, and pre-defined standards is completed by management personnel for each employee. This assessment is documented and maintained. Input is obtained from other managerial personnel as needed.

2.7.1 New Hire Training

New employees learn about personnel and safety policies as well as business strategies through a formal process administered by our Corporate Human Resources Team, Laboratory Director, and the Safety

Committee. All new employees are also required to attend the Quality Assurance Orientation. Completion of the orientation is documented in the employee's Training Record. The course outline includes:

- Introduction to QA
- Definitions of SOPs and LQAM
- How to use CARS
- Logbook protocol
- Chain-of-custody procedures
- Training Documentation
- Overview of Eurofins Air Toxics classes including Ethics and Integrity courses
- Overall Training Record organization and upkeep

New employee training continues with review and signing of the Eurofins' Ethics Policy Statement, a review of the Quality Assurance Manual, and signing of the Quality Policy. Upon completion of this training, employees move on to analytical method training if required for their position. Other non-testing training materials may be required by the departments.

In general, the laboratory staff reviews the department's SOPs and/or the regulatory method as well as the instrument manual. The employee will then observe while an experienced analyst prepares samples and operates the instrument. Training includes sample handling and preparation, documentation protocols, calibration procedures, QC requirements, data management, data reporting and troubleshooting.

2.7.2 Ongoing Training

Annual Ethics and Data Integrity training is provided by the QA Manager. All employees are required to attend. Training is documented with signing of the attendance sheet and Eurofins' Ethics Policy Statement which are maintained in the QA department.

After successful completion of the initial Demonstration of Capability, all laboratory staff must demonstrate continued proficiency. Whenever there is a change in test method, instrument method type, and/or personnel a new DOC must be performed. At least once per year, each analyst must demonstrate continued proficiency on assigned technical methods. The QA Department notifies personnel via an automated e-mail generated from the document control program, D4, or via personal e-mail whenever a new SOP is generated or a current SOP is updated. Employees

responsible for that method or procedure must read the new or updated SOP within 30 days and document the review in the document control program, D4, and in the LIMS SOP Tracker module. In addition, the Laboratory Quality Assurance Manual and the Chemical Hygiene Plan must be annually reviewed by all employees.

Employees are re-trained if an issue or investigation warrants that it is a necessary corrective action. Management provides direction as to when employee re-training is required, and to the extent of the re-training.

2.8 Employee Safety

Laboratory staff may, on occasion, be exposed to handling of solvents, compressed gases, calibration standards, or other hazards. Eurofins Air Toxics designates an assigned Safety Officer and several staff members who comprise the Safety Committee. Some members are 40-hour OSHA-trained and respirator-fitted.

Employee education in the safe handling and disposal of these materials is accomplished as follows:

- Each new employee is given a safety tour of the facility within the first two days of employment. Documentation of this orientation appears in the employee's Training Record.
- The Safety Committee meets frequently to discuss safety concerns and ways of improving safety in the work place.
- The Safety Committee schedules ongoing safety training throughout the year.
- If special precautions must be taken to perform a method, a safety section is included in the method SOP or in a stand-alone SOP which discusses protocols and other measures for risk reduction through exposure prevention.
- Safety Data Sheets (SDSs), formerly Material Safety Data Sheets (MSDS), are maintained for each chemical used on-site. The SDSs are accessible to personnel in a designated area in the laboratory and/or electronically through the chemical inventory database (CISpro) at all times. SDSs are also accessible on the Internet from product vendors.

- The Safety Committee members are assigned to duties that include hazardous waste disposal, incident or spill management, scheduling staff training, safety site assessments, Chemical Hygiene Plan review, and the overall leadership of the Safety Program.

2.9 Project Management Responsibilities

The Project Management group is responsible for organizing and managing client projects. Clients are assigned a Project Manager who serves as their primary contact. It is the Project Manager's responsibility to act as client advocate by communicating client requirements to laboratory personnel and ensuring that clients provide complete information needed by the laboratory to meet those requirements. All client verbal and electronic communications are documented by the project managers in the LIMS Contacts module. In addition to information management, project management responsibilities include:

- Coordinating and preparing proposals in conjunction with technical staff, including review of project-specific documents and negotiations of variance requests
- Documentation of project requirements
- Coordinating and communicating turnaround-time (TAT) requirements
- Scheduling sample submissions, sample containers, and sample pickup via Eurofins Air Toxics courier service
- Informing clients of deviation from their contract

2.10 Confidentiality

Strict confidentiality is maintained in all of Eurofins Air Toxics dealings with clients. All employees are required to protect company data, including client names and/or test results from disclosure to any third party. This policy is presented to employees in SOP #99 and during their orientation period.

Clients are promptly notified if their data is subpoenaed or requested by a regulatory or legal body.

In order to ensure the confidentiality of our systems and procedures within the laboratory, it is Eurofins Air Toxics' policy to restrict the distribution of our internal procedures to clients. Clients are, however, permitted to review the laboratory's procedures while on-site as part of an audit or visit. Based on this policy, the laboratory requests that any document viewed is not shared or made available to any third parties without the permission of Eurofins Air Toxics.

2.11 Operational Integrity

All employees sign an Employee Ethics Statement on their first day of employment. Employees responsible for generating, handling, or reviewing

laboratory data understand that Eurofins Air Toxics' mission is to perform all work with the highest level of integrity. Shortcuts or generating results to suit a client's purpose, rather than adhering to good scientific practices, is not considered acceptable under any circumstances. Any violation of the laboratory ethics policy results in a detailed investigation that could lead to termination. Examples of violations of data integrity are listed below:

- Knowingly recording inaccurate data
- Fabrication of data without performing the work needed to generate the information; this includes creating any type of fictitious data or documentation
- Time travel or adjusting clocks on computerized systems to make it appear that data was acquired at some time other than the actual time
- Manipulation of data for the express purpose of passing systems suitability or quality control criteria
- Selective use of data generated, or not using data that was legitimately generated to impact the outcome of a test
- Executing significant deviations from approved test methods and procedures without prior approval from Eurofins Air Toxics management and/or the client

If an issue does arise which could compromise data integrity, personnel are instructed to perform the following activities:

- Clearly document the situation and maintain all data generated. There is a big difference between poor judgment and fraud. Fraud usually involves intent to conceal an action taken. Therefore, the more documentation that is maintained the less likely an action is considered fraudulent if further scrutinized. All documentation of the inquiry and subsequent disciplinary actions will be maintained by the Laboratory Director, Supervisor and the Corporate Human Resources Team for at least five years.
- When out-of-specification results or quality control-type issues are detected, all supporting data and relative background information must be documented and presented for management review. The QA Manager or member of the management team will notify the client and the Accreditation Body (AB) verbally followed by a written memo detailing the issue within 15 business days of discovery. A Corrective Action Report is initiated documenting the root cause and resolution or proposed resolution will also be submitted to the client and AB within 30 days of discovery.
- Any questionable situations and decisions must be reviewed with a supervisor.

- Questionable or uncomfortable issues are brought directly to the QA Manager or a member of the QA Department as part the QA "open door" policy. If an employee desires to remain anonymous, he or she is encouraged to contact our anonymous reporting hotline – Lighthouse Services, Inc. The Lighthouse hotline discusses the situation with the employee and provides a report to the QA Manager while maintaining employee anonymity. A poster with the contact information for Lighthouse Services is displayed in the company's breakroom.

3. BUILDINGS AND FACILITIES

3.1 Facility

The Eurofins Air Toxics laboratory occupies approximately 27,000 square feet of space in Folsom, California, including 2,514 square feet of office space. The single-story building is custom-designed to suit the specifications of an air laboratory. Design criteria included floor plans to accommodate segregation of conflicting tests and provide an environment that is conducive for cross-functional work teams. The main instrumentation laboratory is based on an "open" concept in which walls were removed to promote a sense of community and teamwork. Wide hallways with alcoves were designed to encourage congregation and discussion. The number of private offices was minimized so that barriers between management and staff are absent. Elements of the quality system are evident throughout the facility design. The facility's map is available for review at the laboratory.

3.2 Security

Security at Eurofins Air Toxics is maintained through a controlled access system. Representatives of State, Federal, and private entities have access to the laboratory facility and records during normal business hours. Guests and employees must enter/exit through Sample Receiving or the reception area. All visitors must sign in and out upon arrival and departure. After work hours, the building is secured and linked to a commercial security agency. The security system is equipped with perimeter alarms, motion sensors, and speakers that monitor background sounds. Heat-activated fire alarms are monitored by an outside agency. A fire alarm also activates the security system. Security and controlled access protocols are described in SOP #30.

4. DOCUMENT CONTROL

4.1 Controlled Documents at Eurofins Air Toxics

It is Eurofins Air Toxics' policy to restrict the distribution of internal procedures to clients, and we discourage the distribution of company confidential documents outside of the facility. Clients are permitted to review our procedures while on-site as part of an audit or visit. Any documents that are distributed are only done so with the approval of QA.

4.1.1 Quality Policy Manual and Company Policies

Eurofins Air Toxics' Quality policies and Quality Systems must comply with all State and Federal requirements for those programs for which the laboratory maintains accreditation.

All Eurofins Air Toxics employees are required to read the Quality Assurance Manual within 30 days of release of the latest version and maintain current documentation in their Training Record binders and in the QA department. The Quality Assurance Manual is available to all employees electronically on a shared server located at O:\QA\LQAM.

4.1.2 Laboratory Standard Operating Procedures (SOPs)

The SOPs at Eurofins Air Toxics detail the work processes used on a regular basis that are to be conducted and followed within the organization. They document the way activities are to be performed to facilitate consistent conformance to technical and quality system requirements and to support data quality. These SOPs can be administrative or technical. All employees should maintain a record of review of the most current SOPs.

4.1.3 Work Instructions (at the department level)

The intent of these procedures or documents is to define in greater detail the specific "how to". The level of detail in these documents must be sufficient so any appropriately trained person can perform the task accurately.

4.1.4 Logbooks, Forms, and Instructions

The intent of these documents is to provide documented evidence to support Eurofins Air Toxics quality systems and operations. They are

used as part of regular laboratory operations to record necessary information.

4.2 Document Approval, Issue, Control, and Maintenance

The Quality Assurance Department is responsible for the approval, issue, control, and maintenance of all documents that are part of the laboratory's quality systems including, but not limited to, the Quality Assurance Manual (LQAM), Standard Operating Procedures (SOPs), Logbooks, Forms and Instructions, Certificates of Analysis (C of As), and calibration and training documents.

All documents issued to personnel in the laboratory as part of the quality system shall be reviewed and approved for use at a minimum by the Quality Assurance Manager and as needed by the Technical Director and Laboratory Director prior to use.

The LQAM and SOPs are reviewed to ensure they remain accurate and current. The frequency of review is either annual at the least or as needed, depending on the procedure. Upon generation of new or updated documents, all copies of obsolete documents are removed from the laboratory and its computer network, then archived or destroyed as appropriate. Pertinent staff members are notified of the updates. A new revision number is assigned to the LQAM or SOP at every review that results in updates.

All technical changes must have the approval of the Technical Director, the Laboratory Director, and the Quality Assurance Manager.

Detailed instructions regarding document control and how to write SOPs are available in SOPs #46 and #119.

4.3 Laboratory Logbooks and Forms

Procedures are in place to ensure that all data is traceable, authentic, complete, and retrievable. Logbooks, forms, and instructions are created and distributed by the Quality Assurance Department as needed. Used logbooks are returned to QA for archival. The QA Department maintains a master index to uniquely number and identify each logbook and form distributed. Logbooks can contain blank or preformatted pages. They are bound and uniquely identified, and have sequentially pre-numbered pages.

4.4 Archival and Storage of Documents

The majority of documents at Eurofins Air Toxics are stored electronically. Documents which remain in hard-copy format include chain-of-custody forms (COCs), Data Review Checklists, scanned packets (run logs, spectral defenses, manual integrations, etc.), FedEx/UPS air and freight bills, and most logbooks. All other hard-copy documentation is stored in its specific workorder folder. The hard-copy workorder folder is placed in a bar-coded storage box for long-term storage. Bar codes are maintained in an inventory log. An off-site company archives the boxes using the bar-coding system. The storage company provides one-day retrieval service upon request.

Used logbooks are returned to Quality Assurance for archival and remain in the QA Department for no less than five years.

5. SAMPLE HANDLING

5.1 Sample Collection

It is the responsibility of the client to submit representative and/or homogeneous and properly preserved samples of the system from which they are collected. In all cases, field sampling personnel are ultimately responsible for having expertise and knowledge in air sampling methodology or product/materials collection protocols sufficient to ensure that the defensibility of the data will not be compromised due to deficiencies in the field sampling, handling, or transportation. General information regarding the proper use of sampling media provided by Eurofins Air Toxics is available as a resource for field personnel. The laboratory provides sample containers, chain-of-custody forms, sampling labels, chemical ice packs (if appropriate), shipping containers, and custody seals (per client request).

Air sampling media provided by a qualified vendor or prepared by the laboratory for field use is certified for cleanliness. The laboratory's media cleaning process is typically verified using batch certification protocols. Individually certified canisters are also available per specific client request.

5.2 Sample Receipt and Entry

5.2.1 Sample Receipt

Samples can be received at the laboratory during normal laboratory operating hours. Receipt occurs in one of three ways:

- Commercial courier
- Eurofins Air Toxics courier service
- Personal delivery

Upon arrival at the laboratory, samples are received and inspected following Eurofins Air Toxics' Sample Acceptance Policy as outlined in SOP #50. This SOP establishes specific guidelines for sample acceptance, which are generally accepted practices under U.S. Environmental Protection Agency (USEPA), Department of Defense (DoD), ISO/IEC 17025:2017, and NELAP protocols.

5.2.2 Sample Entry

As soon as is practical after sample receipt, the samples are entered into LIMS. Samples awaiting log-in are stored in temporary holding areas, at appropriate storage conditions to maintain sample integrity.

At the time of entry, the LIMS system assigns a unique laboratory sample number to each sample. This number is sequentially assigned; a label is then generated and is attached to the sample container.

A sample acknowledgment in the form of a Sample Receipt Confirmation prints from LIMS for each sample delivery group (SDG), which is the same number as the workorder. This notification is sent to the client to confirm sample receipt and entry.

5.2.3 Sample Rejection Policy

Any time a sample is received in a condition that does not meet the method requirements, if there is doubt about the suitability of items received, if items do not conform to the description provided, or the testing required is not clear or specified, the condition of the sample is clearly documented on a Sample Discrepancy Report (SDR). The SDR is delivered to the Project Manager for review and communicated to the client as needed. Directions on next steps, which may include canceling the sample or proceeding with qualifiers and/or narrative, are documented on the SDR. Details are outlined in SOP #50.

5.3 Sample Identification and Tracking

A sample label is generated for each sample, and in addition to the assigned Eurofins Air Toxics' sample number the following information is printed on the label: workorder number, laboratory sample ID, and, if needed, a sample release

date. For canister analysis, the label is not affixed directly to the canister but attached with a tag.

To ensure traceability of results, the unique sample number assigned is used to identify the sample in all laboratory data documentation, including logbooks, instrument printouts, and final reports.

5.4 Sample Storage

After entry into LIMS, samples are placed in an assigned and identified storage location until needed for analysis. Room temperature, refrigerated, and freezer storage are available, and samples are stored in accordance with regulatory, method, or client directions. The LIMS system is used to assign storage locations for bar-coded media, which promotes orderly storage of samples. Sample storage locations for sorbent and condensate samples requiring refrigeration are monitored for accurate temperature control.

When a canister, bag, sorbent tube, or product sample is scheduled for analysis, the analyst obtains custody of the sample by scanning the canister tag or sticker bar code as well as the bar-coded destination location of each individual sample. The scanned information is electronically transmitted to LIMS to reflect the custody of canister and bag samples at all times. All other media samples are logged into the Internal Extractable Sample Tracking Logbook and the pertinent storage area.

5.5 Sample Return/Disposal

Samples are released for disposal upon satisfactory completion of analysis unless prior contractual arrangements have been made. The release of samples is electronically documented in the LIMS tracking system via scanning of the canisters and bags. This ensures verification of completion of all analyses including all samples in each workorder. Samples are released following the procedures outlined in SOP #63.

Sample disposal varies based on the sampling media. Whole air samples are vented through a charcoal scrubber, while liquid samples are disposed of according to procedures noted in SOP #24.

5.6 Chain of Custody

Samples received by the laboratory must be documented using a chain-of-custody (COC) form and relinquished following standard EPA-approved guidelines, including the following:

- Unique sample name or number
- Location, date, and time of collection
- Canister number (if applicable)
- Collector's name
- Preservation type (if applicable)
- Matrix or product type
- Any special remarks

Additional information may be required depending on the requested analysis.

A copy of the signed COC will be e-mailed to the client in conjunction with the Sample Receipt Confirmation.

Once a sample is received by the laboratory, the internal chain-of-custody procedure is followed.

Disclaimer: *Eurofins Air Toxics assumes no real or implied responsibility or liability for client-related field sampling and shipping activities. It is the responsibility of the individual client to ensure that referenced methodologies are followed with respect to sample collection and shipment to the laboratory. Air sampling media and equipment should only be used by experienced field engineers. It is the ultimate responsibility of the client to be knowledgeable both in sample preservation requirements as well as relevant State, Federal, and international shipping requirements. Any time a chemical substance is collected using Eurofins Air Toxics media, the client bears sole responsibility for understanding and abiding by the laws involving shipment of potentially hazardous substances by common carrier.*

6. TECHNICAL REQUIREMENTS – TRACEABILITY OF MEASUREMENTS

6.1 Reagents and Solvents

The reliability of Eurofins Air Toxics' analytical results can be directly affected by the quality of reagents used in the laboratory. Procedures are in place to control labeling, storing, and evaluation of these materials. All purchased supplies, reagents, solvents, and standards are verified as acceptable and meeting criteria for analysis prior to use. The Eurofins Air Toxics' Chemical Hygiene Plan (CHP) provides safety information in regard to the storage and handling of laboratory chemicals. All reagent certificates and Safety Data Sheets (SDSs) are retained by the laboratory (see section 2.8).

6.2 Calibration Standards

Written calibration procedures are required, where applicable, for all instruments and equipment used in the laboratory. The source and accuracy of standards used for calibration purposes are integral to obtaining quality data. Requirements for calibration are provided in each analytical method including specifications for the standard used. Calibration measurements made by the laboratory must be traceable to national standard of measurement (e.g., NIST) where available. Certificates of Analysis are maintained for each material, as applicable.

Standards are usually purchased from commercial suppliers either as neat (pure) compounds or as solutions with certified concentrations. The accuracy and quality of these purchased standards are documented on the C of A, and hard-copy certificates are maintained on file in the laboratory. Upon receipt at Eurofins Air Toxics, material is labeled with a date of receipt and stored appropriately.

Stock standard solutions are recorded in the proper standard logbook and are assigned a unique standard code number. When a working standard is prepared, the compound(s), standard code number, date prepared, analyst, expiration date, and solvent are noted in the working standard logbook. All working standards are kept in containers and at temperatures that will not alter their integrity. All containers are clearly labeled with concentrations, unique standard code number, and expiration date. Standards are not to be used in the laboratory past their expiration date.

6.3 Equipment and Instrumentation

The laboratory is equipped with all equipment and instrumentation required for testing the scope of work it supports. All equipment and instrumentation is maintained in proper working order. Eurofins Air Toxics' major equipment capabilities are summarized in the table below:

Major Instrumentation

Number	Instrumentation
29	GC-MS
2	Gas Chromatographs with various detectors (TCD, FID)
2	TOF
3	Entech Air Concentrators
7	Markes Air Auto-Samplers
12	Markes Automated Thermal Desorption Units
3	Liquid Auto-Samplers
1	Extractor

1	Precision Diluter
1	Industrial Air Compressor

6.3.1 General Requirements

- Equipment and instrumentation are assigned a unique identifier designation to identify them within the data documentation.
- An equipment logbook is established in conjunction with installation and is readily available to document all incidents that pertain to the equipment and instruments as they occur.
- All test, measuring, and inspection of laboratory systems, equipment, and instruments used at Eurofins Air Toxics are routinely calibrated and maintained in accordance with applicable Standard Operating Procedures.
- Trained technical staff, or a designated individual, performs routinely scheduled maintenance and calibration of laboratory equipment as required by laboratory procedures. These activities are documented.
- If appropriate standards or expertise for calibration or maintenance are not available in-house, the operation is conducted by an outside service firm.
- All equipment taken out of service is tagged accordingly.

6.3.2 Standard Operating Procedures

Information regarding operation, maintenance, and calibration of equipment and instrumentation are found in respective SOPs. The procedures include a routine schedule for preventative maintenance and calibration as applicable, along with acceptance criteria and remedial action to be taken in the event of failure. These procedures are maintained in the document control system and reviewed on a regular basis to verify they remain current and accurate. Equipment manuals are also available to provide additional information with regard to operations and maintenance.

6.3.3 Maintenance

- Equipment maintenance is performed as either a preventative or corrective operation.
- Preventative maintenance procedures and schedules for each piece of equipment are assigned where applicable. Preventative maintenance operations are performed by an analyst, scientist, senior scientist, or contracted manufacturer's representative or service firm personnel. Documentation is maintained for the procedures performed

as part of the preventative maintenance operation. It is the responsibility of Supervisors to ensure that a preventative maintenance schedule is addressed by a procedure where appropriate and is followed.

- A supply of commonly needed replacement parts is maintained by the laboratory.

6.3.4 Calibration

- Calibration is the establishment of, under specified conditions, the relationship between the values/response indicated by a measuring instrument or system and the corresponding known/certified values associated with the standard used. Some types of calibrations are performed within a set of frequency (e.g., daily), while others provide intermediate checks to ensure that the instrument response has not changed significantly.
- All measuring and testing equipment having an effect on the accuracy, precision, or validity of calibrations and tests are calibrated and/or verified on an ongoing and routine basis. Methods for calibration of instruments and equipment vary widely with the nature of the device and the direction given by analytical procedures, department procedures, or manufacturer recommendations. Frequency of calibration can also depend on additional factors, including robustness of the instrument or equipment and the frequency of use.
- Calibration information is recorded in a logbook that is associated with the instrument/equipment and/or a calibration certificate is maintained and/or data printouts are generated to document the activity.
- Calibration measurements are traceable to national standard of measurement (e.g., NIST) where available. Physical standards, such as NIST-certified weights or thermometers are re-certified on a routine basis. Calibration certificates are maintained on file, where applicable, to indicate the traceability to national standard of measurement.
- Calibration failures are documented in the logbook for the instrument and/or within the data printouts from the instrument.
- After repair, adjustments, or relocation that could affect instrument response, calibration/verification activities are performed, as applicable, before the unit is returned to service.
- Analytical data is not reported from instrumentation or equipment that fails to meet calibration requirements.

6.4 Computerized Systems and Computer Software

6.4.1 Computer Usage

Eurofins Air Toxics provides computer equipment for employees to use as a tool in performing their work. Computer equipment is the property of Eurofins Air Toxics and is to be used in accordance with defined terms and conditions. The laboratory's goal is to provide standard hardware and software that meets the needs of the user.

6.4.1.1 Physical security of computer systems: It is company policy to protect computer hardware, software, and data documentation from misuse, theft, unauthorized access, and environmental hazards. All of the laboratory servers are housed in a locked office, which maintains favorable environmental conditions to allow for optimal server performance. Access to the laboratory's networks is granted by the Corporate IT Team. Network access is tightly controlled for the entire company. Users maintain individual network accounts and are allowed to access specific areas of the network based on the privileges assigned to them. A user is granted access to only those areas needed to fulfill his or her job function.

6.4.1.2 Passwords: All software used to reduce sample data or generate sample reports is password-protected; users are granted rights to these systems based on a "read/write/none" privilege system. The following procedures apply regardless of what system(s) is being utilized:

- Passwords must be kept confidential.
- Users must log-out of a system when not in use to prevent unauthorized access.
- Forgotten passwords can only be reset by the Corporate IT Team.
- Network passwords automatically expire every 180 days. The computer prompts a user to change the password when the expiration date nears.

6.4.1.3 Computer viruses: The Corporate IT Team continuously monitors its computer network for computer viruses. Anti-virus software is employed to detect viruses on the Windows network. Employees must report any virus concerns to Corporate IT as soon as possible. Employees who share files between their home computer and the laboratory should install anti-virus software on their home computer. If an employee does not have such software, the laboratory can suggest various no-cost anti-virus software products.

6.4.1.4 Internet and e-mail System: The e-mail system is used primarily for Eurofins Air Toxics business purposes. The Employee Handbook provides additional information in regard to system usage. Employee access to the Internet is restricted to those employees who have a business need for it. All employees have access to e-mail. All Internet and e-mail activity is subject to monitoring. All messages created, sent, or received over the Internet are property of Eurofins Air Toxics and can be regarded as public information. E-mail and Website filtering software is utilized.

6.4.1.5 Software Policy:

Eurofins Air Toxics' Software Policy is as follows:

- Copyright laws protect software, and Eurofins Air Toxics' intent is to abide by all software agreements.
- Software purchases must be formally requested and approved by management, Corporate IT, and/or validation personnel, as necessary.
- All software is used in accordance with applicable license agreements.
- Employees are not to install any software on computer(s) unless authorized by Corporate IT.
- Employees must not give software to outsiders (e.g., clients, contractors, etc.), unless approval is granted by management.
- Users must not make copies of any licensed software or related documentation without permission. Any user that illegally reproduces software is subject to civil and criminal penalties including fines and imprisonment.

6.4.1.6 Computer system backup, data restoration, and data archival: All data systems are backed up on a daily, weekly, and 6 month basis using a modified "grandfather-father-son" (GFS) rotation protocol. Specifically, these backups are conducted on the servers responsible for all laboratory production data files and databases (i.e., Project Management files, analytical data, audit trails, Quality Assurance documents, etc.). A daily incremental backup is scheduled to run each night Monday through Saturday. The daily incremental backup is limited to files modified the same day. On Sunday, a weekly full backup of all files on each server is completed. At the end of 6 months, a full backup of each data system is conducted. This monthly backup tape is then placed in permanent storage. The permanent historical backup tapes are stored in an off-site data storage facility. Data is not removed from the server until the server achieves 85% capacity. In addition, before removing data off the server another compressed copy is stored on to another server where this server is duplicated off-site. A more comprehensive description of the laboratory's electronic data archiving system can be found in SOP #55.

6.4.1.7 Remote access to computer systems: With special permissions, employees are able to remotely connect to the laboratory computer network through a VPN system. When logging in, users are authenticated with their Windows account and password.

6.4.2 System and software verification: Before each new computer system or significant modification of an existing system is implemented in the laboratory, the following requirements must be met:

- Required documents – Describe the required system functionality and specification (e.g., Software Development Change Control, Change Control Log, IT Logic New Rule or Rule Update)
- Design documents – System overview, screen design, report layout, data description, system configuration, file structure, and module design
- Testing documentation for system development/verification – structural testing of the internal mechanisms and user testing of the installation and system qualification.

7. PURCHASING EQUIPMENT AND SUPPLIES

7.1 Procurement

The primary materials procured by the laboratory are analytical instrumentation and software, media and reagents including standards, carrier gases and cryogens, miscellaneous laboratory supplies, computer hardware and software, and service contracts.

Control of the purchase of these items and services is maintained using a standard purchase order system described in SOP #105 and briefly outlined below:

- Purchase requests must be approved by a director or manager.
- A request for purchasing through the COUPA system must be completed in order to initiate purchase.
- An evaluation of the supplier is conducted to determine whether it has been deemed a qualified vendor.
- Once the requesting agent completes the order from in the COUPA system, the purchasing agent will place the order with the vendor.
- Requires that upon receipt or delivery of services the product is inspected by the requesting/purchasing agent and compared to the packing slip and/or request for services.
- The Eurofins National Service Center (NSC) purchaser matches each purchasing request with the invoice prior to payment to insure that purchased items or services were delivered as expected.

Purchasing documents are maintained by the Accounting Department, calibration certificates are maintained by the Quality Assurance Department, and Certificates of Analysis for reagents and media are maintained by laboratory personnel.

7.2 Supplier Evaluation

Suppliers and vendors are evaluated in accordance with SOP #105 to assure that the quality of the products purchased meet the quality expectations of Eurofins Air Toxics, LLC and do not interfere in the quality of testing. A laboratory database is maintained with a list of approved vendors.

8. ANALYTICAL METHODS

8.1 SCOPE OF TESTING

Soil vapor, indoor and outdoor ambient air, and other types of air-phase samples are analyzed in accordance with official published methods or validated in-house methods. Method modifications made by Eurofins Air Toxics, LLC are detailed in a summary of modifications table in the method SOP. Our capabilities extend from trace level measurements required for indoor air testing to identifying and quantifying organics in high-level sources.

The methods used by Eurofins Air Toxics are approved by a broad range of regulatory agencies.

A list of methods covered under the laboratory's NELAP accreditation can be found in the table in section 8.2.

Eurofins Air Toxics specializes in and has expertise with the following types of projects:

- Vapor Intrusion investigations
- Environmental assessments
- Remediation system monitoring (soil vapor extraction)
- Soil vapor
- Ambient air monitoring
- Indoor air quality (IAQ)
- EPA 325-Fenceline Monitoring

Appendix E contains summaries for each commonly performed analytical procedure in the laboratory. Each summary contains the following information:

- A brief method description
- Laboratory variances to method compendium or other regulatory reference methodologies
- Tables containing analyte lists, Reporting Limits (RLs), Limits of Quantitation (LOQs), and quality control (QC) acceptance criteria
- A table of calibration and QC procedures

This Quality Assurance Manual references methods in a general manner; specific procedures used by the laboratory can be found in the method-specific SOPs.

8.2 Analytical Test Methods

Eurofins Air Toxics' NELAP accredited analytical methods, parameters, instrumentation, sampling media, holding times, and SOP numbers are summarized in the table below:

Method	Parameter	Type	Sampling Container	Holding Time in days	Eurofins Air Toxics SOP #
TO-14A/TO-3	BTEX/TPH	GC/FID/PID	Summa Canister Tedlar Bag	30 3	43
TO-12	Non-methane Organic Carbon (NMOC)	GC/FID	Summa Canister Tedlar Bag	30 3	36
TO-13A	PAHs/ Semi-volatiles	GC/MS	XAD/PUF	7	10/74
TO-14A/TO-15	VOCs	GC/MS	Summa Canister Tedlar Bag	30 3	6/38/83/91/ 132/134
TO-17	VOCs	GC/MS	Sorbent Tube	30	109/ 112
ASTM D-1946	Fixed Gases, CH_4, C_2+	GC/TCD/FID	Summa Canister Tedlar Bag	30 3	08
ASTM D-1945	Fixed & Natural Gases	GC/TCD/FID	Summa Canister Tedlar Bag	30 3	54
PM10/TSP	Particulate Matter	Mass	Quartz Filter	14	66
EPA 325B	BTEX + Styrene	GC/MS	Sorbent Tube	30	131
TO-17 – Passive Samplers WMS and Radiello 130	VOCs	GC/MS	Sorbent Tube	30	100
TO-15 HSS	VOCs	GC/MS	Summa Canister	30	133

Method	Parameter	Type	Sampling Container	Holding Time in days	Eurofins Air Toxics SOP #
MADEP APH	VOCs	GC/MS	Summa Canister	30	42

8.3 Method Validation

As part of the initial test method evaluation for new standard methods, analytical runs must be performed the same way an analyst would perform an initial Demonstration of Capability (DOC) to evaluate precision and bias along with a Method Detection Limit (MDL) study as applicable.

Non-standard methods, including laboratory-developed methods, standard methods outside their intended scope or application, and requested changes to existing instrumentation will follow a planned process explained in detail in SOP #107 and outlined below:

- Measurement Quality Objectives (MQOs) – should be clearly outlined prior to validation.
- Development of Test Plan – Technical Director and assigned personnel are responsible for the development of such plan.
- Validation – Implementation of the test plan with documentation of all results will be reviewed by the Technical Director.
- Review and Approval – Review of performance against the MQOs, supporting documents, and written procedures is performed by the Technical Director. After approval, the QA Manager reviews for completeness and finalizes the method for production.

8.4 Procedural Deviations

Eurofins Air Toxics communicates and addresses procedural deviations in the following ways:

- Modifications to standard methods made by Eurofins Air Toxics are detailed in a summary of modifications table in the analytical method SOP. The modification table is also included in the laboratory narrative of the final data report.
- Differences between a project request and laboratory standard protocol are documented in a variance table created by the Project Managers, Quality

Assurance Manager or Technical Director for submission with the proposal to the client. Agreement is documented by the client's initials and date in the approval column or with written documentation from the client that all variances have been approved.

- If a sample received did not meet the established criteria for quality testing, the Sample Receiving Department will issue a Sample Discrepancy Report (SDR), and the Project Manager will communicate the discrepancy to the client. If the client still wants the sample to be processed, the discrepancy will be narrated in the final report.
- Other analytical procedural deviations that are within allowable variations established for every method and listed in the method SOPs are discussed with the client, and if accepted the sample results will be reported with a narrative of the deviation and the affected result will be flagged accordingly.
- Analytical procedural deviations that are not within allowable variations and directly affect the sample result will require the initiation of a Corrective Action Report request.

The Corrective Action Program is explained in detail in section 12 of this Quality Manual.

9. INTERNAL QUALITY CONTROL CHECKS

9.1 LABORATORY QUALITY CONTROL SAMPLES AND ACCEPTANCE CRITERIA

9.1.1 Blanks: for the whole air methods for which no sample preparation step is required, a blank is a designated sample designed to monitor for contamination originating from the analytical system. The Laboratory Blank is comprised of clean, humidified air or nitrogen. A Laboratory Blank is analyzed after any applicable standards and prior to the analysis of project samples. A blank is also analyzed in the event saturation-level concentrations are incurred to demonstrate that contamination does not exist. The blank and the field samples are treated with the same internal standards and surrogate standards and carried through the entire analytical procedure. For methods requiring a sample preparation step (e.g., TO-13A), a Laboratory Blank is prepared using un-sampled media and extracted alongside the batch of field samples. Ideally, blanks demonstrate that no artifacts were introduced during the preparation and/or analysis process. The specific acceptance criterion for each test is given in the analytical method and is usually based on the required Reporting Limit (RL).

9.1.2 Surrogates: Surrogates are organic compounds that are chemically similar to the analytes of interest but are not naturally occurring in environmental samples. For GC-MS methods and some GC methods, the recovery of the surrogate standard is used to monitor for unusual matrix effects and gross sample processing errors, and to provide a measure of recovery for every sample matrix. When required by the analytical method, surrogates are spiked into all the field and QC samples to monitor analytical efficiency by measuring recovery on an individual sample basis. The percent recovery is determined and compared to the acceptance criteria. Acceptance criteria limits are set as required by the method or based on a statistical determination from laboratory data.

9.1.3 Matrix Spikes: Matrix spikes are not required QC for whole air samples collected in Summa canisters. Accurately spiking target compounds into an evacuated canister prior to deployment in the field for sample collection or post-sample collection is neither practical nor technically appropriate. Therefore, matrix spiking is performed only on samples submitted as part of a sampling train, such as condensates, or on extractable samples, provided they are submitted in duplicate for matrix spike and in triplicate for the matrix spike duplicate. It is the responsibility of the client to provide additional samples to fulfill any method requirements regarding matrix spikes. When applicable, matrix and matrix duplicate spiking is performed using a subset of target analytes. Recoveries and demonstrated reproducibility values that do not meet the acceptance criteria are flagged and explained in the laboratory narrative.

9.1.4 Laboratory Control Samples: Laboratory control samples (LCS) are samples of known composition that are analyzed with each batch of samples to demonstrate laboratory accuracy. The LCS is prepared by fortifying clean matrix with known target concentrations. In the case of non-extracted batches, the LCS is generally analyzed daily prior to sample analysis, but could also serve as an end check standard. Percent recovery is calculated and compared to acceptance criteria, which are set as required by the method or based on a statistical determination from laboratory data.

9.1.5 Sample Duplicates and Laboratory Control Sample Duplicates: A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test. To compare the values for each compound, the relative percent difference (RPD) is calculated by dividing the difference between the numbers by their average. Precision for analytes that are not typically found in environmental samples is

determined by analyzing a pair of Laboratory Control Samples (LCS), and comparing the RPD for the spiked compounds. The acceptance criteria are described as a maximum for the RPD value as required by the method or based on a statistical determination from laboratory data.

9.1.6 Internal Standards: Internal standards (IS) are organic compounds that are chemically similar to the analytes of interest but are not naturally occurring in environmental samples. For extractable methods and when required by the method, IS are added to every field and QC sample typically after extractions but prior to analysis. For all GC-MS methods an IS blend is introduced into each standard and blank to monitor the stability of the analytical system. Comparison of the peak area of the IS is used for quantitation of target analytes. The IS peak area and retention time also provide a check for changes in the instrument response and chromatographic performance. The acceptance criteria are stipulated in the analytical method.

9.1.7 Second Source Check: A second source check is analyzed using either the Laboratory Control Sample (LCS) and/or an Initial Calibration Verification (ICV). The second source is a standard that is made from a solution or neat compound purchased from a different vendor than that used for the calibration standards. For some organic custom mixes, the same vendor but a different lot and preparation is used. This ensures that potential problems with a vendor supply would be evident in the analysis. Some areas of the laboratory use continuing calibration verification standards as a second source from the initial calibration.

9.2 Quality Control Sample Frequency and Corrective Action

Each analytical method defines the frequency for required quality control (QC) samples. A summary is provided in Appendix E. The corrective action required when a QC result fails to meet acceptance criteria is also given. If the method reference requires the use of specific limits, the laboratory uses the published limits that are documented as part of the analytical method. Many methods require that each laboratory determine their own acceptance criteria based on statistics from performance of the method. In these cases, the limits are available to the analyst and are entered into the laboratory computerized QC system described in SOP #48. Statistically determined acceptance criteria are frequently subject to change as the laboratory recalculates its control limits. Due to their dynamic nature, acceptance criteria are not included in this manual.

9.3 Quality Control Charts

Quality control (QC) results entered into the computer are used to generate control charts that are plotted via computer and can be accessed at any time by all analysts and by the Quality Assurance Department. The system charts results from surrogates and laboratory control samples. These charts provide a graphical method for monitoring precision and bias over time. The computerized quality control system is used to report QC data to clients and to collect data for assessment of precision and accuracy statistical limits.

9.4 Measurement Uncertainty

As stated in ISO/IEC 17025:2017, "All contributions that are of significance, including those arising from sampling shall be taken into account using appropriate methods of analysis" (7.6.1).

This means the laboratory must determine the uncertainty contribution of all steps in the testing process such as equipment, calibration, standards, reagents, preparation, etc. Since, in most methods, the laboratory control sample (LCS) goes through the entire process of preparation to analysis, all factors that would contribute to uncertainty is evident through the LCS results. As such, LCSs are performed with every batch of samples where appropriate for the method.

Measurement uncertainty is calculated as two times the standard deviation of the LCS recoveries for the group and date range of data points selected for all applicable methods. This is reported as a percentage. At this point, it is not necessary to apply or report the uncertainty determination with sample results. When a client requests the measurement uncertainty it is applied by multiplying the determined analyte concentration by the uncertainty percentage.

10. ASSURING QUALITY OF TEST RESULTS

10.1 Data Management

At a minimum, data management is initiated when Eurofins Air Toxics receives samples from the client. More often, the process begins with client communication of their needs and requirements for a specific project and/or testing. The Project Managers are responsible for entering this information into the client services modules of LIMS. Upon receipt of the samples, a unique tracking number is generated based on this information in the project profile. At this point, computer technology becomes an integral part of tracking the samples through laboratory operations.

10.2 Data documentation

Analytical data generated in the laboratory is collected through the associated data system or is manually documented in bound logbooks. Analysts review data as it is generated to determine that the instruments and systems are performing within specifications. If any problems are observed during an analytical run or the testing process, corrective action is taken and documented.

Procedures are in place to ensure that all data is traceable, authentic, and complete. The following general requirements outline the Eurofins Air Toxics' system for logbooks, notebooks, and documentation recording:

- Observations, data, and calculations are recorded at the time they are made and are identifiable to the specific task.
- Entries are legible, signed, and dated.
- Errors are corrected in a manner that does not obliterate the original entry, initialed, and dated.
- Blank pages or substantial portions of pages which are left blank are crossed out to eliminate the possibility of data entry at a later date.
- Logbook pages and instrument printouts are signed and dated to indicate completion.
- At periodic intervals the Quality Assurance Department checks equipment/instrument logbook entries and temperature recordings for completeness, legibility, and conformance to procedures.
- At a minimum, the following is recorded as part of data documentation:
    - Date of analysis/operation
    - Initials/date of analyst performing test/operation
    - Identification of client sample(s) and material(s) analyzed
    - Materials, reagents, and standards used to perform the test/operation
    - Method used to perform test/operation
    - Equipment/instrumentation used to perform test/operation
    - Deviations, planned or unplanned, from the analytical method
    - Signature/date of person reviewing data documentation
- For computer-generated data, the following information is recorded:
    - Samples(s) analyzed/operations performed
    - Date of analysis/operation
    - Unique instrument identification

- Name or initial/date of person operating the instrument
- Name or initial/date of person reviewing data
- Any manual notation, interpretations, or integrations made on instrument printouts are signed, dated, and reviewed.

10.3 Data Calculations

Most instruments either include or are connected to a data system programmed to perform calculations needed to reduce the raw data to a reportable form. All calculations are maintained in the method SOPs and/or work instructions.

In many cases, data from the local instrument system are uploaded directly to LIMS for review and reporting. This direct upload eliminates the need to re-type data and any associated source of transcription errors from the analytical scheme. In those cases where the raw data from analytical measurements need to be manually entered into the LIMS, the data is transcribed from the logbook or controlled form directly into the LIMS report. Review of the manually entered results is completed if the analyst has not completed training in data review.

Some instruments report data that require application of additional factors before the data is in final form. Analysts input these additional factors into the laboratory sample management system, where final calculations are performed.

10.4 Reporting Limits

It is important to ascertain the Limit of Quantitation (LOQ) that can be achieved by a given method, particularly when the method is commonly used to determine trace levels of analyte. The USEPA has established one method for determining Method Detection Limits (MDLs) from which LOQs can be extrapolated, which is summarized in the laboratory procedures.

MDLs are verified or determined annually on each instrument and are the basis for the LOQ used in the default reporting format. Because MDLs change each time they are re-evaluated, they are not included in this manual but are available at the laboratory and available to clients upon request.

Methods and compounds that are included on the U.S. Department of Defense (DoD) scope of accreditation require quarterly analysis and evaluation of the LOQ and determination of Limit of Detection (LOD). The LOQ evaluation entails the calculation of precision and accuracy at the LOQ or Reporting Limit. The LOD for each compound is determined by analyzing a calibration standard or set of standards between the MDL and LOQ. The LOD is assigned the concentration at which the peak meets the signal-to-noise criteria.

The Reporting Limit used to determine whether a result is significant and reported as detectable is dependent upon agency and client requirements. A variety of formats are available and include use of the MDL, LOD, LOQ, method-specified limits, and project-specific limits.

10.5 Data Review

Final review and verification of the data is performed by a trained analyst or scientist using the sample results and quality control information entered into the laboratory sample management system. Another tool used for data review involves the use of proprietary in-house data validation software to review every data point generated and to alert the reviewer when manual integrations occur. The software is also programmed to report each analyte that does not meet acceptance criteria in the quality control and/or sample(s).

After determining that all necessary requirements for valid data are met, the reviewer electronically approves the data by updating the “Report Approved By” status with their initials. This action applies the electronic signature of the Technical Director. The computer is programmed with a list of approved reviewers for each test, and the system is password-protected to ensure that only qualified individuals verify the data.

10.6 Data Qualification

Data qualifiers are used to provide additional information about the results reported. The most typical use for data qualifiers is for results that fall below the quantitation limit. The data systems used to generate and report results are programmed to flag values in this range as estimated.

Other qualifiers are applied to advise data users of any validation issues associated with the data. The laboratory makes every effort to meet all of the requirements for generation of data. Occasionally, data is generated that does not meet all the method requirements due to sample matrix or other analytical problems. If the test cannot be repeated, or re-analysis would not yield more useable data, qualified data is reported. Qualifiers can be in the form of comments on the analytical report or flags applied to the results.

10.7 Data Reporting

When each analysis is completed, reviewed, and verified, a report is generated. The client receives a copy of the report containing the results of the analysis, plus comments added by the analyst when necessary. The report contains the

electronic signature of the Technical Director. Copies of the reports and associated supporting raw data are retained in the Eurofins Air Toxics' archives.

Eurofins Air Toxics offers a variety of data levels and formats, from a basic report of sample and QC results only (Level II) to a comprehensive data package including all supporting quality control information and raw sample data (Level IV). The client directs the selection of report type. Various electronic formats are also available, formatted to client-specific file structure and sent via e-mail, direct upload, Website access, or commercial courier.

Client confidentiality of Eurofins Air Toxics' Web data is ensured by the use of a secured firewall Internet environment coupled with the use of a user ID and password to gain log-in access to the system.

If amendments to a final report are required due to omissions, errors, or additional requests, a workorder reissue is initiated. All reissues receive a unique workorder number to distinguish them from the original issue. Reissued reports require a reason for the reissue and date of the reissue in the laboratory narrative. The laboratory maintains all supporting documentation for the revision including corrections, additions, or deletions relative to the original report.

10.7.1 Reporting the Results

Analytical reports are printed with a cover page that summarizes all samples in that group. This page lists the Eurofins Air Toxics' assigned sample number and the corresponding client description. The cover page identifies the laboratory contact person's name and the laboratory's phone number in case there is a question about the report. Within this package, each page is uniquely identified and paginated. Analytical test results which meet all the requirements of NELAP and ISO/IEC 17025:2017 are noted as so in the footer of the summary cover page.

10.8 Data Storage, Security, and Archival

Eurofins Air Toxics has documented procedures and instructions for the identification, collection, access, filing, storage, maintenance, and disposal of data records. Records are in the form of hard-copy paper records, electronic data files, and magnetic tape,.

Eurofins Air Toxics maintains records to demonstrate conformance to specified requirements and the effective operation of its quality systems. Records are stored and maintained in such a way that they are readily retrievable in facilities that provide a suitable environment to minimize deterioration or damage and

prevent loss. Retention time for the records is in accordance with NELAP's minimum five-year requirement and/or specific procedures or instructions. Prior to the destruction of data/records, and if requested by a client or agency, the laboratory will notify the client/agency that their data is scheduled for destruction so arrangements can be made to have the original data sent to the client.

If specified in client contract(s), archived records are transferred according to their instructions in the event of a change in laboratory ownership or if the laboratory goes out of business. If not specified by the client, the sale agreement must require that archived records be maintained as scheduled by the new owners. In the case of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed.

The laboratory maintains all documentation necessary for historical reconstruction of data, as follows:

- Analysis reports
- Data logbooks
- Instrument printouts
- Correspondence and client files
- Instrument and equipment logbooks
- Quality Assurance records
- Corporate documents
- Electronic records

11. AUDITS AND INSPECTIONS

11.1 Internal Quality Assurance Audits

Internal audits are performed by trained Quality Assurance personnel following a schedule planned yearly by the Quality Assurance Manager or at any time by the request of management. The audits cover all quality systems including but not limited to documentation practices, training, and adherence to current SOPs and methodology.

The following areas are identified to be audited by Quality Assurance:

a. Operations
b. Support Services

c. Sample Receiving and Login
d. Project Management and Sales
e. Information Technology (IT)
f. Quality Assurance

A written report with findings, observations, and/or recommendations is presented to the audited personnel, the Supervisors, and management by the auditor. Responses to findings and observations are then submitted to the Quality Assurance Department within 30 days.

All audit notes, documentation, and reports are either scanned and filed on the QA network drive or maintained as a hard-copy and filed in the QA department.

11.2 Management Review System

A review of the laboratory's systems is performed by senior management on an annual basis to evaluate effectiveness, identify areas requiring improvement, and establish timelines and accountability in addressing agreed-upon action items. This review includes internal assessment of the quality program and laboratory operations and external assessment through client feedback and audits. The management report (Form #F2.28) is generated by management or designated personnel and includes the following:

11.2.1 **Quality Assurance Status:** Summarizes the results of internal and external assessments, the numbers and types of Corrective Action Reports (CARs) generated, status of any outstanding CARs, a summary of client inquiries received, proficiency tests (PT) results, and training.

11.2.2 **Production Status:** Summarizes performance against key metrics such as turnaround time, details changes in sample mix and sample numbers, the number and types of reissued sample reports, outlines resource needs, and equipment performance.

11.2.3 **Client Assessment:** Summarizes feedback from clients based on daily communication with project management and sales team as well as feedback collected by a third party as part of our Client Satisfaction Index (CSI) determination.

The report and record of the meeting is stored on a secure drive with management-only access for a minimum of five years.

11.3 Client Audits and Agency Inspections

Clients may audit our facility as assurance that their objectives are being met and that the laboratory is compliant with all applicable regulations, data quality, and project requirements.

Client audits can range from a laboratory tour to an intensive inspection of technical operations, procedures, regulatory compliance, and/or review of specific projects. Clients can only review data that pertains to their projects, and a non-disclosure agreement must be signed as per SOP #99.

Inspections can be performed by investigators or auditors from the USEPA, DoD, state and other regulatory agencies, third party accreditors (ANAB), or regulatory agencies outside of the U.S.

The Quality Assurance Department is assigned the responsibility of hosting and working with agency and client representatives.

The Quality Assurance role includes:

- Escorting the investigator(s)
- Ensuring all questions are answered promptly and accurately
- Making note of all unresolved issues
- Informing management of the audit status and outcome
- Responding to the audit report
- Ensuring that appropriate corrective action is completed

11.4 Proficiency Testing Program

11.4.1 Proficiency Testing Samples (TNI/DoD)

Proficiency testing (PT) samples are used to measure analytical accuracy, precision, and report completeness. To be accredited under NELAP and DoD-ELAP, the laboratory contracts with an outside approved PT sample provider in each field of testing (FOT). Testing is limited by availability of samples that meet NELAP and DoD-ELAP criteria (noted below). The provider must be a TNI and DoD approved PT provider. It may be necessary to participate in more than one proficiency testing program to be evaluated for multiple interdependent analyte groups. Currently, Eurofins Air Toxics participates in PT programs for EPA Method TO-15, which is ISO/IEC 17025:2017 compliant, TO-15 SIM,

TO-3, TO-13A, TO-14A, TO-17 and 325B. In each calendar year, the laboratory will complete a minimum of two PT samples for each analyte or interdependent analyte group.

The following policies apply to laboratory PT sample analysis and reporting:

- The samples shall be analyzed and reported to the PT provider within 45 calendar days of receipt or the specific deadline specified by the PT provider.
- The PT sample is received and logged into an electronic sample receiving database in the same fashion as field samples.
- The laboratory must follow the PT provider's instructions for preparing the PT sample.
- The laboratory management and bench chemist ensure that the PT samples are prepared, analyzed, and reported in the same fashion as field samples using the same staff, equipment, and methods.
- Initial and continuing calibrations for the PT sample are analyzed at the same frequency of field samples.
- The PT sample cannot undergo duplicate or replicate analyses that would not ordinarily be performed on field samples. The PT sample result cannot be derived from averaging the results of multiple analyses unless specifically called for in the reference method.
- The PT sample can only be analyzed on equipment leased or owned by the company and handled only by bona fide employees of the company.
- The analysis of PT samples by temporary or contract employees is explicitly forbidden.
- The laboratory shall not subcontract any PT sample or portion.
- The laboratory shall not knowingly receive any PT sample or portion from another laboratory.
- The laboratory shall not communicate in any fashion with another laboratory concerning the PT sample or results.
- The laboratory shall not attempt to obtain the PT sample result prior to reporting.
- The PT sample reporting forms provided by the sample provider will be used to report the results and will be maintained in the laboratory's record system.
- The laboratory shall maintain copies of all written, printed, and electronic records relating the analysis or reporting of the PT sample

for a period of five years or as required by the applicable regulatory program.

- A CAR will be generated any time an analyte result fails the PT assessment. A copy of the PT results will be sent to the accrediting agency, and associated corrective action summary will be sent upon request.
- The laboratory authorizes provider to release any PT assessment information to the accrediting agency.
- The QA Manager must sign the PT results form and, by so doing, attests that the sample was analyzed and reported in the same fashion as a field sample and followed the PT provider instructions for preparation.
- The laboratory must notify its primary accrediting agency and any other agencies under reciprocity that it has enrolled with a particular PT provider.
- The laboratory must notify its primary accrediting agency and any other agencies under reciprocity in the event it wishes to change PT providers.
- For each analyte or interdependent analyte group for which proficiency is not available, the certified laboratory will establish, maintain, and document the accuracy and reliability of its procedures through a system of internal quality management.
- Results of any failed PT samples are summarized in the Yearly Management Review report.

11.4.2 Proficiency Testing Samples (Non-NELAP/DoD)

Occasionally proficiency testing (PT) samples are submitted along with field samples by private clients. The laboratory processes and reports the samples in the same fashion as field samples. When the client notifies the laboratory that one or more analytes appear to have failed, the report is processed through the normal Client Inquiry Corrective Action Process. The QA Manager will carry out an assessment and investigation into the circumstances surrounding the proficiency results, including aspects relating to how the client prepared the sample for submission. The outcome of the assessment will be documented as a CAR and maintained on file for a period of five years. Results of any failed external PT samples are summarized in the Yearly Management Review report.

12. CORRECTIVE AND PREVENTIVE ACTION

12.1 Laboratory Investigations and Corrective Action

The Quality Assurance (QA) Department manages the Corrective Action Program and maintains the Corrective Action tracking database using the c.Support software program. A Corrective Action Report is initiated any time sample results are affected by non-conformance with established SOPs or program requirements, any time an external assessment results in a finding, any time there is a failed proficiency evaluation sample, and when a client inquiry results in a quality finding. The expectation is that any CAR should be resolved within 30 days.

The client is notified if there is an issue that could potentially affect the quality of sample results. The communication with the clients is recorded.

The software program tracks all parts of the CAR system: root cause investigation, immediate corrective action, long-term corrective action, and preventive action. It also tracks client communications regarding the incident. The QA Manager reviews all opened CARs for completeness and resolution.

Detailed information about the CAR process is described in SOP #61.

13. SERVICE TO CLIENTS

The Project Management System is defined in SOP #1. The following are brief descriptions of the elements comprising project management systems.

13.1 Review of Work Requests, Tenders, and Contracts

Eurofins Air Toxics places great importance on understanding client requirements for a project. The laboratory ensures, to the best of our ability, that client and project requirements are outlined and understood prior to acceptance of the project, including required laboratory accreditations and nonstandard work requests. All inconsistencies are discussed and addressed with both the client and the technical laboratory staff before the project is initiated and samples arrive. This is achieved in various ways, including the review of client work plans, Request for Proposals (RFPs) project Quality Assurance Project Plans (QAPPs), requested analytical methods and protocols, business contracts, and quality agreements. A key client contact is assigned to oversee each project. Communication between the client and Eurofins Air Toxics technical staff is coordinated through the Project Managers. The Project Management group relays any project changes or modifications to the Laboratory Director and

designated technical staff. They also relay issues encountered by the laboratory back to the client.

13.2 Timely Delivery

Evaluating laboratory capacity, assignment of resources, and ability to perform specific projects is a joint responsibility between the Technical Director and the Laboratory Director. Eurofins Air Toxics recognizes that one of the most important aspects of the services offered is turnaround time.

To ensure timely delivery, many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in the laboratory creating operation flexibility for routine work. Larger projects are reviewed against capacity estimates before a bid is submitted in order to meet a client's schedule.

Management regularly monitors the status of turnaround time including those projects that have exceeded a current turnaround time. Proactive communication regarding potentially missed deadlines is expected from the laboratory management to the Project Managers to keep the client informed of report delivery status.

Any changes to the established timeline by the client or the laboratory must be communicated to the client or laboratory as soon as possible. Upon communication of changes, a new timeline is established and agreed upon by both parties.

13.3 Subcontracting

Occasionally, Eurofins Air Toxics subcontracts analyses to other laboratories if the requested testing is not routinely performed in our laboratory. Testing is only subcontracted with the client's knowledge and approval. Subcontract laboratories are selected based on their qualifications. If tests require a specific agency certification, only an appropriately accredited laboratory will be used. Additional details are available in SOP #90

eurofins | Air Toxics

LABORATORY QUALITY ASSURANCE MANUAL

(LQAM)

Appendix A

Terms and Definitions

(Seven total pages including this cover)

Current as of September 9, 2019

TERMS AND DEFINITIONS

Accuracy: The degree of agreement between an observed value and an accepted reference value.

Active sampling: The process of collecting a sample using pump or vacuum source to pull a known volume of vapor through a sorbent cartridge, or filter.

Ambient air: Outdoor air (also can include indoor air).

Analyte: The substance or component for which a sample is analyzed to determine its presence or quantity.

APH (air-phase hydrocarbons): Aliphatic and aromatic fractions identified in vapor-phase samples.

Approved: The determination by a state or federal accrediting agency that a certified laboratory may analyze for an analyte under the specified method.

Assessment: The process of inspecting, testing, and documenting findings for purposes of certification or to determine compliance.

ASTM International (formerly known as American Society for Testing and Materials)**:** Organization which develops international voluntary consensus-based standards.

Bag: An air-sampling container consisting of inert polymeric material.

Batch: A group of analytical samples (≤ 20) of the same matrix processed together, including extraction, concentration, and analysis using the same process, staff, and reagents.

BFB (4-Bromofluorobenzene): Compound used to verify that the mass spectrometer meets the tuning requirements of the method. Also can be used as an internal standard or surrogate.

Blank samples: Negative control samples used to assess potential contamination from sampling procedures or analytical processes. They can be field blanks or laboratory blanks.

BTEX: Benzene, toluene, ethylbenzene, and xylenes.

Canister: A stainless steel spherical air-sampling device consisting of Summa polished or glass-lined internal walls and a leak-tight on/off valve.

Certificate of Analysis (C of A): An authenticated document, issued by an appropriate authority, that assures a regulated product has met its product specification and quality.

Chain of Custody (COC): The chronological documentation of the custody of an environmental sample from the time it is taken until it is disposed.

Contamination: The effect caused by the introduction of a target analyte from an outside source into the test system.

Continuing Calibration Verification (CCV): A component of Quality Control used to verify instrument linearity with respect to the Initial Calibration (ICAL). A CCV is analyzed at the beginning of every analytical sequence and then periodically depending on the method. Certain methods also include a CCV in every analytical sequence as an End Check.

Control charts: Statistical tools for monitoring the performance of a particular task on a continuing basis. The control chart is prepared for each test parameter after 20 determinations have been performed. The mean is plotted with the warning limits being ±2s and the control limits being ±3s (s = Standard deviation).

Corrective action: An action taken to eliminate the cause(s) of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

Corrective Action Report: See NCCAR.

Data reduction: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results by a specific method and meet measurement quality objectives.

Detection Limit (DL): The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence.

%Difference (%D): A measure of precision between the expected value and the actual value, typically used to measure performance of the daily CCV RRF as compared to the Initial Calibration average RRF.

U.S. DoD: U.S. Department of Defense.

Duplicate sample: A sample collected for checking the preciseness of the sampling process. Duplicate samples are collected at the same time and from the same source as the study samples.

Equipment Blank: A sample that is known not to contain the target analyte, used to check the cleanliness of sampling devices. It is collected in a sampling container from a clean sample collection device and returned to the laboratory as a sample.

Field Blank: A sample that is known not to contain the target analyte, used to check for analytical artifacts or contamination introduced by sampling and analytical procedures. It is taken to the sampling site and exposed to sampling conditions, then returned to the laboratory and treated as an environmental sample.

Field Duplicate: A sample collected at the same time from the same source but submitted and analyzed as a separate sample.

GC (gas chromatograph): Analytical instrumentation used to resolve complex mixtures into individual peaks for identification and quantitation. Separation is achieved as chemicals are retained at varying rates by the column phase.

Holding time: The maximum time that a sample may be held prior to preparation or analysis.

Initial Calibration (ICAL): Demonstration of a linear response to different concentrations of calibration standards within a defined range.

Initial Calibration Verification (ICV): Verifies the Initial Calibration using a different source standard from the one used for Initial Calibration.

Initial Demonstration of Analytical Capability: The procedure described in USEPA 40 CFR 136 Appendix A, used to determine a laboratory's accuracy and precision in applying an analytical method.

Instrument Blank: A sample that is known not to contain the target analyte, processed through the instrumental steps of the measurement process and used to determine the absence of instrument contamination prior to analysis of field samples.

Instrument Detection Limit (IDL): The concentration of the analyte that produces a signal greater than five times the signal-to-noise ratio of the instrument.

Interference: The effect on the final result caused by the sample matrix.

Internal Standard (IS): A measured amount of a certain compound added after preparation or extraction of a sample.

Key Personnel: The laboratory director, technical director, quality assurance manager, and supervisor (group leader), all of whom meet the requirements of the NELAP rule.

Laboratory Control Sample (LCS): An independent second source reference standard that goes through the same pretreatment and preparation procedures as the samples. It validates the accuracy of the Initial Calibration.

Laboratory Duplicate: An aliquot of the same sample that is prepared and analyzed at the same time.

Laboratory Information Management System (LIMS): A laboratory's electronic data system that collects, analyzes, stores, and archives records and documents.

Limit of Detection (LOD): The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence.

Limit of Quantitation (LOQ): The smallest concentration that produces a quantitative result with known and recorded precision and bias.

MADEP APH: Massachusetts Department of Environmental Protection Air-Phase Petroleum Hydrocarbons.

Matrix: The component or substrate (e.g., surface water, drinking water, air, liquid waste) which contains the analyte(s) of interest.

Matrix Spike (MS): A sample prepared to determine the effect of the matrix on a method's recovery efficiency by adding a known amount of the target analyte to a specified amount of matrix sample for which an independent estimate of the target analyte concentration is available. It is used to evaluate accuracy.

Matrix Spike Duplicate (MSD): Duplicate of the matrix spike sample. Results are compared with MS to determine precision.

Mass spectrometer (MS): Analytical instrumentation used to identify and quantify chemicals utilizing spectral fragmentation patterns based on chemical structures.

Measurement uncertainty: Measurement uncertainty is the estimation of potential errors in a measurement process and is expressed as $\pm$ 2X(*s*) of the historical mean of LCS recoveries.

Method Detection Limit (MDL): The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero as determined from analysis of a sample containing the analyte in a given matrix (40 CFR Part 136, Appendix B, July 1995).

NCCAR (Non-conformance/Corrective Action Report): A report that identifies, communicates, tracks, and resolves a non-conformance.

NIST: National Institute of Standards and Technology.

NMOC: Non-methane organic compounds.

OSHA: Occupational Safety and Health Administration.

PAHs (polycyclic aromatic hydrocarbons): Hydrocarbons made up of fused aromatic ring molecules.

Passive sampling: Sample collection conducted without the use of mechanical pumps or vacuums. Collection relies on principle of diffusion.

ppbv: parts per billion by volume.

ppmv: parts per million by volume.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance or a range, in either absolute or relative terms.

Preservation: The temperature control or the addition of a substance to maintain the chemical or biological integrity of the target analyte.

Proficiency Testing (PT): A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.

Proficiency Test (PT) sample: A sample, the composition of which is unknown to the laboratory and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

Quality Assurance (QA): An integrated system of activities involving planning, quality control, reporting, and quality assessment and improvement to ensure that the product meets defined standards of quality with a stated level of confidence.

Quality Assurance Project Plan (QAPP): An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives for a specific data collection activity.

Quality Control (QC): A procedure or set of procedures intended to ensure that a product or performed service adheres to a defined set of quality criteria.

%R: %Recovery.

Relative Percent Difference (RPD): A measure of precision between two measurements calculated by dividing the absolute value of the difference between the measurements by their average and expressed as a percentage.

Reporting Limit (RL): The smallest concentration of an analyte that can be measured with a stated probability of significance. All Initial Calibrations contain a standard at the Reporting Limit. The Reporting Limit is never less than the Practical Quantitation Limit (PQL).

Reporting Limit verification: A re-quantification of the lowest concentration data point of an Initial Calibration to test the percent recovery of each component. Analyte recovery should be between 50–150% to verify detection limit accuracy.

Relative Standard Deviation (RSD): A measure of precision often used to evaluate linearity of an Initial Calibration. The relative response factor is calculated at each calibration level, and the RSD is calculated by dividing the standard deviation by the average value.

RRF: Relative Response Factor.

RT: Retention Time.

Safety Data Sheet (SDS): A technical document that contains information on the chemical make-up, use, storage, handling, emergency procedures, and potential health effects related to a hazardous material (formerly Material Safety Data Sheets).

Selectivity: The capability of a method or instrument to respond to the target analyte in the presence of other substances or things.

Semivolatile compound (SVOC): An organic compound which has a boiling point higher than water and which may vaporize when exposed to temperatures above room temperature.

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels of a target analyte.

Soil vapor (also referred to as "soil gas"): Vapor-phase volatile compounds that migrate or evaporate from contaminated soil.

Soil vapor extraction (SVE): A physical treatment process for in situ remediation of volatile contaminants in vadose zone (unsaturated) soils.

Standard Operating Procedure (SOP): A written document that details the steps of an operation, analysis, or action, the techniques and procedures for which are thoroughly prescribed and accepted as the procedure for performing certain routine or repetitive tasks.

Surrogate: A substance unlikely to be found in the environment that has properties which mimic the target analyte and that is added to a sample to check for analytical efficiency.

Target analyte: The analyte that a test is designed to detect or quantify.

Technical employee: A designated individual who performs the analytical method and associated techniques.

TIC: Tentatively Identified Compound.

TNMOC: Total non-methane organic compounds.

TOF: Time-of-Flight.

TPH: Total petroleum hydrocarbons.

Trip Blank: A sample known not to contain the target analyte, which is carried to the sampling site and transported to the laboratory for analysis without having been exposed to the sampling procedures.

TVH: Total volatile hydrocarbons.

Vapor intrusion (VI): The process by which vapors originating from contaminated soil or groundwater migrate through the subsurface into nearby buildings, potentially impacting indoor air quality.

VPH: Volatile Petroleum Hydrocarbons.

eurofins | Air Toxics

LABORATORY QUALITY ASSURANCE MANUAL

(LQAM)

Appendix B

Procedure Cross-Reference List

(Three total pages including this cover)

Current as of September 9, 2019

Procedure Cross-Reference List

Section	*Title*	*SOP*
2	***Organization and Personnel***	
2.7	Training	87
2.8	Employee Safety	30 17
2.9	Client Services/Project Management Responsibilities	1
2.10	Confidentiality	99
2.11	Operational Integrity	
3	***Buildings and Facilities***	
3.2	Security	30
4	***Document Control***	
4.1	Controlled Documents used at Eurofins Air Toxics	46 119
4.2	Document Approval, Issue, Control, and Maintenance	119
4.3	Laboratory Logbooks and Forms	119
4.4	Archival and Storage of Documents	119
5	***Sample Handling***	
5.2	Sample Receipt and Entry	50
5.3	Sample Identification and Tracking	50 96
5.4	Sample Storage	63
5.5	Sample Return/Disposal	63
5.6	Chain of Custody	63
6	***Technical Requirements - Traceability of Measurements***	
6.2	Calibration Standards	33
6.3	Equipment and Instrumentation	19 34 118
6.4	Computerized Systems and Computer Software	96 104
7	***Purchasing Equipment and Supplies***	
7.1	Procurement	105
7.2	Supplier Evaluation	105
8	***Analytical Methods***	
8.3	Method Validation	39 107
8.4	Procedural Deviations	61

Section	*Title*	*SOP*
9	***Internal Quality Control Checks***	
9.3	Quality Control Charts	48
9.4	Measurement Uncertainty	48
10	***Assuring Quality of Test Results***	
10.1	Data Management	96
10.2	Data Documentation	96
10.3	Data Calculations	
10.4	Reporting Limits	
10.5	Data Review	78
10.6	Data Qualification	
10.7	Data Reporting	68 78
10.8	Data Storage, Security, and Archival	55
11	***Audits and Inspections***	
11.1	Internal Quality Assurance Audits	27
11.2	Management Review System	106
11.3	Client Audits Agency Inspections	27
11.4	Proficiency Testing Program	
12	***Corrective and Preventive Action***	
12.1	Laboratory Investigations and Corrective Action	61
13	***Service to Clients***	
13.1	Review of Work Requests, Tenders, and Contracts	1
13.2	Timely Delivery	1
13.3	Subcontracting	90

eurofins | Air Toxics

LABORATORY QUALITY ASSURANCE MANUAL

(LQAM)

Appendix C

Certifications and Accreditations

(Two total pages including this cover)

Current as of September 9, 2019

Certifying Agency	Air Toxics Certificate #	Basis of Certification/Approval	Location of Certificate and Parameter List
Alaska DEC's CSLAP	18-006	Primary Certificate and Scope, Approval Program	Laboratory internal network: O:\QA\Certifications
Arizona DHS	AZ0775	Onsite assessment (biennial), LQAM and SOP	Laboratory internal network: O:\QA\Certifications
Florida DOH	E87680	Primary Certificate and Scope, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
Louisiana DEQ	02089	LQAM, SOPs, PT, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
New Hampshire DES	209218	LQAM, SOPs, PT, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
New Jersey DEP	CA016	LQAM, PT, SOPs, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
New York DOH	11291	LQAM, PT, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
Oregon DHS (Primary NELAP)	CA300005-012	Onsite assessment (biennial) LQAM, PT and SOP Review	Laboratory internal network: O:\QA\Certifications
Texas CEQ	T104704434-18-13	LQAM, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
Utah DOH	CA009332019-11	LQAM, PT, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
Virginia DCLS	10016	Secondary NELAP	Laboratory internal network: O:\QA\Certifications
Washington DOE	C935	PT, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
DoD-ELAP_ ISO/IEC 17025:2005	ADE-1451	DOD QSM for Environmental Laboratories v.5.1 Onsite assessment (biennial)	Laboratory internal network: O:\QA\Certifications

All latest certificates and licenses are posted by the laboratory entrance.

eurofins | Air Toxics

LABORATORY QUALITY ASSURANCE MANUAL

(LQAM)

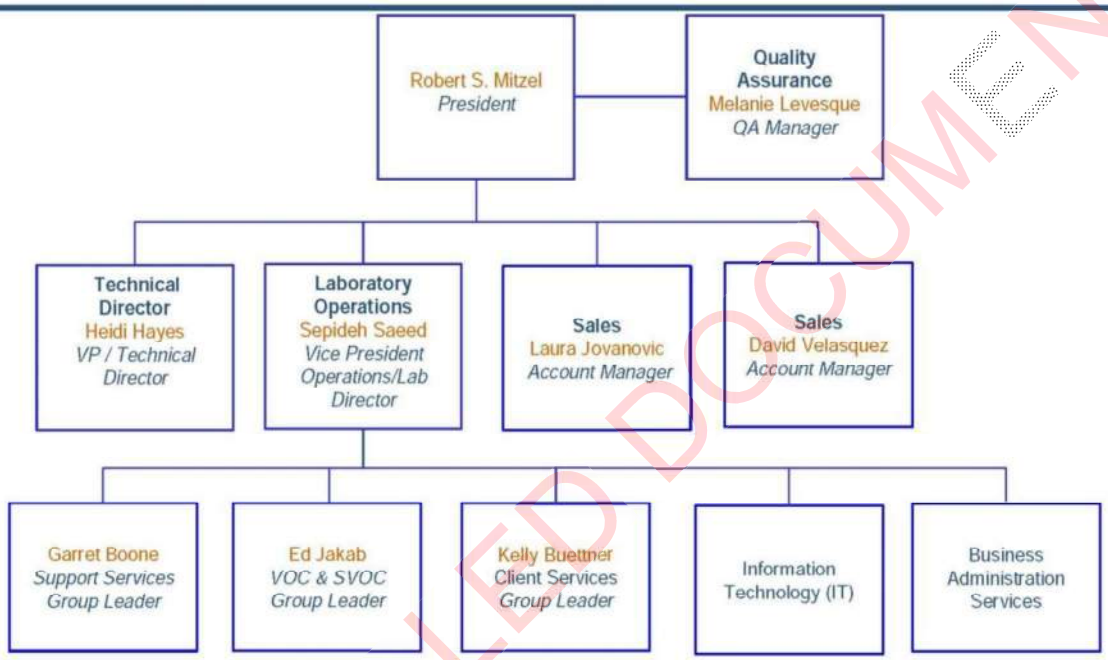
Appendix D

Organizational Charts

(Two total pages including this cover)

Current as of September 9, 2019

Organization Chart



eurofins | Air Toxics

LABORATORY QUALITY ASSURANCE MANUAL

(LQAM)

Appendix E

Analytical Methods

(Ninety total pages including this cover)

Current as of September 9, 2019

ANALYTICAL METHODS

SECTION	TITLE	PAGE
1.0	325B – VOCs	3
2.0	Aliphatic And Aromatic VPH Fractions and TRH Fractions	7
3.0	ASTM D1945 – Fixed Gases	12
4.0	ASTM D1946 – Atmospheric Gases	15
5.0	MADEP APH – VOCs and Air-Phase Petroleum Hydrocarbons	18
6.0	PM10/TSP – Particulate Matter	20
7.0	TO-3 and TO-14A – BTEX AND TPH	21
8.0	TO-12 – NMOC	24
9.0	TO-13A/TO-13A SIM – PAHs	26
10.0	TO-14A/TO-15 – VOCs (5&20 ppbv)	33
11.0	TO-14A/TO-15 – VOCs (Standard)	39
12.0	TO-14A/TO-15 – VOCs (Extended)	46
13.0	TO-14A/TO-15 – VOCs (Low Level)	55
14.0	TO-14A/TO-15 – VOCs (SIM)	62
15.0	TO-15 HSS – VOCs	67
16.0	TO-15 – Ethylene Oxide (SIM)	70
17.0	TO-17 – VOCs and SVOCs	72
18.0	TO-17 – Passive Sampling for VOCs (Charcoal-Based)	79
19.0	TO-17 – Passive Sampling for VOCs	86

ANALYTICAL METHODS

Section 1.0

Method: EPA 325B

Eurofins Air Toxics SOP #131 Revision 14 Effective Date: August 27, 2019 Methods Manual Summary

Description: This method involves measurement of VOCs in ambient air collected passively using standard 3.5 in L x 0.25 in OD inert-coated stainless steel sorbent tubes equipped with a diffusive screen cap and analyzed by thermal desorption- GC/MS. These procedures are specifically designed to comply with the EPA's refinery fenceline monitoring provisions in 40 CFR Section 63.658 which require the measurement of benzene over a 14-day period. This method also can be applied to additional VOCs outlined in Table 1 ranging from approximately 0.5 µg/m^3 to 5 mg/m^3 over sampling durations of up to 4 weeks.

VOCs in the sampling environment pass through a diffusive barrier at a controlled rate and adsorb to the sorbent bed of the passive sampler tube. The tubes are thermally desorbed by heating and purging with UHP Helium. The resulting gaseous effluent is transferred to secondary trap for re-concentration and desorption onto the GC/MS.

To calculate the concentration of the target compound, the mass measured on the sorbent tube is divided by the sampling rate and the sample collection time. The diffusive uptake rate is adjusted based on local temperature and pressure, and the final concentration is expressed at conditions of 25 degrees C and 1 atm (See ATM-122 for corrected EPA 325B concentration equation). The list of applicable compounds and associated sampling rates are summarized in Tables 1 and 3. The compound list can be extended beyond Table 1 if sampling rates are available for a given sorbent and duration in ISO 16017-2, ASTM D6196, or in a peer-reviewed journal article, and the analytical performance meets the quality requirements outlined in the following tables.

Table 1. Method 325B Reporting Limits and QC Limits (Carbopack X)

Analytes	RL (ng)	ICAL (%RSD)	ICV (%R)	CCV/LCS (%R)	Duplicate Standards (%RPD)
Benzene	5.0	≤30	70-130	70-130	≤20
Toluene	5.0	≤30	70-130	70-130	≤20
Ethyl Benzene	5.0	≤30	70-130	70-130	≤20
m,p-Xylene	5.0	≤30	70-130	70-130	≤20
o-Xylene	5.0	≤30	70-130	70-130	≤20
Styrene	5.0	≤30	70-130	70-130	≤20
1,3-Butadiene	2.5	≤30	70-130	70-130	≤20

Table 2. Internal Standards

Analyte	CCV IS %Recovery	Sample IS %Recovery
1,1-Dichlorofluoroethane	≥60	60-140
1,4-Difluorobenzene	≥60	60-140
Chlorobenzene-d5	≥60	60-140

Table 3. Uptake Rates (UR) and Sample Reporting limits (RLs) at 14 days (Carbopack X)

Compound	UR (ml/min)*	RL (ng)	14 day RL(ug/m3)	14 day RL(ppbv)
Benzene	0.67	5.0	0.37	0.12
Toluene	0.52	5.0	0.48	0.13
Ethylbenzene	0.46	5.0	0.54	0.12
m,p-Xylene	0.46	5.0	0.54	0.12
o-Xylene	0.46	5.0	0.54	0.12
Styrene	0.50	5.0	0.50	0.12
1,3-Butadiene	0.45**	2.5	0.28	0.12

*From Table 12.1 EPA Method 325
** 14-day UR from Martin et al. Atmospheric Environment 39 (2005) 1069-1077

Table 4. Method 325B Reporting Limits and QC Limits (Carbopack B)

Analytes	RL (ng)	ICAL (%RSD)	ICV (%R)	CCV/LCS (%R)	Duplicate Standards (%RPD)
Benzene*	5.0	≤30	70-130	70-130	≤20
Toluene*	5.0	≤30	70-130	70-130	≤20
Ethyl Benzene*	5.0	≤30	70-130	70-130	≤20
m,p-Xylene*	5.0	≤30	70-130	70-130	≤20
o-Xylene*	5.0	≤30	70-130	70-130	≤20
Naphthalene	1.0	≤30	70-130	70-130	≤20

*From Table 12.1 EPA Method 325

Table 5. Uptake Rates (UR) and Sample Reporting limits (RLs) at 14 days (Carbopack B)

Compound	UR (ml/min)	RL (ng)	14 day RL(ug/m3)	14 day RL(ppbv)
Benzene	0.63	5.0	0.394	0.123
Toluene	0.56	5.0	0.443	0.118
Ethyl Benzene	0.50	5.0	0.496	0.114

m,p-Xylene	0.47	5.0	0.528	0.122
o-Xylene	0.47	5.0	0.528	0.122
Naphthalene*	0.45	1.0	0.11	0.021

*Uptake rate from McAlary et. al., Environmental Science: Processes & Impacts, 17, 2015, 896-905

Table 6. Summary of Calibration and QC Procedures for EPA Method 325B

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
BFB Tune Check	Every 24 hours	EPA 325B Tune Criteria	Correct problem and repeat tune check. Clean source and/or retune MS if needed.
5-point Calibration	Prior to sample analysis and following any significant instrument maintenance or change. ICAL expires after 3 months.	%RSD≤30	Correct problem and repeat initial calibration curve.
Desorption Efficiency Check	After each initial calibration curve	>95% Desorption Efficiency	Evaluate Thermal desorption parameters, and adjust as needed. Recalibrate after parameter changes.
Initial Calibration Verification (ICV)	After each initial calibration curve.	70-130% recovery	Verify accuracy of standard. Re-prepare ICV or primary calibration standard if necessary. If calibration curve and/or system is identified as the problem, re-calibrate.
Initial Continuing Calibration Verification (CCV)	After the tune check at the start of each sequence. ***The RRF of the initial daily CCV is used for sample quantitation.***	70-130%.	If the beginning CCV does not meet criterion, re-prepare CCV and re-analyze. If still fails, then re-calibrate.
Ongoing Calibration Checks using Laboratory Control Sample (LCS)	Every 10 field samples after the initial daily CCV and at the end of the batch.	70-130%	If the mid-check or end check fails, re-analyze samples analyzed after the last passing check unless the recovery was high and no detections were measured. If re-analysis is not possible, then flag and narrate affected samples.
CCV-Recollection(CCV-R)	After each initial calibration curve and daily after the CCV to insure re-collection feature is working.	See criteria in SOP section 8.5.6. If these ranges are met, the 20% RPD criterion will be met.	Evaluate analytical unit and re-collection feature. Re-prepare CCV and CCV-R to verify after evaluation.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Blank	After the beginning CCV.	Beginning lab blank < RL	Re-analyze the lab blank. If still above criterion, flag data accordingly.
Field Blanks	If 2 are submitted with a set of samples, run one at the beginning of the field samples and one at the end.	Less than 1/3 the measured sample target analyte or compliance limit.	Flag and narrate all sample results noting that the associated results are estimated with a high bias due to field blank background.
Field Duplicates	Per method, a frequency of 10%, run with field samples.	≤30%RPD	Apply relevant 'P' flag to data set and narrate discrepancy.
Internal Standard (IS)	Added to each sample and QC sample at the time of desorption.	Initial CCV: ≥60% mid-point ICAL area counts; within 20s of mid-point in ICAL. Blanks, samples and mid- and end checks: IS areas must be ± 40% of the initial CCV IS areas. RT within ±0.33 min as compared to the daily CCV.	CCV: Inspect and correct system prior to sample analysis. Blanks: inspect the system and re-analyze the blank. Samples: Re-analyze the samples. If the IS is still out, flag the associated data and narrate. If the IS recovery is recovering high in samples and on-going CCVs, evaluate recovery of target analytes in affected CCVs to determine if accuracy is impacted or if IS response increase reflects an overall increase in sensitivity.

ANALYTICAL METHODS

Section 2.0

Method: Aliphatic and Aromatic Volatile Petroleum Hydrocarbons (VPH) Fractions and Total Recoverable Hydrocarbon (TRH) Fractions by GC/MS

Eurofins Air Toxics SOP #103 Revision 9.1 Effective Date: Sept 12, 2018 Methods Manual Summary

Description: The WSDE VPH method outlines procedures to estimate the concentrations of gaseous phase Aliphatic and Aromatic ranges in ambient air and soil gas collected in stainless steel Summa canisters. The volatile Aliphatic hydrocarbons are collectively quantified within the C5 to C6 range, C6 to C8 range, C8 to C10 range, and the C10 to C12 range. Additionally, the volatile Aromatic hydrocarbons are collectively quantified within the C8 to C10 range and the C10 to C12 range. The Aromatic ranges refer to the equivalent carbon (EC) ranges.

Data is acquired using standard TO-15 GC/MS instrumentation. Procedures are largely based on the hydrocarbon ranges and calibration reference compounds defined by the Washington State Department of Ecology (WSDE) Method for the Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions, dated June 1997. Additionally, the WSDE VPH calibration and quantitation procedures for the Aromatic fraction have been enhanced to more effectively isolate the compounds of interest. The Aromatic fraction measurement is based on a modification of the Massachusetts Department of Environmental Protection (MADEP) Air Phase Hydrocarbon Method (2009).

In addition to the VPH fractions detailed above, the procedures to calculate Total Recoverable Hydrocarbon (TRH) fractions, F1 and modified F2 fractions are commonly requested as part of the Australian NEPM suite. Two TRH fractions are requested >C6-C10 and >C10-C12. The TRH value includes all sample related peaks including chlorinated compounds, BTEX, aromatics and siloxanes.

Eurofins Air Toxics performs a modified version of the WSDE VPH method. The method modifications, standard target analyte list, reporting limit (RL) or Limit of Quantitation (LOQ), QC criteria, and QC summary can be found in the following tables.

Table 1. Summary of Method Modifications for WSDE VPH

Requirement	VPH	Eurofins Air Toxics Modifications
Detector	Tandem GC/FID/PID	GC/MS
Matrix	Soil, water, and sediments	Whole air samples
C6-C8 Reference Compound	Octane	Heptane
Surrogate	2,5-Dibromotoluene	Bromochloromethane, 1,2-Dichloroethane-d4, Toluene-d8, Chlorobenzene-d5, and 4-Bromofluorobenzene
%RSD for Reference Compounds	≤ 20% RSD	≤ 30% RSD with the exception of Decane, Dodecane, 1,2,4,5-Tetramethylbenzene, and Naphthalene at ≤ 40% RSD

Requirement	VPH	Eurofins Air Toxics Modifications
%D for the CCV	±20%D	±30%D with one allowed out not to exceed ±40%. Decane, Dodecane, 1,2,4,5-Tetramethylbenzene, and Naphthalene at ±40%D with one allowed out not to exceed ±50%.
Laboratory Control Spike	Matrix Spiking Solution	Independently prepared source performed after initial calibration, 70–130% recovery, with the exception of Decane, Dodecane,1,2,4,5-Tetramethylbenzene, and Naphthalene at 60–140%
CCV Frequency	Before and after every 10 samples	Daily before sample analysis
IDOC	4 Replicates of a CCV at ±20%D; %RSD ≤ 20%	Not performed for this method; TO-15 IDOC performed on the same instrument

Table 2. VPH Standard Target Analyte List (Note: TO-15 analytes can also be included.)

Analyte	Standard RL (ppbv)	5&20 RL (ppbv)	Acceptance Criteria		
			ICAL %RSD	ICV (%R)	CCV (%D)
Pentane	NA	NA	≤ 30%	70-130	≤ 30%
Hexane	NA	NA	≤ 30%	70-130	≤ 30%
C_5-C_6 Aliphatics Pentane + Hexane	**10**	**50**	**≤ 30%**	**70-130**	**≤ 30%**
C_6-C_8 Aliphatics ref. to Heptane	**10**	**50**	**≤ 30%**	**70-130**	**≤ 30%**
C_8-C_{10} Aliphatics ref. to Decane	**10**	**50**	**≤ 40%**	**60-140**	**≤ 40%**
C_{10}-C_{12} Aliphatics ref. to Dodecane	**10**	**50**	**≤ 40%**	**60-140**	**≤ 40%**
Ethyl benzene	2	10	≤ 30%	70-130	≤ 30%
m/p-Xylene	2	10	≤ 30%	70-130	≤ 30%
o-Xylene	2	10	≤ 30%	70-130	≤ 30%
1,2,3-Trimethylbenzene	NA	NA	≤ 30%	70-130	≤ 30%
C_8-C_{10} Aromatics	**10**	**50**	**≤ 30%**	**70-130**	**≤ 30%**
Naphthalene	2	10	≤ 40%	60-140	≤ 40%
1,2,4,5-Tetramethylbenzene	NA	NA	≤ 40%	60-140	≤ 40%
C_{10}-C_{12} Aromatics	**10**	**50**	**≤ 40%**	**60-140**	**≤ 40%**

Table 3. Internal Standard Acceptance Criterion – Aliphatic Fraction and TRH

Analyte	Recovery Limits (%R)
1,4-Difluorobenzene	50 – 200%

Table 4. Internal Standard Acceptance Criterion – Aromatic Fraction

Analyte	Recovery Limits (%R)
Chlorobenzene-d_5	60 – 140%

Table 5. Summary of WSDE VPH Calibration and QC Procedures

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours	Compendium of Methods for Toxic Organic Air Pollutants, Method TO-15, January 1999	Correct problem then repeat tune.
Initial Calibration (ICAL)	Prior to sample analysis	Minimum of 5 levels. %RSD ≤ 30% for VPH Target Analyte List with exceptions for 1,2,4,5-Tetramethylbenzene and Naphthalene, which are ≤40%	Correct problem then repeat initial calibration curve.
Initial Calibration Verification (ICV)	After each initial calibration curve	Recoveries for VPH target compounds 70–130%, or 60–140% for 1,2,4,5-Tetramethylbenzene and Naphthalene. If recovery of any compound is above 130%, analyze samples as long as compound is not detected.	Check the system and re-analyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each analytical clock after the tune check	%D ≤ 30% for VPH target compounds with the exceptions for 1,2,4,5-Tetramethylbenzene and Naphthalene which are ≤40%. One compound is allowed to be out as long as it is ≤ 50%D. If recovery of any compound is above 150% the instrument must be re-calibrated.	Perform maintenance and repeat test. If the CCV still fails, perform maintenance and a new calibration curve.
Laboratory Blank	After the CCV	Results less than the laboratory RL	Inspect the system and re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for the blanks and samples must be within ±0.33 min of the RT in the CCV. For the aliphatic fraction using the total ion area, the IS area must be within -50% to 200% of the CCV's IS area for the blanks and samples. For the aromatic fraction using extracted ion areas, the IS area must be within -40% to +40% of the CCV's extracted ion IS area.	**For blanks:** Inspect the system and re-analyze the blank **For samples:** If there is not obvious interference with the internal standard, re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. Dilution of the sample to get IS areas within limits may be used if the RL is being obtained.
Laboratory Duplicates	One per analytical batch; since VPH analysis occurs with TO-15 analysis, the Duplicate is reported from the daily TO-15 LCS/LCSD pair. The	RPD ≤ 25% for detections >5X's the RL	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
	result is not reported with the VPH fraction.		

When referencing EPA TO-15 as the analytical method, the laboratory has implemented the following modifications to the TO-15 requirements:

Table 6. Summary of Method Modifications for TRH

Requirement	TO-15	EATL Modifications
Target Quantitation	Quantify target concentrations using extracted ion areas	Quantify TRH using total ion peak area eluting within defined retention time range and calibrated with the total ion response of the assigned reference compound
Internal standard area recovery	-40 to +40% of the CCV extracted ion response	Because total ion area is used for internal standard and sample peaks, sample recovery criterion is -50 to +200% of the CCV total ion response.
Daily CCV recovery	≤30%D	≤40%D for Decane

Table 7. TRH Compound Reporting and QC Limits

Analyte	RL (ppbv)	ICAL (%RSD)	CCV(%R)	ICV (%R)
>C6-C10 TRH	10	≤30	70-130	70-130
F1	10	NA	NA	NA
>C10-C12 TRH	10	≤40	60-140	60-140
mod F2	10	NA	NA	NA
Toluene (Reference compound)	NA	≤30	70-130	70-130
n-Decane (Reference compound)	NA	≤40	60-140	60-140

NA = Not Applicable

Table 8. Summary of TRH Calibration and QC Procedures

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tune Check	At the start of a 24 hour analytical clock	Method TO-15 (Appendix A)	Correct problem and repeat tune.
Initial Calibration	Prior to sample analysis	Minimum of 5 levels; %RSD≤30% Toluene %RSD≤40% Decane	Correct problem then repeat initial calibration curve.
Initial Calibration Verification (ICV)	After each initial calibration curve	70-130% Toluene, 60-140% Decane	Check the system and re-analyze the standard. Re-prepare the standard

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
			if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	After the tune check and prior to sample analysis	%D≤30% Toluene %D≤40% Decane	Repeat CCV, if still fails, perform maintenance as needed and analyze new ICAL.
Lab Blank	After the CCV and prior to samples.	Results for >C6-C10 TRH and >C10-C12 TRH less than the RL (10 ppbv)	Inspect system and re-analyze blank.
Internal Standard (IS)	As each standard, blank, and sample is loaded on the unit.	Retention time (RT) for the blanks and samples must be within ±0.33 min of the RT in the CCV. Since the total ion is used for quantitation, the total ion area, the IS area must be within -50 to 200% of the CCV's IS area for the blanks and samples	For blanks: inspect the system and re-analyze the blank; For samples: If there is not obvious interference with the internal standard, re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. Dilution of the sample to get IS areas within limits may be used if the RL is being obtained

ANALYTICAL METHODS

Section 3.0

Method: ASTM D1945 – Fixed Gases & C1-C6

Eurofins Air Toxics SOP #54 Revision 24 Effective Date: November 20, 2018 Methods Manual Summary

Description: This method involves gas chromatograph (GC) analysis of soil gas, landfill gas, ambient air, or stack gas collected in Summa™ canisters, Tedlar bags, or any vessel that has been demonstrated to be clean and leak free. Samples are analyzed for Methane and fixed gases and can be used to speciate individual light hydrocarbons up to C6. This method is also used to provide an estimation of the heating value of the gas by method ASTM D3588. Because the sample is withdrawn from the vessel by positive pressure, rigid containers are first filled to positive pressure using UHP Helium or Nitrogen. Samples are then analyzed using a GC equipped with a Flame Ionization Detector (FID) and a Thermal Conductivity Detector (TCD).

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, the laboratory reports these non-standard compounds with partial validation. Validation includes a 5-point calibration with the lowest concentration defining the reporting limit (RL), no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compounds during sample storage is not validated. Full validation may be available upon request.

Since the protocols in the ASTM D1945 standard were designed for the analysis of natural gas, the laboratory has made modifications in order to apply the method to environmental samples covering a wide concentration range and to implement standard NELAP and EPA calibration criteria. The method modifications, standard target analyte list, RL, Quality Control (QC) criteria, and QC summary can be found in the following tables.

Table 1. Summary of Method Modifications for ASTM D1945

Requirement	ASTM D1945	Eurofins Air Toxics Modifications
Sample Injection Volume	0.50 mL to achieve Methane linearity.	1.0 mL
Calibration	A single point calibration is performed using a reference standard closely matching the composition of the unknown. Standard is analyzed such that 2 consecutive runs meet method repeatability requirements.	A minimum 5-point calibration curve is performed. Quantitation is based on the initial calibration response factor. A single run of a mid-level calibration standard is used to verify the initial calibration and may or may not resemble the composition of the associated samples.
Sample Analysis	Equilibrate samples to 20-50° F above source temperature at field sampling.	No heating of samples is performed.
Sample Calculation	Response factor is calculated using peak height for C5 and lighter compounds.	Peak areas are used for all target analytes to quantitate concentrations.
Normalization	Sum of original values should not differ from 100.0% by more than 1.0%.	Sum of original values may range between 85–115%; normalization of data not performed unless client requested.

Table 2. ASTM Method D1945 Compound List and QC Limits

Analyte	Reporting Limit (%)	QC Acceptance Criteria		
		ICAL (%RSD)	CCV/LCS/ICV (%R)	Precision* (%RPD)
Carbon Dioxide	0.01	≤ 15%	± 15%	≤ 25%
Carbon Monoxide	0.01	≤ 15%	± 15%	≤ 25%
Ethene	0.001	≤ 15%	± 15%	≤ 25%
Ethane	0.001	≤ 15%	± 15%	≤ 25%
Acetylene	0.001	≤ 15%	± 15%	≤ 25%
Isobutane	0.001	≤ 15%	± 15%	≤ 25%
Isopentane	0.001	≤ 15%	± 15%	≤ 25%
Methane	0.0001	≤ 15%	± 15%	≤ 25%
n-Butane	0.001	≤ 15%	± 15%	≤ 25%
Neopentane	0.001	≤ 15%	± 15%	≤ 25%
n-Pentane	0.001	≤ 15%	± 15%	≤ 25%
Nitrogen**	0.10	≤ 15%	± 15%	≤ 25%
NMOC (C6+)	0.01	≤ 15%	± 15%	≤ 25%
Oxygen	0.10	≤ 15%	± 15%	≤ 25%
Propane	0.001	≤ 15%	± 15%	≤ 25%
Hydrogen***	0.01	≤ 15%	± 15%	≤ 25%
Helium	0.05	≤ 15%	± 15%	≤ 25%

* For detections at > 5X the Reporting Limit.
**For canisters that have been pressurized with Nitrogen, the amount of Nitrogen in the sample is determined by subtraction.
***For canisters that have been pressurized with Helium, the Reporting Limit is 1.0%.

Note: Results are reported in units of mol %. If required to report volume % or ppmV, a compressibility factor of 1 for all gases will be assumed. As a result, mol % is assumed to be equivalent to volume %. This assumption may result in a bias for highly compressible gases at high concentrations and pressures.

Table 3. Summary of Calibration and QC Procedures for Mod. ASTM Method D1945

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	Prior to sample analysis and annually	≤ 15% RSD	Correct problem, then repeat Initial Calibration.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each Initial Calibration and once per analytical batch.	85–115% Recovery If specified by the project, in-house generated control limits may be used.	Check the system and re-analyze the standard. Re-prepare the standard if necessary. If the primary standard is found to be in error, re-prepare the primary and calibrate the instrument.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis, and can be used as an End Check.	± 15% Difference	Check the system and re-analyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met. If the closing CCV fails, the system is checked and the standard is re-analyzed. Re-prepare the standard if necessary. If the second analysis fails, identify and correct the problem, then re-analyze all samples since the last acceptable CCV.
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory Reporting Limit	Inspect the system and re-analyze the Laboratory Blank.
Laboratory Duplicates- Laboratory Control Spike Duplicate (LCSD)	One per analytical batch	RPD ≤ 25%	Narrate exceedances. Investigate the cause and perform maintenance as required and re-calibrate as needed.

ANALYTICAL METHODS

Section 4.0

Method: ASTM D1946 – Atmospheric Gases

Eurofins Air Toxics SOP #8 Revision 27 Effective Date: June 11, 2019 Methods Manual Summary

Description: This method involves gas chromatograph (GC) analysis of soil gas, landfill gas, ambient air, or stack gas collected in Summa™ canisters, Tedlar bags, or any vessel that has been demonstrated to be clean and leak free. Samples are analyzed for Methane, fixed gases, and Non-Methane Organic Carbon (NMOC) using modified ASTM D1946 protocols. Because the sample is withdrawn from the vessel by positive pressure, rigid containers are first filled to positive pressure using UHP Helium or Nitrogen. Samples are then analyzed using a GC equipped with a FID and a TCD.

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, the laboratory reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Since the protocols in the ASTM D1946 standard were designed for the analysis of reformed gas, the laboratory has taken modifications to apply the method to environmental samples covering a wide concentration range and to implement standard NELAP and EPA calibration criteria. The method modifications, standard target analyte list, reporting limits (RL), Quality Control (QC) criteria, and QC summary can be found in the following tables.

Table 1. Summary of Method Modifications for ASTM D1946

Requirement	ASTM D1946	Eurofins Air Toxics Modifications
Calibration	A single-point calibration is performed using a reference standard closely matching the composition of the unknown.	A minimum 5-point calibration curve is performed. Quantitation is based on the initial calibration, which may or may not resemble the composition of the associated samples.
Reference Standard	The composition of any reference standard must be known to within 0.01 mol % for any component.	The standards used by Eurofins Air Toxics are blended to a ≥ 95% accuracy.
Sample Injection Volume	Components whose concentrations are in excess of 5% should not be analyzed by using sample volumes greater than 0.5 mL.	The sample container is connected directly to a fixed volume sample loop of 1.0 mL. Linear range is defined by the calibration curve. Bags may be loaded by vacuum or by positive pressure.
Normalization	Normalize the mole percent values by multiplying each value by 100 and dividing by the sum of the original values. The sum of the original values should not differ from 100% by more than 1.0%.	Results are not normalized. The sum of the reported values can differ from 100% by as much as 15%, either due to analytical variability or an unusual sample matrix.

Requirement	ASTM D1946	Eurofins Air Toxics Modifications
Precision	Precision requirements established at each concentration level.	Duplicates should agree within 25% RPD for detections >5X the RL.

Table 2. ASTM D1946 Method Compound List and QC Limits

Compound	Reporting Limit (%)	ICAL Criteria (%RSD)	ICV/LCS Criteria (%R)	CCV Criteria (%D)	Precision Limits (RPD)**
Carbon Dioxide	0.010	≤ 15%	85 – 115	± 15%	± 25%
Carbon Monoxide	0.010	≤ 15%	85 – 115	± 15%	± 25%
Methane	0.00010	≤ 15%	85 – 115	± 15%	± 25%
Ethene	0.0010	≤ 15%	85 – 115	± 15%	± 25%
Ethane	0.0010	≤ 15%	85 – 115	± 15%	± 25%
Nitrogen	0.10	≤ 15%	85 – 115	± 15%	± 25%
NMOC	0.010	≤ 15%	85 – 115	± 15%	± 25%
Oxygen	0.10	≤ 15%	85 – 115	± 15%	± 25%
Helium	0.050	≤ 15%	85 – 115	± 15%	± 25%
Hydrogen	0.010*	≤ 15%	85 – 115	± 15%	± 25%

*Reporting limit is 1.0% when sample is pressurized with Helium.
**For detections greater than 5 times the reporting limit.

Note: Results are reported in units of mol %. If required to report volume % or ppmV, a compressibility factor of 1 for all gases will be assumed. As a result, mol % is assumed to be equivalent to volume %. This assumption may result in a bias for highly compressible gases at high concentrations and pressures.

Table 3. Summary of Calibration and QC Procedures for Mod. ASTM Method D1946

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Curve (ICAL)	Prior to sample analysis	RSD ≤ 15%	Correct problem then repeat Initial Calibration.
Second Source Verification (LCS)	All analytes: once per Initial Calibration, and with each analytical batch	%R between 85–115%	Check the system and re-analyze the standard. Verify the accuracy of standards as needed. Re-prepare erroneous standards and/or re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis and after every 20 reportable samples.	%D ±15%	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met.
Laboratory Blank (He) (N_2 for He and H_2 analysis)	After each daily check standard and prior to sample analysis, or when contamination is present.	Results below the RL	Inspect the system and re-analyze the Blank.
End Check	At the end of analytical sequence. It can be primary (CCV) or Independent Source (LCS).	%R between 85–115%	Check system and re-analyze the standard. If the 2nd analysis fails, identify and correct the problem. Samples analyzed after the last acceptable CCV are re-analyzed.
Sample Duplicates - Laboratory Control Spike Duplicate (LCSD)	One per analytical batch	RPD ≤ 25%	Narrate exceedances. Investigate the cause and perform maintenance as required and re-calibrate as needed.

ANALYTICAL METHODS

Section 5.0

Method: Massachusetts DEP Air-Phase Petroleum Hydrocarbons (APH)

Eurofins Air Toxics SOP #42 Revision 9 Effective Date: September 21, 2018 Methods Manual Summary

Description: This method involves full scan gas chromatograph/mass spectrometer (GC/MS) analysis of whole air samples collected in evacuated stainless steel Summa® canisters. Samples are analyzed for air-phase hydrocarbons (APH) using the Massachusetts Department of Environmental Protection method protocols. An aliquot of up to 0.5 liters of air is withdrawn from the canister utilizing a mass flow controller. This volume is loaded onto a hydrophobic multisorbent trap to remove water and to concentrate the vapor sample. The focused sample is then flash-heated and back flushed off the system which removes water from the sample stream prior to analysis by GC/MS. Eurofins Air Toxics performs this analysis without taking modifications to the MADEP APH method. The standard target analyte list, reporting limit (RL) also referred to as Limit of Quantitation, QC criteria, and QC summary can be found in Tables 2 through 4.

Table 1. APH Target Compound List

Analyte	Reporting Limit (ug/m3)	Acceptance Criteria	
		Accuracy Limits (%R)	Precision Limits (RPD)
1,3-Butadiene	2.0	70 - 130	± 25
Methyl-tert-butyl ether (MTBE)	2.0	70 - 130	± 25
Benzene	2.0	70 - 130	± 25
Toluene	2.0	70 - 130	± 25
Ethyl benzene	2.0	70 - 130	± 25
m/p-Xylene	2.0	70 - 130	± 25
o-Xylene	2.0	70 - 130	± 25
Naphthalene	2.0	60 - 140	± 25

Table 2. Aliphatics & Aromatics Hydrocarbon Ranges

Analyte	Reporting Limit ($\mu g/m^3$)	Acceptance Criteria	
		Accuracy Limits (%R)	Precision Limits (RPD)
C_5-C_8 Aliphatics	12	70 – 130	±25
C_9-C_{12} Aliphatics	12	70 - 130	± 25
C_9-C_{10} Aromatics	10	70 - 130	± 25

Table 3. Internal Standards

Analyte	Accuracy Limits (%)
Bromochloromethane	50 to 200
1,4-Difluorobenzene	50 to 200
Chlorobenzene-d_5	50 to 200

Table 4. Summary of Calibration and QC Procedures for Method MADEP APH

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours	TO-15 ion abundance criteria	Correct problem then repeat tune.
Minimum 5-Point Initial Calibration (ICAL)	Prior to sample analysis	%RSD ≤30% for APH Target Analyte or hydrocarbon range Naphthalene is ≤40%.	Correct problem then repeat Initial Calibration curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) (Subset of Target Compounds)	After each Initial Calibration curve, and daily prior to sample analysis	Recoveries for the APH target compounds and hydrocarbon ranges must be ±30%. If recovery of any compound is above 130% (not to exceed 150%), analyze samples as long as compound is not detected. Naphthalene recovery must be 50-150%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each analytical clock after the tune check	%D ≤ 30% for APH target compounds and hydrocarbon ranges. One compound is allowed to be out as long as it is ≤ 50%D. Compound list naphthalene allowed %D ≤ 40%. If recovery of any compound is above 150%, instrument must be re-calibrated.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit Naphthalene and C12 are allowed to be 2X the RL.	Inspect the system and re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded	Retention time (RT) for the blanks and samples must be within ±0.33 min of the RT in the CCV. The IS area must be within -50 to 200% of the CCV's IS area for the blanks and samples.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. Dilution of the sample to get IS areas within limits may be used if the RL is being obtained.
Laboratory Duplicates	Must be 10% of project samples. Duplicate one compound per batch	RPD ≤ 30% for detections >5 X's the RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate.

ANALYTICAL METHODS

Section 6.0

Method: PM10/TSP – Particulate Matter

Eurofins Air Toxics SOP #66 Revision 18 Effective Date: November 30, 2018 Methods Manual Summary

Description: This method involves equilibrating quartz filters in a conditioning environment of a specified temperature and humidity range and weighing the filters before and after field sampling. Samples are analyzed for method PM_{10} using 40 CFR Part 50 Appendix J or for Total Suspended Particulate (TSP) using 40 CFR Part 50 Appendix B. An analytical balance with 0.1 mg resolution is used to measure the filter weights. The corresponding change in mass represents the TSP or PM_{10} result, expressed in µg or µg/m^3. The reporting limit is typically 1000 µg. Sampling volumes are required to calculate results in units of µg/m^3.

Table 1. Conditioning Environment Criteria for Methods PM10 and TSP

Method	Conditioning Environment Temperature (˚F)	Conditioning Environment Relative Humidity (%)
PM10	59°F – 86°F ± 5°F	20% – 45% ± 5%
TSP	59°F – 86°F ± 5°F	≤ 50% ± 5%

Table 2. Summary of Calibration and QC Procedures for Methods PM10 and TSP

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Calibration	Calibration checks of 3.00 grams (g) and 5.00 g are weighed to bracket the expected filter weight of ~4.5 g prior to sample analysis and at the end of the analytical batch.	Accuracy limits of 3.00 g weight: 2.997 g – 3.003 g Accuracy limits of 5.00 g weight: 4.995 g - 5.005 g	Correct problem then repeat calibration.
Laboratory Duplicates	Unexposed filters: One per analytical batch Exposed filters: One duplicate per work order	Unexposed filters: Weights of the clean filters should be within ±0.0028 g of the original value. Exposed filters: ≤ 25% RPD and weights must be within ±0.005 g	Re-condition the filter and re-weigh.
Laboratory Blanks	Immediately after the calibration checks	Post-weight of Lab Blank is less than pre-weight and the difference is < 0.0028 g.	Confirm the weight difference and narrate.

ANALYTICAL METHODS

Section 7.0

Method: EPA Methods TO-3 and TO-14A (TPH)

Eurofins Air Toxics SOP #43 Revision 26 Effective Date: September 9, 2019 Methods Manual Summary

Description: This method involves GC analysis of whole air samples collected in Summa canisters or Tedlar bags. Samples are analyzed for Total Petroleum Hydrocarbons (TPH). Either modified EPA Method TO-3 or Method TO-14A or can be used to reference laboratory protocols. TPH is measured using a Flame Ionization Detector (FID). Depending on the client's request, TPH is analyzed and referenced to either gasoline or jet fuel.

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, the laboratory reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Eurofins Air Toxics performs a modified version for these methods. The method modifications, standard target analyte list, reporting limit (RL), QC criteria, and QC summary can be found in the following tables.

Table 1. Summary of Method Modifications for TO-14A

Requirement	EPA Method TO-14A	Eurofins Air Toxics Modifications
Sample Drying System*	Nafion Dryer	Multi-bed sorbent
Sample collection containers	Specially treated stainless steel canisters	Method TO-14A is validated for samples collected in specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of the method and not recommended for ambient or indoor air samples. Associated results are considered qualified.

*The pre-concentrator modification implemented for sample analysis allows for superior performance over the water management and concentration procedures outlined in Method TO-14A. This multi-bed sorbent approach used in EPA Method TO-15 allows for the inclusion of polar compounds such as MTBE, and demonstrates superior performance by minimizing carryover issues that can be problematic using the Nafion dryer scenario described in Method TO-14A.

Table 2. Summary of Method Modifications for TO-3

Requirement	EPA Method TO-3	Eurofins Air Toxics Modifications
Sample Collection	In-line field method	Collection of sample in specially treated canisters or alternative containers for transport to and analysis by an off-site laboratory.
Preparation of Standards	Levels achieved through dilution of gas mixture	Levels achieved through loading various volumes of the gas mixture.
Initial Calibration Calculation	4-point calibration using a linear regression model	5-point calibration using average Response Factor
Initial Calibration Frequency	Weekly	When daily calibration standard recovery is outside 75–125%, or upon significant changes to the procedure or instrumentation.
Daily Calibration Standard Frequency	Prior to sample analysis and every 4-6 hrs	Prior to sample analysis and at the end of sample analysis. End checks can be demonstrated by use of CCV and/or LCS standards.
Minimum Detection Limit (MDL)	Calculated using the equation DL = A+3.3S, where A is intercept of calibration line and S is the standard deviation of at least 3 reps of low level standard.	40 CFR Part 136, App. B
Sample pre-concentration and moisture management	Cyrogenic pre-concentrator with a Nafion dryer	Multi-bed sorbent system

Table 3. Method Compound List and QC Limits

Analyte	RL (ppmv)	Acceptance Criteria		
		ICAL (%RSD)	LCS/CCV (%R)	Precision* (%RPD)
TPH (Gasoline Range) MW = 100	0.025	≤ 30	± 25	≤ 25
TPH (JP-4 Range) MW = 156	0.025	≤ 30	± 25	≤ 25

*For detections > 5 X RL

Table 4. Surrogate QC Limits

Surrogate	FID Accuracy (%R)
Fluorobenzene	75–150%

Table 5. Summary of Calibration and QC Procedures for TO-3/TO-14A (TPH)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
5-Point Initial Calibration (ICAL)	Prior to sample analysis and annually	%RSD ≤ 30	Correct problem, then repeat the calibration.
Initial Calibration Verification and Laboratory Control Sample (ICV/LCS)	With each initial calibration, and with each analytical batch.	±25% of the expected value	Check the system and re-analyze the standard. Re-prepare the standard or re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis and can be used as an End Check	±25% of the expected value	**For initial CCV:** Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met. **For Mid- and End Checks:** Check system and re-analyze the standard. If the second analysis fails, identify and correct the problem, then re-analyze all samples since the last acceptable CCV.
Laboratory Blank	In between analysis of standards and project samples	Results less than the laboratory Reporting Limit	Inspect the system and re-analyze the Laboratory Blank.
Surrogate	As each standard, blank, and sample is being loaded	75–150% on the FID	Low surrogate recovery results in re-analysis (at a higher dilution if high levels of moisture are present). If recovery is out and still low, report the analysis with the better recovery and flag. Because of TPH interference, high surrogate recoveries do not result in re-analysis. Data is flagged to note high recovery.
Laboratory Duplicate - Laboratory Control Spike Duplicate (LCSD)	One per analytical batch	RPD ≤ 25% for detections > 5 X RL	Narrate exceedances. Investigate the cause, perform maintenance as required, and re-calibrate as needed.

ANALYTICAL METHODS

Section 8.0

Method: EPA Method TO-12 (Non-methane Organic Compounds)

Eurofins Air Toxics SOP #36 Revision 20 Effective Date: August 11, 2018 Methods Manual Summary

Description: This method involves gas chromatograph analysis of whole air samples collected in Summa™ canisters or Tedlar bags. Samples are analyzed for Non-Methane Organic Compounds (NMOC) using EPA Method TO-12 protocols. After concentration on a sorbent bed, samples are analyzed using a Flame Ionization Detector (FID). This method is used when speciation is not required.

NMOC concentrations are quantified using the response factor of heptane. As required by the project, NMOC results referenced to heptane can be converted to units of ppmC (parts per million of Carbon). Additionally, hydrocarbon ranges can be provided based on the elution time of the normal alkanes on the GC column.

Eurofins Air Toxics performs a modified version for each of these methods. The method modifications, standard target analyte list, RL, QC criteria, and QC summary can be found in the following tables.

Table 1. Summary of Method Modifications for TO-12

Requirement	EPA Method TO-12	Eurofins Air Toxics Modifications
Reporting Limit	0.02 ppmC	0.050 ppmv
Initial Calibration	Five levels: Each level three runs with %RSD < 3%; linearity criterion not specified	Minimum of three single levels; %RSD ≤ 30%.
Sample Analysis Frequency	Duplicate analysis with RPD<5%; report average results of two analyses.	Single analysis. Duplicate 10% of samples with RPD ≤ 25% for detections > 5X the RL.
Column*	GC column not used.	GC column used for analysis.
Sample concentration	Cyrogenic concentration	Multibed sorbent concentration

* The column modification implemented for sample analysis allows for additional characterization based on carbon ranges.

Table 2. Method Compound List and QC Limits

Analyte	RL (ppmv)	Acceptance Criteria		
		ICAL (%RSD)	LCS/CCV (%R)	Precision (%RPD)
Total NMOC ref. to Heptane	0.050	≤ 30	75-125%	≤ 25

Table 3. Summary of Calibration and QC Procedures for TO-12 (NMOC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Curve (ICAL)	Prior to sample analysis and/or annually	% RSD ≤ 30	Repeat the calibration.
Laboratory Control Sample (LCS)	With each initial calibration and analytical batch	75–125% of the expected value	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis and after every 20 samples or at the end of the analytical sequence	% Difference ± 25 of expected value	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met. Re-analyze all samples since the last acceptable CCV.
Laboratory Blank	In between analysis of standards and project samples	Results less than laboratory reporting limit	Repeat the Laboratory Blank. If the re-analysis of the Lab Blank contains above but at less than 5X the reporting limit, sample analysis may proceed and the associated sample results will be reported with a B flag.
Laboratory Duplicates/ Laboratory Control Spike Duplicate (LCSD)	One per analytical batch	RPD ≤ 25%	Narrate exceedances. Investigate the cause and perform maintenance as required and re-calibrate as needed.

ANALYTICAL METHODS

Section 9.0

Method: EPA Method TO-13A PAHs (Full Scan and SIM)

Eurofins Air Toxics SOP #10	Revision 25	Effective Date: November 1, 2018	Methods Manual Summary
Eurofins Air Toxics SOP #74	Revision 15	Effective Date: October 12, 2018	Methods Manual Summary

Description: This method involves drawing a measured volume of air through a filter and sorbent cartridge to collect Polychlorinated Biphenyls (PAHs) in the vapor and particulate phases. The cartridge can be PUF/XAD2 or XAD2 only. While TO-13A describes the use of a high-volume sampling pump, which allows for up to 300 cubic meters (m^3) of air to be collected over a 24-hour period, the method can also be applied to low-volume sample applications suitable for indoor air or soil gas. The sample media is extracted in the laboratory using pressurized fluid extraction (PFE). The concentrated extracts are analyzed for PAHs using a quadrupole gas chromatograph/mass spectrometer (GC/MS) in full scan or SIM mode by TO-13A protocol. Eurofins Air Toxics performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation (LOQ), QC criteria, and QC summary can be found in the following tables.

In relation to the prescribed media, sampling and collection efficiencies for compounds not listed in TO-13A have not been evaluated. However, if non-standard compounds are required for a project, the laboratory reports these compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Required Field QC: EPA Method TO-13A requires at least one field blank per sampling episode. Matrix spikes are referenced, but not definitively required in the routine QA specifications.

Table 1. Summary of Method Modifications for TO-13A/TO-13A SIM

Requirements	EPA Method TO-13A	Eurofins Air Toxics Modifications
Filter Cleaning and Certification	TO-13A: Baked @ 400°C for 5 hours; extracted with DCM	Bake quartz fiber filters for at least 4 hrs at approximately 500°C, then extract one filter from a lot by Buchi E-914 with DCM. GC/MS analysis for TO-13A requirement of no target compound hits above RL.
XAD-2 Cleaning and Certification	TO-13A: DCM 2 X 16 hours. Extract cartridge.	Start with recycled XAD-2. Soak in Methanol for approximately 24 hours, (see section 7.1.2 for raw XAD-2 cleaning procedure) followed by extraction for a total time of at least 8 hours with solvent exchange after 4 hours. Approximately 20 mL of XAD-2 is placed in an extraction cell, and extracted by Buchi E-914 for 20 – 25 min.

Requirements	EPA Method TO-13A	Eurofins Air Toxics Modifications
PUF Cleaning and Certification	TO-13A: Acetone for 16 hours. Compound hits < 10 ng for TO-13A	Soak in Methanol for approximately 24 hours, followed by extraction for a minimum of 8 hours with solvent exchange after 4 hours. GC/MS analysis for TO-13A requirement of no target compound hits above the RL.
TO-13A Media Set-Up	TO-13A: Utilize PUF as primary sorbent; if Naphthalene, Acenaphthylene, & Acenaphthene needed then XAD is sorbent.	TO-13A: PUF/XAD-2 sandwich + filter. (PUF/XAD sandwich only for low-volume samples).
Extraction Solvent	PUF sorbent requires use of 10% Ether in Hexane; XAD sorbent requires use of DCM. Final extract in Hexane.	PUF/XAD-2 cartridge is used as sorbent with DCM as extraction solvent. Final extract in DCM.
Extraction Technique	Soxhlet apparatus extractor	Buchi E-914
Extract cleanup	Elute extract through silica gel prior to analysis.	No clean up used, experience shows that step does not improve method performance for typical air samples.
Pre-spike Surrogate	Requires 1.0 µg of Fluoranthene-d_{10} and 1.0 µg of Benzo(a)pyrene-d_{12} spiked on media prior to sampling.	Lab pre-spikes media for all TO-13A methods. For full scan analysis, a concentration of 50 µg is used. For SIM analysis, a concentration of 1.0 µg is used.
Media Certification	Extract one cartridge; criteria is <500 ng/cartridge for Naphthalene; <200 ng total/cartridge for rest of PAHs.	Filters, XAD-2, PUFs extracted and certified in pairs or individually; criteria is < RL for all target analytes.
Glassware Cleaning	Muffle furnace is utilized	Solvent cleaning procedure is used
Solvent Process Blank	One each analytical batch	Each solvent lot is certified
MS Detection Mode	Full Scan	Full Scan or SIM
Target Compound List	PAH list	See Table 2; Additional non-PAH semivolatile compounds can be analyzed; however, sample collection efficiencies have not beem evaluated. These compounds are outside the laboratory's NELAP scope of accreditation
Initial Calibration	0.1–2.5 µg/mL in Hexane	1.0–500 µg/mL in methylene chloride for standard (quad) or 0.1–40 µg/mL for SIM
Method Blank	< MDL	< Reporting Limit
Quantitation	Use RRF of CCV RRT ± 0.01 unit of the ICAL or CCV	TO-13A SIM: Use average RRF of the ICAL Absolute RT ± 0.06 min of CCV

Requirements	EPA Method TO-13A	Eurofins Air Toxics Modifications
Surrogate Recoveries	60-120%	50-150% for Field Surrogates Fluoranthene-d10 and Benzo(a)pyrene-d12

Table 2. Modified Method TO-13A/TO-13A SIM Analyte List and Reporting Limits

Analyte	SIM RL (µg)	RL (µg)	Minimum ICAL RRF	ICAL (%RSD)	ICV (%R)	CCV (%R)	Precision (%RPD)
2-Chloronaphthalene*	0.1	1.0	NA	≤ 30	± 30	± 30	≤ 25%
2-Methylnaphthalene*	0.1	1.0	NA	≤ 30	± 30	± 30	≤ 25%
Acenaphthylene	0.1	1.0	1.3	≤ 30	± 30	± 30	≤ 25%
Acenaphthene	0.1	1.0	0.8	≤ 30	± 30	± 30	≤ 25%
Anthracene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Benzo(a)anthracene	0.1	1.0	0.8	≤ 30	± 30	± 30	≤ 25%
Benzo(e)pyrene**	0.1	1.0	NA	≤ 30	± 30	± 30	≤ 25%
Benzo(a)pyrene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Benzo(b)fluoranthene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Benzo(g,h,i)perylene	0.1	1.0	0.5	≤ 30	± 30	± 30	≤ 25%
Benzo(k)fluoranthene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Chrysene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Dibenz(a,h)anthracene	0.1	1.0	0.4	≤ 30	± 30	± 30	≤ 25%
Fluoranthene	0.1	1.0	0.6	≤ 30	± 30	± 30	≤ 25%
Fluorene	0.1	1.0	0.9	≤ 30	± 30	± 30	≤ 25%
Indeno(1,2,3-c,d)pyrene	0.1	1.0	0.5	≤ 30	± 30	± 30	≤ 25%
Naphthalene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Phenanthrene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Pyrene	0.1	1.0	0.6	≤ 30	± 30	± 30	≤ 25%

* Non-standard analyte. Not included in the TO-13A method.
**No minimum requirement per EPA TO-13A.

The following compounds can be analyzed upon client request:

Analyte*	SIM RL (µg)	RL (µg)	Minimum ICAL RRF	ICAL (%RSD)	ICV (%R)	CCV (%R)	Precision (%RPD)
Perylene	1.0	1.0	0.5	≤ 30	± 30	± 30	≤ 25%
Coronene	1.0	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
1-Methylnaphthalene	1.0	1.0	N/A	≤ 30	± 30	± 30	≤ 25%

*Additional compounds may be available upon client request.

Table 3. Surrogates

Pre-Spike/Field Surrogates	Accuracy (%R)
Fluoranthene-d_{10}	50 – 150
Benzo(a)pyrene-d_{12}	50 – 150

Extraction Surrogates	Accuracy (%R)*
Fluorene-d_{10}	60 – 120
Pyrene-d_{10}	60 – 120

Table 4. Internal Standards

Analyte	Accuracy (%R)
Acenaphthene-d_{10}	-50 to +100
Chrysene-d_{12}	-50 to +100
1,4-Dichlorobenzene-$_{d4}$	-50 to +100
Naphthalene-d_8	-50 to +100
Perylene-d_{12}	-50 to +100
Phenanthrene-d_{10}	-50 to +100

Table 5. Extracted Laboratory Control Samples for TO-13A (PAHs) in Full Scan and SIM

Analyte	(%R)*
Naphthalene	60 – 120
Acenapthylene	60 – 120
Acenaphthene	60 – 120
Fluorene	60 – 120
Phenanthrene	60 – 120
Anthracene	60 – 120
Fluoranthene	60 – 120
Pyrene	60 – 120
Benzo(a)anthracene	60 – 120
Chrysene	60 – 120
Benzo(b)fluoranthene	60 – 120
Benzo(k)fluoranthene	60 – 120
Benzo(a)pyrene	60 – 120
Indeno(1,2,3-cd)pyrene	60 – 120
Dibenzo(a,h)anthracene	60 – 120
Benzo(g,h,i)perylene	60 – 120
2-Methylnaphthalene**	60 - 120
2-Chloronaphthalene**	60 – 120

*The LCS and Surrogate limits are derived from Compendium Method TO-13A, Sections 13.3.7.4 and 13.4.6.3 (January 1999). These limits only apply to samples that are extracted by Eurofins Air Toxics. When sample extracts are sent to the lab for analysis only, limits of 50-150 % are applied.
**These analytes are in addition to the mandated EPA TO-13A list and are required per NELAP to be included in the LCS spiking solution over a 2-year period.

Table 6. Summary of Calibration and QC Procedures for EPA Method TO-13A/TO-13A SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at start of every 12 hours	TO-13A tuning criteria	Correct problem then repeat tune.
Initial 5-Point Calibration	Prior to sample analysis	ICAL criteria in Table 2	Correct problem then repeat initial calibration.
ICV	All analytes: Once per initial calibration	All target compound recoveries must be between 70 – 130%	Determine the source of discrepancy between standards. Re-calibrate if needed.
Continuing Calibration Verification (CCV)	At the start of every clock immediately after the DFTPP tune check	PAHs list: Meet Table 2 Min. RRF requirement; %D ≤ 30%	Investigate and correct the problem, up to and including re-calibration if necessary. High bias associated with non-detects in samples will not result in re-analysis.
Internal Standards (IS)	Injected into each standard, blank, and sample extract prior to analysis	**For CCV:** Area count within 50% to 200% of the midpoint of ICAL. **For blanks, samples, and non-CCV QC checks:** retention times within ± 0.33 minutes (20 seconds) and area counts within 50% to 200% of the CCV.	**For CCVs:** Investigate and correct the problem before proceeding with sample analysis. **For blanks:** Inspect the system and re-analyze the blank. **For samples and non-CCV QC:** Unless there is obvious matrix effect, re-analyze the samples and dilute the sample until the ISs meet the criteria; narrate the data to indicate interference.
Surrogates	Field Surrogates: Blank cartridges prior to transport to field for sampling and lab QC prior to extraction. Extraction Surrogates: All samples and lab QC prior to extraction.	See Table 3.	A new aliquot of the extract is analyzed. If Surrogate recoveries are out-of-control a second time, data is flagged and narrated. Re-analysis is not necessary for obvious matrix effects (data is flagged for out-of-control surrogate recoveries). Air samples cannot be re-extracted.
Extracted Laboratory Control Samples (LCS)	With each set of up to 20 extracted samples	See LCS criteria in Table 5.	Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise, narrate.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Blank	With each set of up to 20 extracted samples	Results less than laboratory reporting limit (Table 2).	Re-aliquot and re-analyze the extract. If less than reporting limit, report the re-analysis. Otherwise, narrate and flag the data.
Solvent Blank	When samples that are extracted together are analyzed on different analytical shifts	All target compounds below the reporting limit (Table 2).	Re-aliquot and re-analyze the solvent. If less than reporting limit, report the re-analysis. Identify the source of contamination, and perform maintenance as needed. If maintenance required, restart the analytical clock.
Laboratory Duplicates – Laboratory Control Spike Duplicates (LCSD)	One per analytical batch	RPD ≤ 25%	Re-analyze duplicate. Investigate the cause and perform maintenance as required and re-calibrate as needed. Narrate exceedances if no error is identified.

ANALYTICAL METHODS

Section 10.0

Method: EPA Method TO-14A/TO-15 Volatile Organic Compounds (5&20 ppbv)

Eurofins Air Toxics SOP #91 Revision 16 Effective Date: June 11, 2019 Methods Manual Summary

Description: This method involves full scan gas chromatograph/mass spectrometer (GC/MS) analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds (VOCs) using EPA Method TO-14A/TO-15 protocols. An aliquot of up to 0.05 liters of air is withdrawn from the canister utilizing a volumetric syringe or mass flow controller. This volume is loaded onto a hydrophobic multibed sorbent trap to remove water and carbon dioxide and to concentrate the vapor sample. The focused sample is then flash-heated to sweep adsorbed VOCs onto a secondary trap for further concentration and/or onto a GC/MS for separation and detection.

Eurofins Air Toxics maintains a suite of TO-14A/TO-15 methods, each optimized to efficiently meet the data objectives for a wide range of targeted concentration ranges. The methods, their reporting limits, and typical applications are summarized in the table below. This method summary describes TO-14A/TO-15 (5&20). The 5&20 analytical configuration is designed to directly measure ppmv concentrations with minimal offline dilutions due to its wide dynamic calibration range.

Eurofins Air Toxics Method	Base Reporting Limits	Typical Application
TO-14A/TO-15 (5&20)	5 – 20 ppbv	Soil gas and ppmv range vapor matrices
TO-14A/TO-15 (Standard or Quad)	0.5 – 5.0 ppbv	Ambient air, soil gas, and ppbv level vapor matrices
TO-15 (Extended)	0.2 – 5.0 ppbv	Ambient air and ppbv level vapor matrices
TO-14A/TO-15 (Low-level)	0.1 – 1.0 ppbv	Indoor and outdoor air
TO-14A/TO-15 SIM	0.01 – 0.5 ppbv	Indoor and outdoor air
TO-15 HSS	0.01 – 0.1 ppbv	Soil gas and other high concentration matrices

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, Eurofins Air Toxics reports these non-routine compounds with partial validation. Validation may include a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification analyzed, and no method detection limit study performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Eurofins Air Toxics takes no modifications of technical significance to Method TO-15 for the "5&20" configuration. Since Eurofins Air Toxics applies TO-15 methodology to all Summa canisters regardless of whether TO-14A or TO-15 is specified by the project, the laboratory performs a modified version of method TO-14A as detailed in Table 1. Please note that Methods TO-14A and TO-15 were validated for specially treated canisters. As such, the use of

Tedlar bags for sample collection is outside the scope of the method and not recommended for ambient air samples. It is the responsibility of the data user to determine the usability of TO-14A and TO-15 results generated from Tedlar bags.

Table 1. Summary of TO-14A Method Modifications

Requirement	TO-14A	ATL Modifications
Sample Drying System	Nafion Drier	Multibed hydrophobic sorbent
Blank acceptance criteria	< 0.2 ppbv	< RL
BFB ion abundance criteria	Ion abundance criteria listed in Table 4 of TO-14A	Follow abundance criteria listed in TO-15
BFB absolute abundance criteria	Within 10% when comparing to the previous daily BFB	CCV internal standard area counts are compared to ICAL; corrective action when recovery is less than 60%.
Initial Calibration	≤ 30% RSD for listed 39 VOCs	≤ 30% RSD with 2 of Eurofins Air Toxics' 62 standard compounds allowed out to ≤ 40%

The standard target analyte list, reporting limit (RL), also referred to as Limit of Quantitation (LOQ), QC criteria, and QC summary can be found in Tables 2 through 5.

Table 2. Method TO-14A/TO-15 Analyte List (5&20)

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,1,2-Trichloroethane	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethane	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethene	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,2,4-Trichlorobenzene	20	≤ 30%	70 – 130	70 – 130	± 25
1,2,4-Trimethylbenzene	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dibromoethane (EDB)	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichlorobenzene	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloroethane	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloropropane	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,3,5-Trimethylbenzene	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,3-Dichlorobenzene	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dichlorobenzene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Benzene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Bromomethane	20	≤ 30%	70 – 130	70 – 130	± 25
Carbon Tetrachloride	5.0	≤ 30%	70 – 130	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Chlorobenzene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Chloroethane	20	≤ 30%	70 – 130	70 – 130	± 25
Chloroform	5.0	≤ 30%	70 – 130	70 – 130	± 25
Chloromethane	20	≤ 30%	70 – 130	70 – 130	± 25
Chlorotoluene (Benzyl Chloride)	5.0	≤ 30%	70 – 130	70 – 130	± 25
cis-1,2-Dichloroethene	5.0	≤ 30%	70 – 130	70 – 130	± 25
cis-1,3-Dichloropropene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Dichloromethane (Methylene Chloride)	20	≤ 30%	70 – 130	70 – 130	± 25
Ethylbenzene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Freon 11 (Trichlorofluoromethane)	5.0	≤ 30%	70 – 130	70 – 130	± 25
Freon 113 (Trichlorotrifluoroethane)	5.0	≤ 30%	70 – 130	70 – 130	± 25
Freon 114	5.0	≤ 30%	70 – 130	70 – 130	± 25
Freon 12 (Dichlorodifluoromethane)	5.0	≤ 30%	70 – 130	70 – 130	± 25
Hexachlorobutadiene	20	≤ 30%	70 – 130	70 – 130	± 25
m,p-Xylene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Methyl Chloroform (1,1,1-Trichloroethane)	5.0	≤ 30%	70 – 130	70 – 130	± 25
o-Xylene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Styrene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Tetrachloroethene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Toluene	5.0	≤ 30%	70 – 130	70 – 130	± 25
trans-1,3-Dichloropropene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Trichloroethene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Vinyl Chloride	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,3-Butadiene	5.0	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dioxane	20	≤ 30%	70 – 130	70 – 130	± 25
2-Butanone (Methyl Ethyl Ketone)	20	≤ 30%	70 – 130	70 – 130	± 25
2-Hexanone	20	≤ 30%	70 – 130	70 – 130	± 25
4-Ethyltoluene	5.0	≤ 30%	70 – 130	70 – 130	± 25
4-Methyl-2-Pentanone (MIBK)	5.0	≤ 30%	70 – 130	70 – 130	± 25
Acetone	20	≤ 30%	70 – 130	70 – 130	± 25
Bromodichloromethane	5.0	≤ 30%	70 – 130	70 – 130	± 25
Bromoform	5.0	≤ 30%	70 – 130	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Carbon Disulfide	20	≤ 30%	70 – 130	70 – 130	± 25
Cyclohexane	5.0	≤ 30%	70 – 130	70 – 130	± 25
Dibromochloromethane	5.0	≤ 30%	70 – 130	70 – 130	± 25
Ethanol	20	≤ 30%	70 – 130	70 – 130	± 25
Heptane	5.0	≤ 30%	70 – 130	70 – 130	± 25
Hexane	5.0	≤ 30%	70 – 130	70 – 130	± 25
Isopropanol	20	≤ 30%	70 – 130	70 – 130	± 25
Methyl t-Butyl Ether (MTBE)	5.0	≤ 30%	70 – 130	70 – 130	± 25
Tetrahydrofuran	5.0	≤ 30%	70 – 130	70 – 130	± 25
trans-1,2-Dichloroethene	5.0	≤ 30%	70 – 130	70 – 130	± 25
2,2,4-Trimethylpentane	5.0	≤ 30%	70 – 130	70 – 130	± 25
Cumene	5.0	≤ 30%	70 – 130	70 – 130	± 25
Propylbenzene	5.0	≤ 30%	70 – 130	70 – 130	± 25
3-Chloroprene	20	≤ 30%	70 – 130	70 – 130	± 25
Naphthalene*	20	≤ 40%	60 – 140	60 – 140	± 25
TPH (Gasoline) **	200	1- Point Calibration	NA	ICV only: 60 – 140	± 25
NMOC (Hexane/Heptane)**	100	1- Point Calibration	NA	NA	± 25

*Due to its low vapor pressure, Naphthalene may exceed TO-15 performance requirements. The wider QC limits reflect typical performance. Although Naphthalene is not on Eurofins Air Toxics "standard" TO-15 list, it is commonly requested and included in Table 2.

**TPH and NMOC are not on Eurofins Air Toxics' "standard" TO-15 list, but are included in Table 2 due to common requests.

Table 2 is the list of Standard compounds, reporting limits and QC acceptance criteria. Each project may be customized as needed. Additional compounds and different reporting limits may be obtainable and/or achieved upon request.

Table 3. Internal Standards

Analyte	Accuracy (% R)
Bromochloromethane	60 – 140
1,4-Difluorobenzene	60 – 140
Chlorobenzene-d_5	60 – 140

Table 4. Surrogates

Analyte	Accuracy (% R)
1,2-Dichloroethane-d_4	70 – 130
Toluene-d_8	70 – 130
4-Bromofluorobenzene	70 – 130

Table 5. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15 (5&20)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours.	TO-15 ion abundance criteria	Correct problem then repeat tune.
Minimum 5-Point Initial Calibration (ICAL)	Prior to sample analysis.	% RSD ≤ 30 with 2 compounds allowed out to ≤ 40% RSD	Correct problem then repeat Initial Calibration Curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each Initial Calibration curve, and daily prior to sample analysis	Recoveries for 85% of "Standard" compounds must be 70-130%. No recovery may be <50%. ICV evaluated on a full list basis at the time of calibration. If specified by the project, in-house generated control limits may be used.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) for Non-standard compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60–140%. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Continuing Calibration Verification (CCV)	At the start of each analytical clock after the tune check.	70–130%	Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than two compounds from the standard list recover outside of 70-130% or > 10% of VOCs if short list is used (20 compounds or less), corrective action will be taken. If any compound exceeds 60-140%, samples are not analyzed unless data meets project needs. Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit	Inspect the system and re-analyze the blank. "B"-flag data for common contaminants.
Internal Standard (IS)	As each standard, blank, and sample is being loaded	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±40% of the area counts of the daily CCV internal standards.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.
Surrogates	As each standard, blank, and sample is being loaded.	70–130% If specified by the project, in-house generated control limits may be used.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** re-analyze the sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %Rs are out-of-limits a second time, report data from first analysis and narrate.
Laboratory Duplicates – Laboratory Control Spike Duplicates (LCSD)	One per analytical batch	RPD ≤25%	Narrate exceedances. If more than 5% of compound list is outside criteria or if compound has >40%RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

ANALYTICAL METHODS

Section 11.0

Method: EPA Method TO-14A/TO-15 Volatile Organic Compounds (Standard/Quad)

Eurofins Air Toxics SOP #6 Revision 39 Effective Date: March 26, 2019 Methods Manual Summary

Description: This method involves full scan gas chromatograph/mass spectrometer (GC/MS) analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds (VOCs) using EPA Method TO-14A/TO-15 protocols. An aliquot of up to 0.2 liters of air is withdrawn from the canister utilizing a volumetric syringe, volumetric loop, or mass flow controller. This volume is loaded onto a hydrophobic multibed sorbent trap to remove water and carbon dioxide and to concentrate the vapor sample. The focused sample is then flash-heated to sweep adsorbed VOCs onto a secondary trap for further concentration and/or directly onto a GC/MS for separation and detection.

Eurofins Air Toxics maintains a suite of TO-14A/TO-15 methods, each optimized to efficiently meet the data objectives for a wide range of targeted concentration ranges. The methods, their reporting limits, and typical applications are summarized in the table below. This method summary describes TO-14A/TO-15 (Standard or Quad).

Eurofins Air Toxics Method	Base Reporting Limits	Typical Application
TO-14A/TO-15 (5&20)	5 – 20 ppbv	Soil gas and ppmv range vapor matrices
⇨ TO-14A/TO-15 (Standard or Quad)	0.5 – 5.0 ppbv	Ambient air, soil gas, and ppbv level vapor matrices
TO-15 (Extended)	0.2 – 5.0 ppbv	Ambient air and ppbv level vapor matrices
TO-14A/TO-15 (Low-level)	0.1 – 1.0 ppbv	Indoor and outdoor air
TO-14A/TO-15 SIM	0.01 – 0.5 ppbv	Indoor and outdoor air
TO-15 HSS	0.01 – 0.1 ppbv	Soil gas and other high concentration matrices

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, Eurofins Air Toxics reports these non-routine compounds with partial validation. Validation may include a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification analyzed, and no method detection limit study performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Eurofins Air Toxics takes no modifications of technical significance to Method TO-15 for the "Quad" configurations. Since Eurofins Air Toxics applies TO-15 methodology to all Summa canisters regardless of whether TO-14A or TO-15 is specified by the project, the laboratory performs a modified version of method TO-14A as detailed in Table 1. Please note that Methods TO-14A and TO-15 were validated for specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of the method and not

recommended for ambient or indoor air samples. It is the responsibility of the data user to determine the usability of TO-14A and TO-15 results generated from Tedlar bags.

Table 1. Summary of TO-14A Method Modifications

Requirement	TO-14A	Eurofins Air Toxics Modifications
Sample Drying System	Nafion Drier	Multibed hydrophobic sorbent.
Blank acceptance criteria	≤ 0.2 ppbv	≤ RL
BFB ion abundance criteria	Ion abundance criteria listed in Table 4 of TO-14A	Follow abundance criteria listed in TO-15.
BFB absolute abundance criteria	Within 10% when comparing to the previous daily BFB	CCV internal standard area counts are compared to ICAL; corrective action when recovery is less than 60%.
Initial Calibration	≤ 30% RSD for listed 39 VOCs	Follow TO-15 requirements of ≤ 30% RSD with 2 of Eurofins Air Toxics' 62 standard compounds allowed out to ≤ 40% RSD

The standard target analyte list, reporting limit (RL) also referred to as Limit of Quantitation, QC criteria, and QC summary can be found in Tables 2 through 5.

Table 2. Method TO-14A/TO-15 Analyte List (Quad)

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,1,2-Trichloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2,4-Trichlorobenzene	2.0	≤ 30%	70 – 130	70 – 130	± 25
1,2,4-Trimethylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dibromoethane (EDB)	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloropropane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,3,5-Trimethylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,3-Dichlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dichlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Benzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Bromomethane	5.0	≤ 30%	70 – 130	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Carbon Tetrachloride	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chloroethane	2.0	≤ 30%	70 – 130	70 – 130	± 25
Chloroform	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chloromethane	5.0	≤ 30%	70 – 130	70 – 130	± 25
Chlorotoluene (Benzyl Chloride)	0.5	≤ 30%	70 – 130	70 – 130	± 25
cis-1,2-Dichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
cis-1,3-Dichloropropene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Dichloromethane (Methylene Chloride)	5.0	≤ 30%	70 – 130	70 – 130	± 25
Ethylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 11 (Trichlorofluoromethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 113 (Trichlorotrifluoroethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 114	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 12 (Dichlorodifluoromethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Hexachlorobutadiene	2.0	≤ 30%	70 – 130	70 – 130	± 25
m,p-Xylene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Methyl Chloroform (1,1,1-Trichloroethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
o-Xylene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Styrene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Tetrachloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Toluene	0.5	≤ 30%	70 – 130	70 – 130	± 25
trans-1,3-Dichloropropene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Trichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Vinyl Chloride	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,3-Butadiene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dioxane	2.0	≤ 30%	70 – 130	70 – 130	± 25
2-Butanone (Methyl Ethyl Ketone)	2.0	≤ 30%	70 – 130	70 – 130	± 25
2-Hexanone	2.0	≤ 30%	70 – 130	70 – 130	± 25
4-Ethyltoluene	0.5	≤ 30%	70 – 130	70 – 130	± 25
4-Methyl-2-Pentanone (MIBK)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Acetone	5.0	≤ 30%	70 – 130	70 – 130	± 25
Bromodichloromethane	0.5	≤ 30%	70 – 130	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Bromoform	0.5	≤ 30%	70 – 130	70 – 130	± 25
Carbon Disulfide	2.0	≤ 30%	70 – 130	70 – 130	± 25
Cyclohexane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Dibromochloromethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Ethanol	2.0	≤ 30%	70 – 130	70 – 130	± 25
Heptane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Hexane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Isopropanol	2.0	≤ 30%	70 – 130	70 – 130	± 25
Methyl t-Butyl Ether (MTBE)	2.0	≤ 30%	70 – 130	70 – 130	± 25
Tetrahydrofuran	0.5	≤ 30%	70 – 130	70 – 130	± 25
trans-1,2-Dichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
2,2,4-Trimethylpentane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Cumene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Propylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
3-Chloroprene	2.0	≤ 30%	70 – 130	70 – 130	± 25
Naphthalene*	1.0	≤40%	60 – 140	60 – 140	± 25
TPH (Gasoline) **	50	1-Point Calibration	N/A	ICV only; 60 – 140	± 25
NMOC (Hexane/Heptane)**	10	1-Point Calibration	N/A	NA	± 25

*Due to its low vapor pressure, Naphthalene may exceed TO-15 performance requirements. The wider QC limits reflect typical performance. Although Naphthalene is not on Eurofins Air Toxics "standard" TO-15 list, it is commonly requested and included in Table 2.

**TPH and NMOC are not on Eurofins Air Toxics' "standard" TO-15 list, but are included in Table 2 due to common requests.

Table 2 is the list of Standard compounds, reporting limits and QC acceptance criteria. Each project may be customized as needed. Additional compounds and different reporting limits may be obtainable and/or achieved upon request.

Table 3. Internal Standards

Analyte	Accuracy (% R)
Bromochloromethane	60 – 140
1,4-Difluorobenzene	60 – 140
Chlorobenzene-d_5	60 – 140

Table 4. Surrogates

Analyte	Accuracy (% R)
1,2-Dichloroethane-d_4	70 – 130
Toluene-d_8	70 – 130
4-Bromofluorobenzene	70 – 130

Table 5. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours	TO-15 ion abundance criteria	Correct problem then repeat tune.
Minimum 5-Point Initial Calibration (ICAL)	Prior to sample analysis	% RSD ≤ 30 with 2 compounds allowed out to ≤ 40% RSD	Correct problem then repeat Initial Calibration curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each Initial Calibration curve, and daily prior to sample analysis	Recoveries for 85% of "Standard" compounds must be 70–130%. No recovery may be < 50%. ICV evaluated on a full list basis at the time of calibration. If specified by the project, in-house generated control limits may be used.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) for Non-standard compounds	Per client request or specific project requirements only	Recoveries of compounds must be 60–140%. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification (CCV) for Standard compounds	At the start of each analytical clock after the tune check	70–130%	Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than two compounds from the standard list recover outside of 70–130% or > 10% of VOCs if short list is used (20 compounds or less), corrective action will be taken. If any compound exceeds 60–140%, samples are not analyzed unless data meets project needs. Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV) for Non-standard Compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60–140%. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit	Inspect the system and re-analyze the blank. "B"-flag data for common contaminants.
Internal Standard (IS)	As each standard, blank, and sample is being loaded	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±40% of the area counts of the daily CCV internal standards.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogates	As each standard, blank, and sample is being loaded	70–130% If specified by the project, in-house generated control limits may be used.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %Rs are out-of-limits a second time, report data from first analysis and narrate.
Laboratory Duplicates – Laboratory Control Spike Duplicates (LCSD)	One per analytical batch	RPD ≤25%	Narrate exceedances. If more than 5% of compound list is outside criteria or if compound has >40%RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

ANALYTICAL METHODS

Section 12.0

Method: EPA Method TO-15 Volatile Organic Compounds (Extended)

Eurofins Air Toxics SOP #132 Revision 6 Effective Date: January 24, 2019 Methods Manual Summary

Description: This method involves full scan gas chromatograph/mass spectrometer (GC/MS) analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds (VOCs) using EPA Method TO-15 protocols. An aliquot of up to 0.5 liters of air is withdrawn from the canister utilizing a mass flow controller. This volume is loaded onto a hydrophobic multibed sorbent trap to remove water and carbon dioxide and to concentrate the vapor sample. The focused sample is then flash-heated to sweep adsorbed VOCs onto a secondary trap for further concentration and/or directly onto a GC/MS for separation and detection.

Eurofins Air Toxics maintains a suite of TO-15 methods, each optimized to efficiently meet the data objectives for a wide range of targeted concentration ranges. The methods, their reporting limits, and typical applications are summarized in the table below. This method summary describes TO-15 (Extended).

Eurofins Air Toxics Method	Base Reporting Limits	Typical Application
TO-14A/TO-15 (5&20)	5 – 20 ppbv	Soil gas and ppmv range vapor matrices
TO-14A/TO-15 (Standard or Quad)	0.5 – 5.0 ppbv	Ambient air, soil gas, and ppbv level vapor matrices
⇨ TO-15 (Extended)	0.2 – 5.0 ppbv	Ambient air and ppbv level vapor matrices
TO-14A/TO-15 (Low-level)	0.1 – 1.0 ppbv	Indoor and outdoor air
TO-14A/TO-15 (SIM)	0.01 – 0.5 ppbv	Indoor and outdoor air
TO-15 HSS	0.01 – 0.1 ppbv	Soil gas and other high concentration matrices

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, Eurofins Air Toxics reports these non-routine compounds with partial validation. Validation may include a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification analyzed, and no method detection limit study performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

The laboratory performs a modified version of method TO-15 as detailed in Table 1. Please note that Method TO-15 was validated for specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of the method and not recommended for ambient or indoor air samples. It is the responsibility of the data user to determine the usability of TO-15 results generated from Tedlar bags.

Table 1. Summary of TO-15 (Extended) Method Modifications

Requirement	TO-15	EATL Modifications
Initial Calibration	≤ 30% RSD with 2 compounds allowed out to ≤40%RSD	Due to the large number of target compounds, the acceptance criterion is ≤30%RSD with 5 out ≤ 40%RSD for the list of 110 certified compounds. Methanol, Acetaldehyde and 1,4-Dioxane are evaluated against ≤50%RSD
Daily Continuing Calibration Verification (CCV)	±30% Difference	±30% D for a specified list of compounds with a subset of compounds set at ±40%D or ±50%D, with 5 compounds allowed outside this criteria.
Sample Internal Standard Recovery	±40% of CCV IS Area	±50% of CCV IS Area

The standard target analyte list, reporting limit (RL) also referred to as Limit of Quantitation, QC criteria, and QC summary can be found in Tables 2 through 5.

Table 2. Method TO-15 Analyte List (Extended) and QC Limits

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%D)	ICV/LCS* (%R)	Precision Limits (Max. RPD)
Ethylene	0.5	≤30%	≤30	70 - 130	± 25
Acetylene	0.5	≤30%	≤50	50 - 150	± 25
Ethane	0.5	≤30%	≤30	70 - 130	± 25
Propylene	0.2	≤30%	≤30	70 - 130	± 25
Propane	0.2	≤30%	≤30	70 - 130	± 25
Dichlorodiluoromethane/Fr12	0.2	≤30%	≤30	70 - 130	± 25
Chloromethane	0.2	≤30%	≤30	70 - 130	± 25
Isobutane	0.2	≤30%	≤30	70 - 130	± 25
Freon 114	0.2	≤30%	≤30	70 - 130	± 25
Acetaldehyde	2.0	≤50%	≤50	50 - 150	± 50
Vinyl Chloride	0.2	≤30%	≤30	70 - 130	± 25
1-Butene/Isobutene*	0.2	≤30%	≤30	70 - 130	± 25
1,3-Butadiene	0.5	≤30%	≤30	70 - 130	± 25
Butane	0.2	≤30%	≤30	70 - 130	± 25
Methanol	5.0	≤50%	≤50	50 – 150	± 50
trans-2-Butene	0.2	≤30%	≤30	70 – 130	± 25
Bromomethane	0.2	≤30%	≤30	70 – 130	± 25
cis-2-Butene	0.2	≤30%	≤30	70 – 130	± 25
Chloroethane	0.5	≤30%	≤30	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%D)	ICV/LCS* (%R)	Precision Limits (Max. RPD)
Dichlorofluoromethane/Fr21	0.2	≤30%	≤30	70 – 130	± 25
Vinyl Bromide	0.2	≤30%	≤30	70 – 130	± 25
3-Methyl-1-Butene	0.2	≤30%	≤30	70 – 130	± 25
Acetonitrile	2.0	≤30%	≤50	50 – 150	± 25
Isopentane	0.2	≤30%	≤30	70 – 130	± 25
Trichlorofluoromethane/Fr11	0.2	≤30%	≤30	70 – 130	± 25
1-Pentene	0.2	≤30%	≤30	70 – 130	± 25
Pentane	0.2	≤30%	≤30	70 – 130	± 25
Acrylonitrile	2.0	≤30%	≤40	60 – 140	± 25
Isoprene	0.2	≤30%	≤30	70 – 130	± 25
trans-2-Pentene	0.2	≤30%	≤30	70 – 130	± 25
1,1-Dichloroethene	0.2	≤30%	≤30	70 – 130	± 25
cis-2-Pentene	0.2	≤30%	≤30	70 – 130	± 25
Methylene Chloride	0.2	≤30%	≤30	70 – 130	± 25
2-Methyl-2-Butene	0.2	≤30%	≤30	70 – 130	± 25
Freon 113	0.2	≤30%	≤30	70 – 130	± 25
2,2,-Dimethylbutane	0.2	≤30%	≤30	70 – 130	± 25
Cyclopentene	0.2	≤30%	≤30	70 – 130	± 25
trans-1,2-Dichloroethene	0.2	≤30%	≤30	70 – 130	± 25
4-Methyl-1-Pentene	0.2	≤30%	≤30	70 – 130	± 25
1,1-Dichloroethane	0.2	≤30%	≤30	70 – 130	± 25
Cyclopentane	0.2	≤30%	≤30	70 – 130	± 25
2,3-Dimethylbutane	0.2	≤30%	≤30	70 – 130	± 25
Methyl tert Butyl Ether/MTBE	0.2	≤30%	≤30	70 – 130	± 25
2-Methylpentane	0.2	≤30%	≤30	70 – 130	± 25
Vinyl Acetate	2.0	≤30%	≤40	60 – 140	± 25
2-Butanone	2.0	≤30%	≤40	60 – 140	± 25
3-Methylpentane	0.2	≤30%	≤30	70 – 130	± 25
Chloroprene	0.2	≤30%	≤30	70 – 130	± 25
cis-1,2-Dichloroethene	0.2	≤30%	≤30	70 – 130	± 25
Bromochloromethane	0.2	≤30%	≤30	70 – 130	± 25
Hexane	0.2	≤30%	≤30	70 – 130	± 25
Chloroform	0.2	≤30%	≤30	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%D)	ICV/LCS* (%R)	Precision Limits (Max. RPD)
trans-2-Hexene	0.2	≤30%	≤30	70 – 130	± 25
Cis-2-Hexene	0.2	≤30%	≤30	70 – 130	± 25
Methylcyclopentane	0.2	≤30%	≤30	70 – 130	± 25
1,2-Dichloroethane	0.2	≤30%	≤30	70 – 130	± 25
2,4-Dimethylpentane	0.2	≤30%	≤30	70 – 130	± 25
1,1,1-Trichloroethane	0.2	≤30%	≤30	70 – 130	± 25
Benzene	0.2	≤30%	≤30	70 – 130	± 25
Carbon Tetrachloride	0.2	≤30%	≤30	70 – 130	± 25
Cyclohexane	0.2	≤30%	≤30	70 – 130	± 25
2-Methylhexane	0.2	≤30%	≤30	70 – 130	± 25
2,3-Dimethylpentane	0.2	≤30%	≤30	70 – 130	± 25
3-Methylhexane	0.2	≤30%	≤30	70 – 130	± 25
1,2-Dichloropropane	0.2	≤30%	≤30	70 – 130	± 25
Bromodichloromethane	0.2	≤30%	≤30	70 – 130	± 25
Trichloroethene	0.2	≤30%	≤30	70 – 130	± 25
2,2,4-Trimethylpentane	0.2	≤30%	≤30	70 – 130	± 25
1,4-Dioxane	0.5	≤50%	≤50	50 – 150	± 50
Heptane	0.2	≤30%	≤30	70 – 130	± 25
cis-1,3-Dichloropropene	0.2	≤30%	≤30	70 – 130	± 25
4-Methyl-2-pentanone	0.5	≤30%	≤40	60 – 140	± 25
Methylcyclohexane	0.2	≤30%	≤30	70 – 130	± 25
trans-1,3-Dichloropropene	0.2	≤30%	≤30	70 – 130	± 25
1,1,2-Trichloroethane	0.2	≤30%	≤30	70 – 130	± 25
2,3,4-Trimethylpentane	0.2	≤30%	≤30	70 – 130	± 25
Toluene	0.2	≤30%	≤30	70 – 130	± 25
2-Methylheptane	0.2	≤30%	≤30	70 – 130	± 25
3-Methylheptane	0.2	≤30%	≤30	70 – 130	± 25
Dibromochloromethane	0.2	≤30%	≤30	70 – 130	± 25
1,2-Dibromoethane	0.2	≤30%	≤30	70 – 130	± 25
Octane	0.2	≤30%	≤30	70 – 130	± 25
Tetrachloroethene	0.2	≤30%	≤30	70 – 130	± 25
Chlorobenzene	0.2	≤30%	≤30	70 – 130	± 25
Ethylbenzene	0.2	≤30%	≤30	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%D)	ICV/LCS* (%R)	Precision Limits (Max. RPD)
m,p-Xylene	0.2	≤30%	≤30	70 – 130	± 25
Bromoform	0.2	≤30%	≤30	70 – 130	± 25
Styrene	0.2	≤30%	≤40	60 – 140	± 25
1,1,2,2,-Tetrachloroethane	0.2	≤30%	≤30	70 – 130	± 25
o-Xylene	0.2	≤30%	≤30	70 – 130	± 25
Nonane	0.2	≤30%	≤30	70 – 130	± 25
Cumene	0.2	≤30%	≤30	70 – 130	± 25
Propylbenzene	0.2	≤30%	≤30	70 – 130	± 25
4-Ethyltoluene	0.2	≤30%	≤30	70 – 130	± 25
1,3,5-Trimethylbenzene	0.2	≤30%	≤30	70 – 130	± 25
1,2,4-Trimethylbenzene	0.2	≤30%	≤30	70 – 130	± 25
2-Ethyltoluene	0.2	≤30%	≤30	70 – 130	± 25
3-Ethyltoluene	0.2	≤30%	≤30	70 – 130	± 25
alpha-Chlorotoluene	0.2	≤30%	≤50	50 – 150	± 25
Decane	0.5	≤30%	≤30	70 – 130	± 25
1,3-Dichlorobenzene	0.2	≤30%	≤30	70 – 130	± 25
1,4-Dichlorobenzene	0.2	≤30%	≤30	70 – 130	± 25
1,2,3-Trimethylbenzene	0.2	≤30%	≤30	70 – 130	± 25
1,2-Dichlorobenzene	0.2	≤30%	≤30	70 – 130	± 25
1,3-Diethylbenzene	0.2	≤30%	≤30	70 – 130	± 25
1,4-Diethylbenzene	0.2	≤30%	≤30	70 – 130	± 25
Undecane	0.2	≤30%	≤30	70 – 130	± 25
1,2,4-Trichlorobenzene	0.2	≤30%	≤50	50 – 150	± 25
Hexachlorobutadiene	0.2	≤30%	≤50	50 – 150	± 25

* 1-Butene and Isobutene co-elute. The response of 1-Butene will be used to report the compounds.

Table 3. TO-15 (Extended) Non-Standard Compounds and QC Limits

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria	
		ICAL (%RSD)	ICV (%R)
Freon 134a	0.2	≤40%	60 – 140
Freon 22 (Chlorodifluoromethane)	0.2	≤40%	60 – 140
Neopentane	0.2	≤40%	60 – 140
Ethanol	2.0	≤40%	60 – 140
Acrolein	2.0	≤40%	60 – 140

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria	
		ICAL (%RSD)	ICV (%R)
Acetone	2.0	≤40%	60 – 140
2-Propanol	2.0	≤40%	60 – 140
Diethyl Ether (Ethyl Ether)	2.0	≤40%	60 – 140
3-Chloroprene	0.2	≤40%	60 – 140
Carbon Disulfide	0.2	≤40%	60 – 140
1-Propanol	2.0	≤40%	60 – 140
cis-4-Methyl-2-pentene	0.2	≤40%	60 – 140
trans-4-Methyl-2-pentene	0.2	≤40%	60 – 140
Butyraldehyde (Butanal)	2.0	≤40%	60 – 140
1-Hexene/2-Methyl-1-pentene	0.4	≤40%	60 – 140
2-Ethyl-1-Butene	0.2	≤40%	60 – 140
cis-3-Hexene	0.2	≤40%	60 – 140
2-Methyl-2-pentene	0.2	≤40%	60 – 140
cis-3-Methyl-2-pentene	0.2	≤40%	60 – 140
Tetrahydofuran	2.0	≤40%	60 – 140
Methylcyclopentene	0.2	≤40%	60 – 140
1-Butanol (n-Butanol)	2.0	≤40%	60 – 140
Cyclohexene	0.2	≤40%	60 – 140
1-Heptene	0.2	≤40%	60 – 140
2-Chloropentane	0.2	≤40%	60 – 140
trans-3-Heptene	0.2	≤40%	60 – 140
cis-3-Heptene	0.2	≤40%	60 – 140
Trans-2-Heptene	0.2	≤40%	60 – 140
2,4,4-Trimethyl-1-pentene	0.2	≤40%	60 – 140
2,4,4-Trimethyl-2-pentene	0.2	≤40%	60 – 140
2,5-Dimethylhexane	0.2	≤40%	60 – 140
2,2,3-Trimethylpentane	0.2	≤40%	60 – 140
1-Methylcyclohexene	0.2	≤40%	60 – 140
2-Hexanone	0.2	≤40%	60 – 140
Hexanal	0.2	≤40%	60 – 140
2,2,5-Trimethylhexane	0.2	≤40%	60 – 140
1-Octene	0.2	≤40%	60 – 140
cis-2-Octene	0.2	≤40%	60 – 140
Butyl Acrylate	2.0	≤40%	60 – 140

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria	
		ICAL (%RSD)	ICV (%R)
Heptanal	0.2	≤40%	60 – 140
1-Nonene	0.2	≤40%	60 – 140
cis-4-Nonene	0.2	≤40%	60 – 140
trans-4-Nonene *	0.2	≤40%	60 – 140
Benzaldehyde	0.2	≤40%	60 – 140
2-,3-Chlorotoluene	0.4	≤40%	60 – 140
4-Chlorotoluene	0.2	≤40%	60 – 140
beta-Pinene	0.2	≤40%	60 – 140
Alpha-Pinene **	0.2	≤40%	60 – 140
1-Decene	0.2	≤40%	60 – 140
tert-Butylbenzene	0.2	≤40%	60 – 140
Isobutylbenzene	0.2	≤40%	60 – 140
4-Isopropyltoluene (p-Cymene)	0.2	≤40%	60 – 140
d-Limonene	0.2	≤40%	60 – 140
Indane (Indan)	0.2	≤40%	60 – 140
Indene	0.2	≤40%	60 – 140
Butylbenzene	0.2	≤40%	60 – 140
1-Undecene	0.2	≤40%	60 – 140
Naphthalene	0.2	≤40%	60 – 140

* Standard not available. The Response from cis-4-Nonene is used to calculate concentrations.
** Standard not available. The Response from beta-Pinene is used to calculate concentrations.

Table 4. Internal Standards

Analyte	Accuracy (% R)
1,4-Difluorobenzene	50 – 150
Chlorobenzene-d_5	50 – 150

Table 5. Surrogates

Analyte	Accuracy (% R)*
2-Bromo-1,1,1-Trifluoroethane	70 – 130
Fluorobenzene	70 – 130
Toluene-d_8	70 – 130
1,4-Dichlorobutane	70 – 130
4-Bromofluorobenzene	70 – 130

* In-house generated control limits may be used.

Table 6. Summary of Calibration and QC Procedures for Method TO-15 (Extended)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours	TO-15 ion abundance criteria	Correct problem then repeat tune.
Minimum 5-Point Initial Calibration (ICAL)	Prior to sample analysis	% RSD ≤ 30 with 5% of compounds allowed out to ≤ 40% RSD	Correct problem then repeat Initial Calibration Curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each Initial Calibration curve, and daily prior to sample analysis	Recoveries for 85% of "Standard" compounds must be 70–130%. No recovery may be < 50%. If specified by the project, in-house generated control limits may be used.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) for Non-standard compounds	After each initial calibration curve.	Recoveries of compounds must be 60–140% for 85% of the compounds. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Continuing Calibration Verification (CCV) for Standard compounds	At the start of each analytical clock after the tune check	70–130% (See Tables above for exceptions.)	Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than 5% of the compounds from the standard list recover outside of 70–130%, corrective action will be taken. If any compound exceeds 60–140%, samples are not analyzed unless data meets project needs. Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV) for Non-standard Compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60–140% for 85% of the compounds. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit	Inspect the system and re-analyze the blank. "B"-flag data for common contaminants.
Internal Standard (IS)	As each standard, blank, and sample is being loaded	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±50% of the area counts of the daily CCV internal standards.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, evaluate impact on data and narrate.
Surrogates	As each standard, blank, and sample is being loaded	70–130% Or in-house generated control limits may be used.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %Rs are out-of-limits a second time, report data from first analysis and narrate.
Laboratory Duplicates – Laboratory Control Spike Duplicates (LCSD)	One per analytical batch	RPD ≤25%	Narrate exceedances. If more than 5% of compound list is outside criteria or if compound has >40%RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

ANALYTICAL METHODS

Section 13.0

Method: EPA Method TO-14A/TO-15 Volatile Organic Compounds (Low-Level)

Eurofins Air Toxics SOP #83 Revision 19 Effective Date: April 30, 2019 Methods Manual Summary

Description: This method involves full scan gas chromatograph/mass spectrometer (GC/MS) analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds (VOCs) using EPA Method TO-14A/TO-15 protocols. An aliquot of up to 400 mL of air is withdrawn from the canister utilizing a mass flow controller. This volume is loaded onto a hydrophobic multibed sorbent trap to remove water and carbon dioxide and to concentrate the vapor sample. The focused sample is then flash-heated to sweep adsorbed VOCs onto a GC/MS for separation and detection. Compounds are detected using a mass spectrometer operating in full scan mode.

Eurofins Air Toxics maintains a suite of TO-14A/TO-15 methods, each optimized to efficiently meet the data objectives for a wide range of targeted concentration ranges. The methods, their reporting limits, and typical applications are summarized in the table below. This method summary describes TO-14A/TO-15 (Low-Level).

Eurofins Air Toxics Method	Base Reporting Limits	Typical Application
TO-14A/TO-15 (5&20)	5 – 20 ppbv	Soil gas and ppmv range vapor matrices
TO-14A/TO-15 (Standard or Quad)	0.5 – 5.0 ppbv	Ambient air, soil gas, and ppbv level vapor matrices
TO-15 (Extended)	0.2 – 5.0 ppbv	Ambient air and ppbv level vapor matrices
⇨ TO-14A/TO-15 (Low-level)	0.1 – 1.0 ppbv	Indoor and outdoor air
TO-14A/TO-15 (SIM)	0.01 – 0.5 ppbv	Indoor and outdoor air
TO-15 HSS	0.01 – 0.1 ppbv	Soil gas and other high concentration matrices

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, Eurofins Air Toxics reports these non-routine compounds with partial validation. Validation may include a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification analyzed, and no method detection limit study performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Since Eurofins Air Toxics applies TO-15 methodology to all Summa™ canisters regardless of whether TO-14A or TO-15 is specified by the project, Eurofins Air Toxics performs a modified version of method TO-14A as detailed in Table 1. Please note that Methods TO-14A and TO-15 were validated for specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of the method and is not recommended for ambient or indoor air samples. It is the responsibility of the data user to determine the usability of TO-14A and TO-15 results generated from Tedlar bags.

All samples submitted for TO-15 Low-Level are screened prior to analysis. If samples contain high concentrations of target and/or non-target VOCs, samples may be analyzed by an alternative TO-15 method (i.e., Standard or 5&20) with a higher dynamic calibration range.

Table 1. Summary of TO-14A Method Modifications

Requirement	TO-14A	Eurofins Air Toxics Modifications
Sample Drying System	Nafion Dryer	Multibed hydrophobic sorbent
Blank acceptance criteria	< 0.2 ppbv	< RL
BFB ion abundance criteria	Ion abundance criteria listed in Table 4 of TO-14A	Follow abundance criteria listed in TO-15.
BFB absolute abundance criteria	Within 10% when comparing to the previous daily BFB	CCV internal standard area counts are compared to ICAL; corrective action taken when recovery is less than 60%.
Blanks and standards	Zero Air	UHP Nitrogen provides a higher purity gas matrix than zero air for trace level measurements.
Initial Calibration	≤ 30% RSD for listed 39 VOCs	≤ 30% RSD with 4 compounds allowed out to ≤ 40%

Table 2. Summary of Method TO-15 Modifications

Requirement	TO-15	Eurofins Air Toxics Modifications
Initial Calibration	≤ 30% RSD with 2 compounds allowed out to < 40% RSD	≤ 30% RSD with 4 compounds allowed out to ≤ 40%
Blanks and standards	Zero Air	UHP Nitrogen provides a higher purity gas matrix than zero air for trace level measurements.

The standard target analyte list, reporting limits (RL), also referred to as Limit of Quantitation (LOQ), Quality Control (QC) criteria, and QC summary can be found in tables 3 through 6.

Table 3. Method TO-14A/TO-15 Standard Analyte List (Low-Level) and QC Limits

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS* (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,1,2-Trichloroethane	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethane	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethene	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,2,4-Trichlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2,4-Trimethylbenzene	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dibromoethane (EDB)	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichlorobenzene	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloroethane	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloropropane	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,3,5-Trimethylbenzene	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,3-Dichlorobenzene	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dichlorobenzene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Benzene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Bromomethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Carbon Tetrachloride	0.1	≤ 30%	70 – 130	70 – 130	± 25
Chlorobenzene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Chloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chloroform	0.1	≤ 30%	70 – 130	70 – 130	± 25
Chloromethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chlorotoluene (Benzyl Chloride)	0.1	≤ 30%	70 – 130	70 – 130	± 25
cis-1,2-Dichloroethene	0.1	≤ 30%	70 – 130	70 – 130	± 25
cis-1,3-Dichloropropene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Dichloromethane (Methylene Chloride)	0.2	≤ 30%	70 – 130	70 – 130	± 25
Ethylbenzene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Freon 11 (Trichlorofluoromethane)	0.1	≤ 30%	70 – 130	70 – 130	± 25
Freon 113 (Trichlorotrifluoroethane)	0.1	≤ 30%	70 – 130	70 – 130	± 25
Freon 114	0.1	≤ 30%	70 – 130	70 – 130	± 25
Freon 12 (Dichlorodifluoromethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Hexachlorobutadiene	0.5	≤ 30%	70 – 130	70 – 130	± 25
m,p-Xylene	0.1	≤ 30%	70 – 130	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS* (%R)	Precision Limits (Max. RPD)
Methyl Chloroform (1,1,1-Trichloroethane)	0.1	≤ 30%	70 – 130	70 – 130	± 25
o-Xylene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Styrene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Tetrachloroethene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Toluene	0.1	< 30%	70 – 130	70 – 130	± 25
trans-1,3-Dichloropropene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Trichloroethene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Vinyl Chloride	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,3-Butadiene	0.1	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dioxane	0.1	≤ 30%	70 – 130	70 – 130	± 25
2-Butanone (Methyl Ethyl Ketone)	0.5	≤ 30%	70 – 130	70 – 130	± 25
2-Hexanone	0.5	≤ 30%	70 – 130	70 – 130	± 25
4-Ethyltoluene	0.1	≤ 30%	70 – 130	70 – 130	± 25
4-Methyl-2-Pentanone (MIBK)	0.1	≤ 30%	70 – 130	70 – 130	± 25
Acetone	1.0	≤ 30%	70 – 130	70 – 130	± 25
Bromodichloromethane	0.1	≤ 30%	70 – 130	70 – 130	± 25
Bromoform	0.1	≤ 30%	70 – 130	70 – 130	± 25
Carbon Disulfide	0.5	≤ 30%	70 – 130	70 – 130	± 25
Cumene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Cyclohexane	0.1	≤ 30%	70 – 130	70 – 130	± 25
Dibromochloromethane	0.1	≤ 30%	70 – 130	70 – 130	± 25
Ethanol	0.5	≤ 30%	70 – 130	70 – 130	± 25
Heptane	0.1	≤ 30%	70 – 130	70 – 130	± 25
Hexane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Isopropanol	0.5	≤ 30%	70 – 130	70 – 130	± 25
Methyl tert-Butyl Ether (MTBE)	0.1	≤ 30%	70 – 130	70 – 130	± 25
Propylbenzene	0.1	≤ 30%	70 – 130	70 – 130	± 25
Tetrahydrofuran	0.5	≤ 30%	70 – 130	70 – 130	± 25
trans-1,2-Dichloroethene	0.1	≤ 30%	70 – 130	70 – 130	± 25
2,2,4-Trimethylpentane	0.5	≤ 30%	70 – 130	70 – 130	± 25
3-Chloroprene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Naphthalene**	0.5	≤ 40%	60 – 140	60 – 140	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS* (%R)	Precision Limits (Max. RPD)
TPH (Gasoline)***	10	1- Point Calibration	N/A	ICV only: 60 – 140	± 25
NMOC (Hexane/Heptane)***	2.0	1- Point Calibration	N/A	N/A	± 25

*See Table 6.

**Due to its low vapor pressure, Naphthalene does not meet TO-15 performance requirements. The wider QC limits reflect typical performance. Although Naphthalene is not on Eurofins Air Toxics "standard" TO-15 list, it is commonly requested and therefore included in Table 3.

***TPH and NMOC are not on Eurofins Air Toxics' standard TO-15 list, but are included in Table 3 due to common requests.

Table 3 is the list of Standard compounds, reporting limits and QC acceptance criteria. Each project may be customized as needed. Additional compounds and different reporting limits may be obtainable and/or achieved upon request

Table 4. Internal Standards

Analyte	Accuracy (% R)
Bromochloromethane	60 – 140
1,4-Difluorobenzene	60 – 140
Chlorobenzene-d_5	60 – 140

Table 5. Surrogates

Analyte	Accuracy (% R)
1,2-Dichloroethane-d_4	70 – 130
Toluene-d_8	70 – 130
4-Bromofluorobenzene	70 – 130

Table 6. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15 Low-Level

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours	TO-15 ion abundance criteria	Correct problem then repeat tune.
Minimum 5-Point Initial Calibration (ICAL)	Prior to sample analysis	% RSD ≤ 30 with 4 compounds allowed out to ≤ 40% RSD	Correct problem then repeat Initial Calibration curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each Initial Calibration curve, and daily prior to sample analysis	Recoveries for 85% of Standard compounds must be 70–130%. No recovery may be < 50%. ICV is evaluated on a full list basis at the time of calibration. If specified by the project, in-house generated control limits may be used.	Check the system and re-analyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) for Non-standard Compounds	Per client request or specific project requirements only	Recoveries of compounds must be 60–140%. No recovery may be <50%.	Check the system and re-analyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Continuing Calibration Verification (CCV) for Standard compounds	At the start of each analytical clock (24-hours) after the tune check	70–130%	Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than 4 compounds from the standard list recover outside of 70–130% or >10% of VOCs if short list is used (40 compounds or less), corrective action will be taken. If any compound exceeds 60–140%, samples are not analyzed unless data meets project needs. Check the system and re-analyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification (CCV) for Non-Standard compounds	Per client request or specific project requirements only	Recoveries of compounds must be 60–140%. No recovery may be <50%.	Check the system and re-analyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present	Results less than the laboratory reporting limit	Inspect the system and re-analyze the blank. "B"-flag data for common contaminants.
Internal Standard (IS)	As each standard, blank, and sample is being loaded	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ± 40% of the area counts of the daily CCV internal standards.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample unless obvious matrix interference is documented. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, report data from first analysis and narrate.
Surrogates	As each standard, blank, and sample is being loaded	70–130% R If specified by the project, in-house generated control limits may be used.	**For blanks:** Inspect the system and re-analyze the blank **For samples:** Re-analyze the sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %Rs are out-of-limits a second time, report data from first analysis and narrate.
Laboratory Duplicates - Laboratory Control Spike Duplicate (LCSD)	One per analytical batch	RPD ≤25%	Narrate exceedances. If more than 5% of compound list is outside criteria or if compound is >40% RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

ANALYTICAL METHODS

Section 14.0

Method: EPA Method TO-14A/TO-15 Volatile Organic Compounds by SIM

Eurofins Air Toxics SOP #38 Revision 24 Effective Date: May 1, 2019 Methods Manual Summary

Description: This method involves Selective Ion Monitoring (SIM) gas chromatograph/mass spectrometer (GC/MS) analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds (VOCs) using EPA Method TO-14A/TO-15 protocols. An aliquot of the sample is withdrawn from the canister through a mass flow controller and concentrated onto a hydrophobic drying system that removes water from the sample stream. The sample is then focused onto a cryogenic-cooled column prior to analysis by GC/MS in the SIM mode.

Mass spectrometer detectors can be set to acquire both SIM and full scan data simultaneously. This generates two separate data files in the analytical software. One file contains full scan data and the other contains SIM data for selected compounds. The results for each sample in a report will be from two separate data files originating from the same analytical run. The two data files have the same base file name and are differentiated with a "sim" extension on the SIM data file.

Eurofins Air Toxics maintains a suite of TO-14A/TO-15 methods, each optimized to efficiently meet the data objectives for a wide range of targeted concentration ranges. The methods, their reporting limits, and typical applications are summarized in the table below. This method summary describes TO-14A/TO-15 SIM.

Eurofins Air Toxics Method	Base Reporting Limits	Typical Application
TO-14A/TO-15 (5&20)	5 – 20 ppbv	Soil gas and ppmv range vapor matrices
TO-14A/TO-15 (Standard or Quad)	0.5 – 5.0 ppbv	Ambient air, soil gas, and ppbv level vapor matrices
TO-15 (Extended)	0.2 – 5.0 ppbv	Ambient air and ppbv level vapor matrices
TO-14A/TO-15 (Low-level)	0.1 – 1.0 ppbv	Indoor and outdoor air
TO-14A/TO-15 SIM	0.01 – 0.5 ppbv	Indoor and outdoor air
TO-15 HSS	0.01 – 0.1 ppbv	Soil gas and other high concentration matrices

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. If full validation of the required compound(s) is not available, the laboratory will present Quality Control (QC) options to the client based on the project objectives.

Please note that Methods TO-14A and TO-15 were validated for specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of the method and not recommended for ambient or indoor air samples. It is the responsibility of the data user to determine the usability of TO-14A and TO-15 results generated from Tedlar bags.

All samples submitted for TO-15 SIM are screened prior to analysis. If samples contain high concentrations of target and/or non-target VOCs, samples may be analyzed by an alternative TO-15 method (i.e. Standard or 5&20) with a higher dynamic calibration range.

Eurofins Air Toxics performs a modified version of TO-15 SIM as detailed in Table 1. Additionally, since Eurofins Air Toxics applies TO-15 methodology to all Summa™ canisters regardless of whether TO-14A or TO-15 is specified by the project, Eurofins Air Toxics performs a modified version of method TO-14A as described in Table 2. The default SIM target list, reporting limits (RL), QC criteria and QC summary may be found in tables 3 and 4.

Table 1. Summary of TO-15 SIM Method Modifications

Requirement	TO-15	Eurofins Air Toxics Modifications
Blank and standards	Zero Air	UHP Nitrogen provides a higher purity gas matrix than zero air for trace level measurements.

Table 2. Summary of TO-14A SIM Method Modifications

Requirement	TO-14A	Eurofins Air Toxics Modifications
Sample Drying System	Nafion Dryer	Multibed hydrophobic sorbent
ICAL %RSD acceptance criteria	≤ 30% RSD for listed 39 VOCs	Follow TO-15 requirements of ≤ 30%RSD with 2 of standard compound list allowed out to ≤ 40%RSD
Blank and standards	Zero air	UHP Nitrogen provides a higher purity gas matrix than zero air for trace level measurements.
BFB ion abundance criteria	Ion abundance criteria listed in Table 4 of TO-14A	Follow abundance criteria listed in TO-15.
BFB absolute abundance criteria	Within 10% when comparing to the previous daily BFB	CCV internal standard area counts are compared to ICAL; corrective action when recovery is less than 60%

Table 3. Method TO-14A/TO-15 Standard Analyte List (SIM) and QC Limits

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Dichlorodifluoromethane (Fr12)	0.020	≤ 30%	70 – 130	70 – 130	± 25
Freon 114	0.020	≤ 30%	70 – 130	70 – 130	± 25
Chloromethane	0. 50	≤ 30%	70 – 130	70 – 130	± 25
Vinyl Chloride	0.010	≤ 30%	70 – 130	70 – 130	± 25
Chloroethane	0.050	≤ 30%	70 – 130	70 – 130	± 25
Freon 11	0.02	≤ 30%	70 – 130	70 – 130	± 25
Freon 113	0.02	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethene	0.010	≤ 30%	70 – 130	70 – 130	± 25
Trans-1,2-Dichloroethene	0.100	≤ 30%	70 – 130	70 – 130	± 25
Methyl tert-Butyl Ether	0.100	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethane	0.020	≤ 30%	70 – 130	70 – 130	± 25
cis-1,2-Dichloroethene	0.020	≤ 30%	70 – 130	70 – 130	± 25
Chloroform	0.020	≤ 30%	70 – 130	70 – 130	± 25
1,1,1-Trichloroethane	0.020	≤ 30%	70 – 130	70 – 130	± 25
Carbon Tetrachloride	0.020	≤ 40%	60 - 140	60 - 140	± 25
Benzene	0.050	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloroethane	0.020	≤ 30%	70 – 130	70 – 130	± 25
Trichloroethene	0.020	≤ 30%	70 – 130	70 – 130	± 25
Toluene	0.050	≤ 30%	70 – 130	70 – 130	± 25
1,1,2-Trichloroethane	0.020	≤ 30%	70 – 130	70 – 130	± 25
Tetrachloroethene	0.020	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dibromoethane	0.020	≤ 30%	70 – 130	70 – 130	± 25
Ethyl Benzene	0.020	≤ 30%	70 – 130	70 – 130	± 25
m,p-Xylene	0.040	≤ 30%	70 – 130	70 – 130	± 25
o-Xylene	0.020	≤ 30%	70 – 130	70 – 130	± 25
1,1,2,2-Tetrachloroethane	0.020	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dichlorobenzene	0.020	≤ 30%	70 – 130	70 – 130	± 25
Naphthalene	0.050	≤ 40%	60 – 140	60 – 140	± 25

Table 3 is the list of Standard compounds, reporting limits and QC acceptance criteria. Each project may be customized as needed. Additional compounds and different reporting limits may be obtainable and/or achieved upon request.

Table 4. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15 by SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours	TO-15 Ion Abundance criteria	Correct problem then repeat tune.
Multi-point Calibration (Minimum of 5 points)	Prior to sample analysis	≤ 30% for standard compounds with 2 compounds allowed out to ≤ 40% RSD	Correct problem then repeat Initial Calibration Curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each initial calibration curve, and daily prior to sample analysis	Recoveries for 85% of standard compounds must be 70–130% 60–140% for Carbon Tetrachloride and Naphthalene). No recovery may be < 50%. ICV evaluated on a full list basis at the time of calibration. If specified by the project, in-house generated control limits may be used.	Check the system and re-analyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) for Non-Standard Compounds	Per client request or specific project requirements only	Recoveries of compounds must be 60–140%. No recovery may be < 50%.	Check the system and re-analyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Continuing Calibration Verification (CCV)	At the start of each day after the BFB tune check	70–130%D; 60-140% D for Carbon Tetrachloride and Naphthalene.	Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than two compounds from the standard list recover outside of 70–130%, corrective action will be taken. If any compound exceeds 60–140%, samples are not analyzed unless data meets project needs. Check the system and re-analyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification (CCV) for Non-Standard Compounds	Per client request or specific project requirements only	Recoveries of compounds must be 60–140%. No recovery may be < 50%.	Check the system and re-analyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit (Table 3) or project required reporting limit.	Inspect the system and re-analyze the blank. "B" flag data for common contaminants.
Internal Standard (IS)	As each standard, blank, and sample is being loaded	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±40% of the area counts of the daily CCV internal standards.	**For blanks:** Inspect the system and re-analyze the blank. **For samples:** Re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.
Surrogates	As each standard, blank, and sample is being loaded	70–130% If specified by the project, in-house generated control limits may be used.	**For blanks:** Inspect the system and re-analyze the blank. **For samples:** Re-analyze the sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %Rs are out-of-limits a second time, report data from first analysis and narrate.
Laboratory Duplicates - Laboratory Control Spike Duplicate (LCSD)	One per analytical batch	RPD ≤ 25%	Narrate exceedances. If more than 5% of compound list outside criteria or if compound is > 40%RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

ANALYTICAL METHODS

Section 15.0

Method: EPA Method TO-15 Volatile Organic Compounds High Selectivity/Sensitivity (HSS)

Eurofins Air Toxics SOP #133 Revision 2 Effective Date: December 13, 2018 Methods Manual Summary

Description: This method involves gas chromatograph/mass spectrometer (GC/MS) analysis of whole air samples collected in evacuated specially treated stainless steel canisters. Samples are analyzed for volatile organic compounds (VOCs) in compliance with EPA Method TO-15 QA/QC protocols. Using the TO-15 air interface, up to 0.05 liters of the vapor sample is concentrated on a multi-bed trap. After removal of air and water and addition of internal standards, the trap is heated and the VOCs are transferred to a customized GC for separation. This customized GC relies on a sequence of chromatographic separations and timed heart-cuts to remove matrix and isolate the target VOC for final detection by MS. The TO-15 HSS utilizes a Time-of-Flight MS for detection in order to generate full scan spectra with SIM-level sensitivity.

Eurofins Air Toxics maintains a suite of TO-14A/TO-15 methods, each optimized to efficiently meet the data objectives for a wide range of targeted concentration ranges and matrices. The methods, their reporting limits, and typical applications are summarized in the table below. This method summary describes TO-15 HSS which is designed for measuring select VOC(s) at trace levels in high concentration matrices.

Eurofins Air Toxics Method	Base Reporting Limits	Typical Application
TO-14A/TO-15 (5&20)	5 – 20 ppbv	Soil gas and ppmv range vapor matrices
TO-14A/TO-15 (Standard or Quad)	0.5 – 5.0 ppbv	Ambient air, soil gas, and ppbv level vapor matrices
TO-15 (Extended)	0.2 – 5.0 ppbv	Ambient air and ppbv level vapor matrices
TO-14A/TO-15 (Low-level)	0.1 – 1.0 ppbv	Indoor and outdoor air
TO-14A/TO-15 SIM	0.01 – 0.5 ppbv	Indoor and outdoor air
⇨ TO-15 HSS	0.01 – 0.1 ppbv	Soil gas and other high concentration matrices

Modifications to EPA Method TO-15 using this application are summarized in Table 1. As a note, TO-15 was published in 1999 when laboratory options were largely limited to linear quadrupole and ion trap MS detection since the TOF-MS technology had not yet developed as an economical or practical platform for commercial environmental laboratories. While TOF-MS is called out as a modification to TO-15 since only linear quadrupole and ion trap are described, the TOF-MS meets all spectral performance requirements outlined in the method and provides full scan data consistent with quadrupole MS systems and NIST reference spectral libraries.

Table 1. Summary of TO-15 HSS Method Modifications

Requirement	TO-15	Eurofins Air Toxics Modifications
Detector	Linear quadrupole or ion trap mass spectrometer	Time-of-Flight mass spectrometer (meets all method spectral performance requirements and generates full scan spectra)
Internal standards	Bromochloromethane, 1,4-Difluorobenzene, Chlorobenzene-d5	Deuterated analogues of the target compounds

The standard target analyte list, reporting limit (RL) also referred to as Limit of Quantitation, QC criteria, and QC summary can be found in Tables 2 through 4.

Table 2. Method TO-15 HSS Analyte List

Application	Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
			ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Application 1	1,2-Dichloroethane (EDC)	0.05	≤ 30%	70 – 130	70 – 130	± 25
	1,2-Dibromoethane (EDB)	0.01	≤ 30%	70 – 130	70 – 130	± 25
Application 2	1,4-Dioxane	0.05	≤ 30%	70 – 130	70 – 130	± 25
Application 3	Naphthalene	0.10	≤ 30%	70 – 130	70 – 130	± 25

Table 3. Internal Standards

Internal Standard (IS)	IS Conc (ppbv)	%D compared to CCV	Target Compound
1,2-Dichloroethane-d4	0.25	±40%	1,2-Dichloroethane
1,2-Dibromoethane-d4	0.25	±40%	1,2-Dibromoethane
1,4-Dioxane-d8	1.0	±40%	1,4-Dioxane
Naphthalene-d8	1.0	±40%	Naphthalene

Table 4. Summary of Calibration and QC Procedures for Method TO-15 HSS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours	TO-15 ion abundance criteria	Correct problem then repeat tune.
Minimum 5-Point Initial Calibration (ICAL)	Prior to sample analysis	≤ 30% RSD	Correct problem then repeat Initial Calibration curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each Initial Calibration curve, and daily prior to sample analysis	Recovery must be 70-130%	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Continuing Calibration Verification (CCV)	At the start of each analytical clock after the tune check	≤ 30% D	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit	Inspect the system and re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±40% of the area counts of the daily CCV internal standards.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.
Laboratory Duplicates – Laboratory Control Spike Duplicates (LCSD)	One per analytical batch	RPD ≤25%	Investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

ANALYTICAL METHODS

Section 16.0

Method: EPA Method TO-15 Analysis of Ethylene Oxide in Specially Treated Canisters by GC/MS Selective Ion Monitoring

Eurofins Air Toxics SOP #134 Revision 0 Effective Date: October 30, 2018 Methods Manual Summary

Description: This method involves the collection of ethylene oxide in ambient air using specially treated evacuated canisters. Up to 0.5 liters of air is withdrawn from the canister using a mass flow controller and concentrated on a series of traps designed to remove water from the sample stream. The sample is then focused onto a cryogenic-cooled column prior to analysis by GC/MS in the Selected Ion Monitoring (SIM) mode.

The mass spectrometer is set to acquire both SIM and full scan data simultaneously. This generates two separate data files in the analytical software. One file contains full scan data and the other contains SIM data for selected compounds. Ethylene oxide is quantified using the SIM file and the full scan data file is used if needed to assist to aid in confirmation and identification of potential interfering compounds.

The reporting limits and QC acceptance criteria are summarized in Table 1. The summary of calibration and QC procedures are summarized in Table 2.

Table 1. Reporting Limits and QC Acceptance Criteria

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Ethylene Oxide	0.050	≤30%	70 - 130	70 - 130	± 25

Table 2. Summary of Calibration and QC Procedures for Method TO-15 SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours.	TO-15 Ion Abundance criteria	Correct problem then repeat tune.
Multi-Point Calibration (minimum of 5 points)	Prior to sample analysis.	≤30% RSD.	Correct problem then repeat Initial Calibration Curve.
Initial Calibration Verification and Laboratory Control Sample (ICV and LCS)	After each initial calibration curve, and daily, prior to sample analysis.	70-130%	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Continuing Calibration Verification (CCV)	At the start of each day after the BFB Tune check.	≤30%D	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit Table 1.	Inspect the system and re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±40% of the area counts of the daily CCV internal standards.	**For blanks:** inspect the system and reanalyze the blank. **For samples:** re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.
Laboratory Duplicates - Laboratory Control sample Duplicate (LCSD)	One per analytical batch.	RPD ≤25% for sample concentrations greater than 5 times the reporting limit.	Investigate the cause including canister pressure and flow rates. Re-prepare standard if needed and re-analyze LCSD. If instrument maintenance is required, calibrate as needed.

ANALYTICAL METHODS

Section 17.0

Method: Modified EPA TO-17 VOCs and SVOCs – Tenax TA and Vapor Intrusion Applications by GC/MS (Full Scan)

Eurofins Air Toxics SOP #109 Revision 17 Effective Date: September 9, 2019 Methods Manual Summary

Description: This method is an alternative to the canister-based sampling and analysis methods that are presented in EPA Compendium Methods TO-14A and TO-15 as well as an alternative to PUF/XAD sampling for semivolatile compounds as described by EPA Compendium TO-13A. The Tenax TA tube is well-suited for compounds in the C5 to greater than C22 range and the multi-bed VI tube provides sufficient retention of light VOCs such as Vinyl Chloride while providing an efficient desorption of semi-volatile compounds up to 2-Methylnaphthalene.

Samples are collected by drawing a measured volume of air through the sorbent tubes. Collection is performed using a low flow vacuum pump or a volumetric syringe attached to the outlet side of the tube. Analysis is accomplished by heating the sorbent tube and sweeping the desorbed compounds onto a secondary "cold" trap for water management and analyte refocusing. The secondary trap is heated for efficient transfer of compounds onto the gas chromatograph (GC) for separation followed by detection using mass spectrometry (MS) in the full scan mode.

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, the laboratory reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage, safe sampling volume, and desorption efficiency are not validated. Full validation may be available upon request.

Since the TO-17 application significantly extends the scope of target compounds addressed in EPA Method TO-15 and TO-17, specifically for the multi-bed VI tube, the laboratory has implemented several method modifications outlined in Table 1.

Table 1. EPA TO-17 Method Modifications

Requirement	TO-17	Eurofins Air Toxics Modifications
Audit Accuracy	70-130%	Second source recovery 70-130%; for Fluoranthene and Pyrene = 60-140%.
Distributed Volume Pairs	Collection of distributed volume pairs required for monitoring ambient air to insure high quality.	If site is well-characterized or performance previously verified, single tube sampling may be appropriate. Distributed pairs may be impractical for soil gas collection due to configuration and volume constraints.
Analytical Precision	≤20%RPD	≤30% RPD for 3- and 4-ringed PAHs

The standard target analyte list, reporting limit (RL), QC criteria, and QC summary can be found in Tables 2 through 7.

Table 2. Method TO-17 VOCs (Tenax TA) Reporting Limits and QC Limits

Analytes	Reporting Limit (ng)	QC Acceptance Criteria		
		ICAL (%RSD)	ICV/LCS (% R)	CCV (%D)
Benzene	10	30	70 – 130	30
Toluene	5.0	30	70 – 130	30
Ethylbenzene	5.0	30	70 – 130	30
Naphthalene	5.0	30	70 – 130	30
m,p-Xylene	10	30	70 – 130	30
o-Xylene	5.0	30	70 – 130	30
2-Methylnaphthalene	5.0	30	70 – 130	30
1-Methylnaphthalene	5.0	30	70 – 130	30
Acenaphthylene	5.0	30	70 – 130	30
Acenaphthene	5.0	30	70 – 130	30
Fluorene	5.0	30	70 – 130	30
Phenanthrene	5.0	30	70 – 130	30
Anthracene	5.0	30	70 – 130	30
Fluoranthene	10	30	60 – 140	40
Pyrene	10	30	60 – 140	40
2-Propanol*	50	30	50 – 150	50

*2-Propanol is poorly retained on Tenax; Safe sampling volume of 0.2 L has been verified by the lab. Volumes greater than 0.2 L are considered screening values only. Compound is calibrated using a 3-point calibration with no MDL study.

Table 3. Method TO-17 VOCs (VI) Reporting Limits and QC Limits

Volatile Organic Compounds	Reporting Limit(ng)	Acceptance Criteria			
		ICAL (%RSD)	ICV (% R)	CCV (%D)	LCS (%R)
Freon 114	14	30	70 – 130	30	70 – 130
Vinyl Chloride	5.1	30	70 – 130	30	70 – 130
1,3-Butadiene	2.2	30	70 – 130	30	70 – 130
Isopentane	5.9	30	70 – 130	30	70 – 130
Freon 11	11	30	70 – 130	30	70 – 130
1,1-Dichloroethene	4.0	30	70 – 130	30	70 – 130
Methylene Chloride	21	30	70 – 130	30	70 – 130
Freon 113	7.7	30	70 – 130	30	70 – 130
Trans-1,2-Dichloroethene	4.0	30	70 – 130	30	70 – 130
1,1-Dichloroethane	4.0	30	70 – 130	30	70 – 130
Cis-1,2-Dichloroethene	4.0	30	70 – 130	30	70 – 130
Hexane	35	30	70 – 130	30	70 – 130
Chloroform	4.9	30	70 – 130	30	70 – 130
1,2-Dichloroethane	4.0	30	70 – 130	30	70 – 130
1,1,1-Trichloroethane	5.4	30	70 – 130	30	70 – 130
Benzene	6.4	30	70 – 130	30	70 – 130
Carbon Tetrachloride	6.3	30	70 – 130	30	70 – 130
Cyclohexane	6.9	30	70 – 130	30	70 – 130
1,2-Dichloropropane	4.6	30	70 – 130	30	70 – 130
Trichloroethene	5.4	30	70 – 130	30	70 – 130
1,4-Dioxane	11	30	70 – 130	30	70 – 130
2,2,4-Trimethylpentane	9.4	30	70 – 130	30	70 – 130
Heptane	12	30	70 – 130	30	70 – 130
Methylcyclohexane	8.0	30	70 – 130	30	70 – 130
1,1,2-Trichloroethane	5.4	30	70 – 130	30	70 – 130
Methyl isobutyl ketone	8.2	30	70 – 130	30	70 – 130
Toluene	7.5	30	70 – 130	30	70 – 130
Methylbutylketone	8.2	30	70 – 130	30	70 – 130
Tetrachloroethene	6.8	30	70 – 130	30	70 – 130
Chlorobenzene	4.6	30	70 – 130	30	70 – 130
Ethylbenzene	4.3	30	70 – 130	30	70 – 130
m,p-Xylene	8.7	30	70 – 130	30	70 – 130
o-Xylene	8.7	30	70 – 130	30	70 – 130
Styrene	8.5	30	70 – 130	30	70 – 130
Cumene	9.8	30	70 – 130	30	70 – 130
n-Propylbenzene	9.8	30	70 – 130	30	70 – 130
4-Ethyltoluene	9.8	30	70 – 130	30	70 – 130
1,3,5-Trimethylbenzene	9.8	30	70 – 130	30	70 – 130
1,2,4-Trimethylbenzene	29	30	70 – 130	30	70 – 130
1,3-Dichlorobenzene	6.0	30	70 – 130	30	70 – 130
1,4-Dichlorobenzene	6.0	30	70 – 130	30	70 – 130
1,2-Dichlorobenzene	6.0	30	70 – 130	30	70 – 130
1,2,4-Trichlorobenzene	15	30	70 – 130	30	70 – 130
Hexachlorobutadiene	21	30	70 – 130	30	70 – 130

Volatile Organic Compounds	Reporting Limit(ng)	Acceptance Criteria			
		ICAL (%RSD)	ICV (% R)	CCV (%D)	LCS (%R)
Chloroethane†	16	30	70 – 130	30	70 – 130
Isopropyl alcohol†	49	30	70 – 130	30	70 – 130
Carbon Disulfide†	6.2	30	70 – 130	30	70 – 130
MTBE†‡	22	30	70 – 130	30	70 – 130
Methyl Ethyl Ketone†	59	30	70 – 130	30	70 – 130
1,1,2,2-Tetrachloroethane*	6.9	40	60 – 140	40	60 – 140
Polyaromatic Hydrocarbons	**Reporting Limit(ng)**	**Acceptance Criteria**			
		ICAL (%RSD)	ICV (% R)	CCV (%D)	LCS (%R)
Naphthalene	1.0	30	70 – 130	30	70 – 130
2-Methylnaphthalene	1.0	30	70 – 130	30	70 – 130
1-Methylnaphthalene	1.0	30	70 – 130	30	70 – 130

†Non-routine compounds by special request only.
‡Poor recovery performance when dry purge is applied for sample collection volumes greater than 1-L.
* Compound by special request. Erratic recoveries on the VI tube sorbent. Literature confirms lab observations.

Table 4. Commonly requested TPH parameters (Tenax TA and VI)

TPH	Reporting Limit (ng)	ICAL (%RSD)	ICV (% R)	CCV (%D)	LCS (%R)
GRO (Gasoline Range)	1000	30	60 – 140	40	60 – 140
DRO (C10-C22 Diesel Range)	1000	30	60 – 140	40	60 – 140

Table 5. Internal Standard (Tenax TA and VI)

Analyte	CCV IS % Recovery	Sample IS % Recovery
Bromochloromethane*	>60	60 – 140
1,4-Difluorobenzene	>60	60 – 140
Chlorobenzene-d5	>60	60 – 140
Bromofluorobenzene	>60	60 – 140

*BCM may not be required for Tenax TA list based on the requested VOC list.

Table 6. Field Surrogate Recoveries (Tenax TA and VI)

Analyte	% Recovery
1,2-Dichloroethane-d4	50 – 150
Toluene-d8	50 – 150
Naphthalene-d8	50 – 150

Table 7. Summary of Calibration and QC Procedures for TO-17 General Application

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
BFB Tune Check	Before initial and daily calibration. Check is valid for 24 hours.	TO-15 tune criteria	Correct problem then repeat tune.
5-Point Calibration	Prior to sample analysis	%RSD ≤ 30% with 2 compounds exceeding up to 40%RSD	Correct problem then repeat Initial Calibration Curve.
Initial Calibration Verification (ICV)	After each initial Calibration Curve	See tables 2 and 3; 20% of the compounds are allowed to exceed criterion.	Determine if the exceedance is due to an inaccurate calibration standard or inaccurate ICV standard. Recalibrate with an accurate standard or re-prepare the ICV as necessary. If any VOC exceeds 50-150% recovery, system is checked and the ICV is reanalyzed. For compounds with recoveries greater than 150% and no positive detections in the samples, approval to proceed will be granted on a case-by-case basis.
Continuing Calibration Verification (CCV)	At the start of each 24-hour clock after the Tune Check	70 – 130 %; 60-140% for Fluoranthene and Pyrene (see table 3)	If project-specified risk drivers exceed these criteria, more than 5% of the compounds exceed these criteria, or any VOC exceeds 50–150% recovery, maintenance is performed and the CCV test repeated. If the system still fails the CCV, perform a new 5-point Calibration Curve.
Laboratory Blank	After the CCV, before samples and at the end of the sequence	Results less than the laboratory RL.	Inspect the system and re-analyze the Blank. Flag associated data as appropriate.
Laboratory Control Spike (LCS)	Each analytical batch	Recovery 70 – 130%; 60-140% Fluoranthene and Pyrene; Or as noted in table 3; 20% of the compounds may exceed criteria before corrective action is required.	Verify accuracy of standard. Re-prepare LCS if necessary. If calibration curve and/or system is found to be out of control, perform maintenance and re-calibrate. If any VOC exceeds 50-150% recovery, maintenance is performed and the LCS test is repeated. For compounds with recoveries greater than 150% and no positive detections in the samples, approval to proceed will be granted on a case by case basis.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Spike Duplicate (LCSD)	Once per analytical batch –(reanalysis of LCS)	≤20%RPD for all compounds with exception of Fluorene, Phenanthrene, Anthracene, Fluoranthene, Pyrene,TPH and 2-Propanol (Tenax only) which must be ≤30%RPD	Verify accuracy of standard. Re-prepare LCSD if necessary. If calibration curve and/or system is found to be out of control, perform maintenance and re-calibrate. If any VOC exceeds 50-150% recovery, maintenance is performed and the LCSD test is repeated. For compounds with recoveries greater than 150% and no positive detections in the samples, approval to proceed will be granted on a case by case basis.
Internal Standard (IS)	As each QC sample and sample are being loaded	**CCVs:** area counts >60% recovery, RT w/in 20 sec of mid-point in ICAL **Blanks and samples**: Retention time (RT) must be within ±0.33 minutes of the RT in the CCV. The IS area must be within ±40% of the CCV's IS area for the Blanks and samples.	**CCV:** Inspect and correct system prior to sample analysis. **Blanks:** Inspect the system and re-analyze the Blank. **Samples:** Investigate the problem by verifying the instrument is in control by running a Lab Blank. Re-analyze recollected samples to verify recovery. Report the run with acceptable IS recovery. If both runs are unacceptable, narrate and flag associated data.
Field Surrogates	Added to each tube prior to shipment to field. Added to QC samples prior to analysis	50 – 150%	**Blanks:** Inspect the system and re-analyze the Blank. **Samples:** Review data to determine whether sample collection parameters or matrix interference resulted in the exceedances. If so, narrate and flag recovery. If no cause is evident, verify the instrument is in control by running a Lab Blank. Re-analyze recollected sample to verify recovery. If sample matrix is causing a systematic change in internal standard response with each successive run, an End Check spiked at the CCV concentration can help to assess the impact on target compound accuracy.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Field Blank	Project dependent	Artifact levels should be less than the reporting limit or less than 10% of the mass measured on the sampled tubes, whichever is less	Flag associated results and evaluate tube conditioning and storage procedures.
Distributed Pairs	Project dependent	%RPD $\leq$ 25%	Narrate discrepancy

ANALYTICAL METHODS

Section 18.0

Method: ANALYSIS OF VOCS BY GC/MS COLLECTED ON CHARCOAL-BASED PASSIVE SAMPLERS USING MODIFIED EPA TO-17

Eurofins Air Toxics SOP #100 Revision 14 Effective Date: May 22, 2019 Methods Manual Summary

Description: This method involves gas chromatograph/mass spectrometer (GC/MS) analysis of volatile organic compounds (VOCs) collected using charcoal-based passive samplers. These passive samplers include the Radiello® 130, SKC badges (575 and Ultra), and the Waterloo Membrane Samplers (WMS™). Passive samplers are used to measure vapor-phase VOCs in a variety of gaseous matrices including indoor air, outdoor air, and soil gas. VOCs in the sampling environment pass through the diffusive barrier or permeable membrane of the sampler at a known, controlled rate (defined as the sampling rate) and adsorb to the charcoal-based sorbent pad of the sampler.

The sorbent is extracted using a volume of carbon disulfide, and the extract is directly injected into a GC equipped with an MS. The retention time and spectral pattern of a compound are compared with that of known standard. Concentrations of the analytes are calculated from the average relative response factors of calibration curves obtained from analysis of standard solutions. The results are reported in units of μg/sample or $\mu g/m^3$ if the sampling rate and duration is known. To minimize a low bias in the subsurface soil gas $\mu g/m^3$ concentration due to starvation effects, the WMS-Low Uptake version (WMS-LU) is recommended. Starvation effects occur when the uptake rate of the sampler exceeds the delivery rate of vapors from the surrounding soil.

There are currently no EPA methods for the preparation and analysis of charcoal-based passive samplers for environmental monitoring of VOCs in air. The reference method used for this procedure is EPA TO-17, which describes the collection of VOCs in ambient air using sorbents and analysis by GC/MS. Because TO-17 describes active sample collection using a pump and thermal desorption as the preparation step, several modifications are required. Specifically, the extraction steps using carbon disulfide and internal standard addition are based on the recommended procedures published by Radiello (FSM). The modifications taken to EPA Method TO-17 are outlined in Table 1.

Table 1. Summary of Method TO-17 Modifications

Requirement	TO-17	EATL Modifications
Sample collection	Pump pulls measured air volume through sorbent tube	VOCs in air adsorbed onto sorbent bed passively through diffusion
Sample preparation	Thermal extraction	Solvent extraction
Sorbent tube conditioning	Condition newly packed tubes prior to use	Charcoal-based sorbent is a single use media and conditioning is conducted by vendor

Requirement	TO-17	EATL Modifications
Instrumentation	Thermal desorption system	Liquid injection system
Internal Standard	Gas-phase internal standard introduced on the tube or focusing trap during analysis	Liquid-phase internal standard introduced on the tube at the time of extraction
Media and sample storage	<4 deg C, 30 days	Media shelf life is determined by vendor; sample hold-time is 6 months for the RAD130 and WMS. Sample preservation requirements are storage in a cool, solvent-free refrigerator and optional use of ice during shipping
Internal Standard Recovery	+/-40% of daily CCV area	-50% to +100% of daily CCV area

Tables 2 through 4 list the target analytes routinely calibrated, along with the reporting limits and QC acceptance criteria. Tables 5 through 8 list the reporting limit for each sampler type in units of mass and the sampling rate. The sampling rates for the WMS sampler are maintained as proprietary and are not published as part of this document. To calculate the sample reporting limit in terms of $\mu g/m^3$, the compound sampling rate and the sample duration are required. Please consult with the laboratory to determine the appropriate sampler to meet project objectives.

Table 2. Target Analytes Reporting Limits and QC Criteria

Analytes	Reporting Limit (µg/mL)	Acceptance Criteria			
		ICAL (%RSD)	ICV (% R)	LCS (%R)	CCV (%D)
Chloromethane	0.2	30	70 – 130	50 – 140	≤ 40%
Vinyl Chloride	0.2	30	50 – 140	50 – 140	≤ 40%
Ethanol	0.5	30	70 – 130	50 – 130*	≤ 30%
1,1-Dichloroethene	0.2	30	70 – 130	70 – 130	≤ 30%
MTBE	0.05	30	70 – 130	70 – 130	≤ 30%
trans-1,2-Dichloroethene	0.1	30	70 – 130	70 – 130	≤ 30%
Hexane	0.05/0.20***	30	70 – 130	70 – 130	≤ 30%
1,1-Dichloroethane	0.05	20	80 – 120	70 – 130	≤ 20%
Ethyl Acetate	0.2	30	70 – 130	70 – 130	≤ 30%
2-Butanone	0.05/0.10***	30	70 – 130	70 – 130	≤ 30%
cis-1,2-Dichloroethene	0.05	20	80 – 120	70 – 130	≤ 20%
Chloroform	0.05	20	80 – 120	70 – 130	≤ 20%
Cyclohexane	0.05	30	70 – 130	70 – 130	≤ 30%
1,1,1-trichloroethane	0.05	20	80 – 120	70 – 130	≤ 20%
Carbon Tetrachloride	0.05	20	80 – 120	70 – 130	≤ 20%
Benzene	0.2	30	70 – 130	70 – 130	≤ 30%
1,2-Dichloroethane	0.05	20	80 – 120	70 – 130	≤ 20%
Heptane	0.05	20	80 – 120	70 – 130	≤ 20%
Trichloroethene	0.05	20	80 – 120	70 – 130	≤ 20%
4-Methyl-2-pentanone	0.1	30	70 – 130	70 – 130	≤ 30%
Toluene	0.05	20	80 – 120	70 – 130	≤ 20%

Analytes	Reporting Limit (µg/mL)	Acceptance Criteria			
		ICAL (%RSD)	ICV (% R)	LCS (%R)	CCV (%D)
1,1,2-Trichloroethane	0.05	20	80 – 120	70 – 130	≤ 20%
Tetrachloroethene	0.05	20	80 – 120	70 – 130	≤ 20%
Chlorobenzene	0.05	20	80 – 120	70 – 130	≤ 20%
Ethylbenzene	0.05	20	80 – 120	70 – 130	≤ 20%
m,p-Xylene	0.05	20	80 – 120	70 – 130	≤ 20%
o-Xylene	0.05	30	70 – 130	70 – 130	≤ 30%
Styrene	0.05	30	70 – 130	20-100*	≤ 30%
1,1,2,2-Tetrachloroethane	0.05	30	70 – 130	60 – 130	≤ 30%
Propylbenzene	0.05	20	80 – 120	70 – 130	≤ 20%
1,3,5-Trimethylbenzene	0.05	20	80 – 120	70 – 130	≤ 20%
1,2,4-Trimethylbenzene	0.05	20	80 – 120	70 – 130	≤ 20%
1,3-Dichlorobenzene	0.05	30	70 – 130	50 – 110**	≤ 30%
1,4-Dichlorobenzene	0.05	30	70 – 130	50 – 110**	≤ 30%
1,2-Dichlorobenzene	0.05	30	70 – 130	50 – 110**	≤ 30%
Naphthalene	0.05	30	70 – 130	5-80*	≤ 30%

*Acceptance limits based on desorption efficiency studies
**60 – 130% for WMS, RL for WMS

Table 3. Internal Standard

Analyte	CCV IS (%R)	Sample IS (%)R
2-Fluorotoluene	-50 to +200	-50 to +200

Table 4. Surrogate

Analyte	%R
Toluene-d8	70-130

Table 5. Reporting Limits and Sampling Rates for "Standard" target compounds (RAD 130)

Analytes	Reporting Limit (µg/sampler)	Sampling Rates for Radiello 130 Sampler
Vinyl Chloride**	0.4	90*
Ethanol	1.0	102
1,1-Dichloroethene	0.4	76*
MTBE	0.1	65
trans-1,2-Dichloroethene	0.2	60*
Hexane	0.1	66
1,1-Dichloroethane	0.1	63*
Ethyl Acetate	0.4	78
2-Butanone	0.1	79
cis-1,2-Dichloroethene	0.1	62*
Chloroform	0.1	75
Cyclohexane	0.1	54
1,1,1-trichloroethane	0.1	62
Carbon Tetrachloride	0.1	67
Benzene	0.4	80

Analytes	Reporting Limit (µg/sampler)	Sampling Rates for Radiello 130 Sampler
1,2-Dichloroethane	0.1	77
Heptane	0.1	58
Trichloroethene	0.1	69
4-Methyl-2-pentanone	0.2	67
Toluene	0.1	74
1,1,2-Trichloroethane	0.1	66*
Tetrachloroethene	0.1	59
Chlorobenzene	0.1	68
Ethylbenzene	0.1	68
m,p-Xylene	0.1	70
o-Xylene	0.1	65
Styrene	0.1	61
1,1,2,2-Tetrachloroethane	0.1	60*
Propylbenzene	0.1	57
1,3,5-Trimethylbenzene	0.1	53*
1,2,4-Trimethylbenzene	0.1	50
1,3-Dichlorobenzene	0.1	59*
1,4-Dichlorobenzene	0.1	51
1,2-Dichlorobenzene	0.1	58*
Naphthalene	0.1	25

*Estimated using the diffusion coefficient in air and the geometric constant 14.145 cm ('white' diffusive body, code 120).

**Vinyl chloride is included in the calibration standard; however, it is not a "standard" compound as it is not recommended on the RAD130 cartridge due to poor retention. Applications using the RAD130 for VC measurements for extended durations (>8 hours) will result in significant low bias using the theoretical uptake rate. All lab reports with vinyl chloride reported must have the appropriate narration regarding poor retention and potential low bias.

Table 6. Reporting Limits and Sampling Rates for "Standard" target compounds (SKC 575/Ultra)

Analytes	Reporting Limit (µg/sampler)	Sampling Rates for Indoor Air Applications 'Zero Face velocity'	Sampling Rates for Outdoor/worker exposure (ml/min)
Vinyl Chloride	0.4	17.4*	21.2*
Ethanol	1.0	11.7	20.0
1,1-Dichloroethene	0.4	9.74	12.3
MTBE	0.1	9.84	13.6
trans-1,2-Dichloroethene	0.2	10.2	14.8
1,1-Dichloroethane	0.1	13.14	12.3
Ethyl Acetate	0.4	9.26	13.75
2-Butanone	0.1	6.27	17.1
cis-1,2-Dichloroethene	0.1	11.54*	14.8*
Chloroform	0.1	10.14	13
Cyclohexane	0.1	7.76	15.6
1,1,1-trichloroethane	0.1	9.40	14.1
Carbon Tetrachloride	0.1	10.41	14.1
Benzene	0.4	10.69	16
1,2-Dichloroethane	0.1	11.79	14.2
Heptane	0.1	9.38	13.9
Trichloroethene	0.1	11.47	14.9
4-Methyl-2-pentanone	0.2	7.29	13.5
Toluene	0.1	8.90	14.5
1,1,2-Trichloroethane	0.1	9.64	12.5
Tetrachloroethene	0.1	10.02	13.1
Chlorobenzene	0.1	8.23*	18.74*
Ethylbenzene	0.1	9.02	12.9
m,p-Xylene	0.1	8.1	12.65
o-Xylene	0.1	8.11	11.9
Styrene	0.1	9.04	13.7
1,1,2,2-Tetrachloroethane	0.1	9.98	11.8
Propylbenzene	0.1	6.41*	11.69*
1,3,5-Trimethylbenzene	0.1	7.29*	12.1*
1,2,4-Trimethylbenzene	0.1	9.92*	12.1*
1,3-Dichlorobenzene	0.1	5.79*	12.7*
1,4-Dichlorobenzene	0.1	10.74*	12.7*
1,2-Dichlorobenzene	0.1	4.97*	12.6*
Naphthalene	0.1	2.71*	13.7*

*Calculated by SKC: (Concentrations reported using a calculated rate will be qualified with a C-flag to indicate an estimated value. Compounds which are poorly retained on the sorbent over the planned duration will be biased low).

Table 7. Summary of Calibration and QC Procedures

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at the start of every 12-hour clock	Method TO-17 tuning criteria	Correct problem then repeat tune.
Initial 5-Point Calibration (ICAL)	Prior to sample analysis	Compound criteria in Table 2	Correct problem then repeat initial calibration. Analysis may proceed if no more than 2 VOCs exceed criteria or 5% of VOCs if short list is used. Narrate exceedances.
Initial Calibration Verification (ICV)	Once per initial calibration	See Table 2	Verify concentrations and standard preparation. Analysis may proceed if no more than 2 VOCs exceed criteria or 5% of VOCs if short list is used. Narrate exceedances.
Continuing Calibration Verification (CCV)	At the start of every shift immediately after the BFB tune check	See "CCV criteria" column in Table 2	Investigate and correct the problem, up to and including recalibration if necessary. Analysis may proceed if no more than 2 VOCs exceed criteria or 5% of VOCs if short list is used. Associated results are flagged.
Internal Standards (IS)	IS is added at the time of extraction to all samples and QC samples.	**For CCVs:** Area counts 50 –200%; RT w/in 30 seconds of midpoint in ICAL **For blanks, samples and non-CCV QC checks:** Area counts 50 – 200%; RT within 20 seconds of RT in CCV	**CCV:** Inspect and correct system prior to sample analysis. **For blanks:** Inspect the system and re-analyze the blank. **For samples:** Re-analyze; if out again, flag data.
Surrogate	Surrogate is added at the time of extraction to all samples and QC samples.	70–130%	Same as for Internal Standards.
Solvent Blanks	Immediately after the calibration standard or after samples with high concentrations	Results less than laboratory reporting limit (see Table 2)	Re-aliquot and re-analyze solvent blank. If detections remain, flag concentrations in associated samples.
Extracted Laboratory Blank	Each set of up to 20 samples	Results less than the reporting limit	Flag sample concentrations in associated extraction batch.
Extracted Laboratory Control Spike (LCS)	Each set of up to 20 samples	See Table 2.	Analysis may proceed if no more than 2 VOCs exceed criteria (or 5% for short list exceed criteria). Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise, narrate.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Extracted Laboratory Control Spike Duplicate (LCSD)	Each set of up to 20 samples	%RPD ≤ 25%	Analysis may proceed if no more than 2 VOCs exceed criteria (or 5% for short list exceed criteria). Narrate as appropriate.

ANALYTICAL METHODS

Section 19.0

Method: Modified EPA TO-17 Volatile Organic Compounds (Passive Sample Collection)

Eurofins Air Toxics SOP #112 Revision 11 Effective Date: May 7, 2019 Methods Manual Summary

Description: This method involves gas chromatograph/mass spectrometer (GC/MS) analysis of volatile organic compounds (VOCs) collected using the Radiello passive sampler. The Radiello sample is paired with thermally desorbable sorbent. This sampler is used to measure vapor-phase VOCs in a variety of gaseous matrices including indoor air, outdoor air, and soil gas. The VOCs in the sampling environment pass through a diffusive barrier at a controlled rate and adsorb to the sorbent bed of the sampler.

The Radiello sampler consists of a sorbent cartridge and a diffusive body. The diffusive body is cylindrical and is designed for the cartridge to slide in its center.

The sorbent is transferred to an empty tube, if needed, and the tubes are thermally desorbed by heating and purging with UHP Helium. The resulting gaseous effluent is transferred to secondary trap for re-concentration and desorption onto the gas chromatograph equipped with a mass spectrometer. The retention time (RT) and spectral pattern of a compound are compared with that of a known standard. Concentrations of the analytes are calculated from the average relative response factors of calibration curves obtained from analysis of standard solutions. Results are reported in ng/sample or ug/m3 if the sampling rate and duration are known. Sampling rates can be estimated to provide semi-quantitative concentration results. Concentrations derived from estimated rates are flagged as estimated values.

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, the laboratory reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the Reporting Limit (RL), no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage as well as desorption efficiency are not validated. Full validation may be available upon request.

The analysis is performed using the analytical protocols of EPA Method TO-17. The significant deviation from the TO-17 method is that the samples are collected using passive samplers as opposed to the method-defined procedure of using a pump to actively pull vapors through the sorbent.

Table 1. Summary of Method TO-17 Modifications

Requirement	TO-17	Eurofins Air Toxics Modifications
Sample collection	Active	Passive

The standard target analyte lists, Reporting Limits (RLs), Limit of Quantitation (LOQ), and Quality Control (QC) criteria are presented in tables 2 through 4. Table 5 summarizes the calibration and QC procedures.

Table 2. Carbograph 4 (Radiello 145 cartridge) Analyte List and QC Limits

Sampling parameter	Recommended			
Exposure period	<3 days with 1 day ideal if the C2 chlorinated VOCs are included; Vendor cites 7 to 14 days; however, high sampling rate means high mass loading with extended period. Less retained VOCs can be displaced resulting in a low bias.			
Sample matrix	**<**2000 ug/m3 total VOCs			
Analytes	**Reporting Limit (ng)**	**Acceptance Criteria**		
		ICAL (%RSD)	**ICV/LCS (% R)**	**CCV (%R)**
1,1,1-Trichloroethane	10	≤40	60 – 140	70 – 130
Benzene	20	≤30	70 – 130	70 – 130
Ethyl Benzene	10	≤30	70 – 130	70 – 130
m,p-Xylene	20	≤30	70 – 130	70 – 130
o-Xylene	10	≤30	70 – 130	70 – 130
Tetrachloroethene	5.0	≤30	70 – 130	70 – 130
Toluene	50	≤30	70 – 130	70 – 130
Trichloroethene	5.0	≤30	70 – 130	70 – 130
Cyclohexane	10	≤30	70 – 130	70 – 130
Styrene	10	≤30	70 – 130	70 – 130
1,1-Dichloroethene	5.0	≤30	70 – 130	70 – 130
Freon 113	5.0	≤30	70 – 130	70 – 130
trans-1,2-Dichloroethene	5.0	≤30	70 – 130	70 – 130
1,1-Dichloroethane	5.0	≤30	70 – 130	70 – 130
cis-1,2-Dichloroethene	5.0	≤30	70 – 130	70 – 130
Chloroform	5.0	≤30	70 – 130	70 – 130
1,2-Dichloroethane	5.0	≤30	70 – 130	70 – 130
1,1,2-Trichloroethane	5.0	≤30	70 – 130	70 – 130

Compounds in **bold** indicate that the associated Sampling Rate is calculated. A "C" flag will be applied to these results, as they should be considered as estimated.

Table 3. Internal Standards for Carbograph 4 (Radiello 145 cartridge)

Analyte	CCV IS % Recovery	Sample IS % Recovery
Bromochloromethane	≥60	60 – 140
1,4-Difluorobenzene	≥60	60 – 140
Chlorobenzene-d_5	≥60	60 – 140

Table 4. Analytical Surrogate for Carbograph 4 (Radiello 145 cartridge)

Analyte	% Recovery
4-Bromofluorobenzene	70 – 130

Table 5. Summary of Calibration and QC Procedures for EPA Method TO-17-Passive Sorbent Sampling

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
BFB Tune Check	Every 24 hours	TO-15/TO-17 tune criteria.	Correct problem then repeat tune check.
5-Point Calibration	Prior to sample Analysis.	%RSD ≤ 30%, 2 allowed out up to 40%	Correct problem then repeat Initial Calibration Curve.
LCS/ICV	After each initial Calibration Curve and daily prior to analysis.	Recovery 70-130% or as indicated in Table 2, 20% of compound list may exceed criterion before corrective action is required. Also, if any VOC exceeds 50-150%, corrective action is required. For compounds with recoveries greater than 150% and no positive detections in the samples, approval to proceed will be granted on a case-by-case basis by QA or management.	Verify accuracy of standard. Re-prepare LCS if necessary. If calibration curve and/or system is found to be out of control, perform maintenance and re-calibrate.
LCSD	Each analytical batch – reanalysis of LCS	See LCS recovery acceptance criterion; %RPD ≤ 20%	Evaluate whether the precision outlier is due to recollection failure of the TDU. If so, correct system and re-start analytical sequence with the BFB.
Continuing Calibration Verification (CCV)	At the start of each analytical clock	70 – 130 %	Two compounds are allowed to exceed criterion up to ±40%D prior to initiation of corrective action. If more than 2 VOCs exceed the ±30% D criterion or > 10% of VOCs if short list is used (20 compounds or less), the CCV tube is re-spiked and the test repeated. If the system still fails the CCV, the system is evaluated. As necessary, a new initial calibration curve is analyzed. CCV recoveries >140% may be approved by QA or management after evaluation of project objectives and risk drivers.
Laboratory Blank	After the CCV and at the end of the analytical batch.	Results less than the laboratory RL.	Inspect the system and re-analyze the Blank. No corrective action for Lab Blank at end of batch.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Internal Standard (IS)	As each standard, Blank, and sample is being loaded.	**CCVs:** area counts ≥60% and RT w/in 20 sec of mid-point in ICAL. **Blanks and samples**: Retention time (RT) must be within ±0.33 minutes of the RT in the CCV. The IS area must be within ±40% of the CCV's IS area for the Blanks and samples.	**CCV:** inspect and correct system prior to sample analysis. **Blanks:** inspect the system and re-analyze the Blank. **Samples:** Analyze re-collected samples to confirm internal standard recoveries. If recovery is out of acceptance criteria in the initial and recollected sample, the initial sample is reported and associated data is qualified if appropriate. If sample matrix is causing a systematic change in internal standard response with each successive run, an End Check spiked at the CCV concentration can help to assess the impact on target compound accuracy.
Analytical Surrogate	Each passive sampler and Lab Blank and QC samples during sample desorption	70-130%	**For blanks:** inspect the system and re-analyze the Blank. **For samples:** If no obvious reason can be ascertained after evaluation of the data, the sample should be reanalyzed to verify out of control recovery. If recovery is out of acceptance criteria in both the initial and recollected sample, the initial sample is reported with the surrogate flagged.

eurofins | Air Toxics

LABORATORY QUALITY ASSURANCE MANUAL

(LQAM)

Appendix F

References

(Two total pages including this cover)

Current as of September 9, 2019

REFERENCES

- Air Monitoring Quality Assurance Manual, Volume VI: Standard Operating Procedures for Stationary Sources Emission Monitoring and Testing. State of California Air Resources Board. Available at: http://www.arb.ca.gov//aaqm/qa/qa-manual/qa-manual.htm
- Annual Book of ASTM Standards, ASTM International. Available at: www.astm.org
- Compendium of Methods for the Determination of Air Pollutants in Indoor Air. Winberry et al. Atmospheric Research and Exposure Assessment Laboratory, Office of Research and Development, U.S. Environmental Protection Agency May 1990.
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air. U.S. EPA Center for Environmental Research Information. Second Edition. January 1999.
- Environmental Sampling and Analysis: Lab Manual. Maria Csuros. Lewis Publishers, New York. 1997.
- Guidelines Establishing Test Procedures for the Analysis of Pollutants. U.S. EPA Clean Water Act. 40 CFR 136. 2013.
- Quality Systems Manual (QSM) for Environmental Laboratories. Department of Defense (DoD). Version 5.3 2019.
- Statement of Work for Organics Analysis. U.S. EPA Contract Laboratory Program. OLM04.2. May 1999.
- TNI Standard. Volume 1: Management and Technical Requirements for Laboratories Performing Environmental Analysis. The NELAC Institute Environmental Laboratory Sector. 2016. Available at: http://www.nelac-institute.org/docs/standards/2016/EL_Volume1_2016.pdf
- International Standard ISO/IEC 17025:2017 (E), Third Edition 2017-11

Appendix D
Laboratory Standard Operating Procedures

EMAX
LABORATORIES, INC.
1835 W. 205th Street
Torrance, CA 90501
Tel: (310) 618-8889

Page 1 of 1

ADDENDUM TO	
Document	ALL ANALYTICAL SOPS
Revision Number	CURRENT REVISIONS
Section	10.2 Instrument Parameters
Date	11 September 2019
Reference Number	AA.7

This applies to all GC/MS, GC, HPLC-MS, HPLC, ICP-MS, ICP and IC Method SOPs

Section 10.2 shall include:

10.2. **Instrument Parameters**

10.2.1. Instrument parameters setup stipulated in the SOPs, are instrument suggested parameters. Fine tune the instrument to obtain optimum instrument condition.

10.2.2. Print and staple a copy of the current instrument parameter on the instrument log for easy access when performing daily instrument routine check.

10.2.3. In the event that instrument parameters necessitate a change, replace the instrument parameter printout with the new parameter setup. Archive the previous instrument parameters at the back of the instrument maintenance log.

PREPARED BY: _____ Date: 9/11/19

Name Farina Madamba

Title QA / QC Coordinator

APPROVED BY: _____ Date: 09.11.19

Name Kenette Pimentel

Title QA Manager

APPROVED BY: _____ Date: 09-11-19

Name Caspar Pang

Title Laboratory Director

STANDARD OPERATING PROCEDURES

TRACE METALS BY ICP-MS

SOP No.:	EMAX-6020	Revision No.	10	Effective Date:	16-Nov-18
Prepared By:	Tina Huang			Date:	11-15-18
Approved By:	Kenette Pimentel QA Manager			Date:	11.15.18
Approved By:	Caspar Pang Laboratory Director			Date:	11-15-18

Control Number: 6020-10

1.0 SCOPE AND APPLICATION

1.1. This procedure is applicable for the determination of sub-µg/L concentrations of a large number of elements in wastewater, groundwater, aqueous, extract, soil, sludge, and sediment samples using the Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) method. All matrices require proper sample preparation prior to analysis.

1.2. The elements and their corresponding isotopes are listed in Table 1.

1.3. This SOP is an adaptation of the SW846 Method 6020A.

2.0 SUMMARY OF METHOD

2.1. Metal analytes in water are acid digested from a pre-measured sample. Nitric acid and hydrochloric acid are added to the sample and heated without boiling until the volume is substantially reduced. The digestate is diluted back to its original sample volume using reagent water.

2.2. Metal analytes in soil are acid digested from a pre-measured sample. Nitric acid is added to the sample and heated to initialize digestion. It is further oxidized with 30% hydrogen peroxide and the acid used for final reflux is hydrochloric acid.

2.3. Digestates are introduced by pneumatic nebulization resulting aerosol into a high temperature argon plasma, where they are decomposed, atomized and ionized. The ions produced are extracted from the plasma via the sample and skimmer orifices in the interface region of the mass spectrometer. The extracted ions are guided by an off-axis Lens System to reduce background noise, passes through an Octopole Reaction System (ORS) where some ions require a simple reaction with H_2 or He to eliminate matrix interference prior to entering the Quadrupole Mass Filter (QMF). The QMF separates ions based on their mass-to-charge ratios and ions are counted by electron multiplier detector.

2.4. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a calibration curve.

2.5. **Interference**

2.5.1. Isobaric Elemental Interference. Are caused by isotopes of different elements which form singly or doubly charge ions of the same nominal mass-to-charge ratio. The signal of an isotope of an interfering element is determined and subtracted from the analyte isotope signal.

2.5.2. Isobaric Polyatomic Ion Interference. Are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest. To correct for isobaric polyatomic ion interferences, optimize the cell gas pressure on each analyte so that when the ORS employs simple reaction gases (H_2 and He) side reactions create new and unpredictable interferences. The ORS is equipped with notch filters and by using scanning voltages the created interferences are prevented from reaching the analyzer.

2.5.3. Physical Interference. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. An internal standard can be used to correct physical interference if carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When the intensity level of the internal standard is less than 70% of the intensity of the first standard used during calibration, the sample must be diluted and re-analyzed.

2.5.4. Memory Interference. Contamination by carry-over can occur whenever high concentrations are analyzed in sequence with a low concentration sample. To reduce potential carry-over the rinse period between samples must be long enough to eliminate significant memory effect.

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.

3.1.2. Established limits are shown in Table 5.

4.0 DYNAMIC RANGE

4.1. Linear Dynamic Range (LDR) is the concentration over which the instrument response remains linear.

4.2. Establish LDR of each analyte by determining the signal response from a minimum of three preferably five different concentration across the range. The upper limit should be within 10% (± 10%) of the true value.

4.3. Verify the established LDR every six months or when there is a significant change in the instrument signal, whichever comes first.

5.0 SAMPLE HOLDING TIME AND PRESERVATION

5.1. **Holding Time**

5.1.1. Analyze all samples within 180 days from collection date.

5.2. **Preservation**

5.2.1. Expected sample condition when received in the lab:

- water samples in HDPE or as specified by the project, preserved to pH < 2 with HNO_3
- soil samples in glass jar, brass tubes or as specified by the project

5.2.2. When water sample preservation is requested to be done in the lab, preserve the sample to pH < 2 with HNO_3 and observe at least 24 hours from the time preservative is added before sample digestion.

5.2.3. Store water samples in the same condition as received unless specified in the project requirement.

5.2.4. Store soil samples at ≤ 6°C without freezing.

6.0 ASSOCIATED SOPs

6.1. EMAX-DM01 Data Flow and Review

6.2. EMAX-QA04 Detection Limit (DL)

6.3. EMAX-QA05 Training

6.4. EMAX-QA08 Corrective Action

6.5. EMAX-QC01 Quality Control for Chemicals

6.6. EMAX-QC02 Analytical Standard Preparation

6.7. EMAX-QC04 Balance Calibration

6.8. EMAX-QC05 Calibration of Thermometers

6.9. EMAX-QC06 Volumetric Labware and Micropipette Verification

6.10. EMAX-QC07 Glassware Cleaning

6.11. EMAX-SM03 Waste Disposal

6.12. EMAX-SM04 Analytical and QC Sample Labeling

7.0 SAFETY

7.1. Read all SDS of chemicals listed in this SOP.

7.2. Observe the following precautions during operation or maintenance of the instrument:

- Close the instruments hoods and panels prior to operation.
- Check the exhaust system for a positive extraction at the exhaust duct.
- Handle acids properly.
- Check the drain vessels frequently.
- Make sure that the argon tank is chained.
- Wait for the instrument interface region to cool down prior to instrument maintenance.
- Observe all cautions and warnings stipulated in the Agilent 7500 ICPMS CE/CX Manuals.

7.3. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Observe all chemical hygiene procedures as mentioned in the Chemical Hygiene Plan.

7.4. DO NOT DISPOSE ACIDIC WASTE IN THE TRASH CAN OR IN THE SINK.

7.5. If for any reason, solvent and/or other reagents get in contact with the skin or any other part of the body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS AND REAGENTS

8.1. **Instruments and Supplies**

8.1.1. ICP-MS: Agilent 7500CE Octopole Reaction System : Agilent 7500CX Octopole Reaction System

8.1.2. Autosampler: CETAC ASX-520

8.1.3. Computer: IBM Compatible

8.1.4. RF Generator: Agilent RF Generators

8.1.5. Data Acquisition: Agilent Chemstation version B.03.04 or as updated : Agilent Chemstation version B.04.00 or as updated

8.1.6. Autosampler rack(s): 17 x 100 mm, 60 positions

8.1.7. Culture tubes: 17 x 100 mm, polypropylene

8.1.8. Volumetric Flask: 10 mL, 50 mL, 500 mL, 1000 mL

8.1.9. Micropipettes: 1 mL, 0.100 mL, 5 mL

8.1.10. Pipet Tips: 100 - 1000 µL

8.1.11. Polyethylene bottles: 250, 500, 1000 mL

8.1.12. Liquid argon

8.1.13. Hydrogen, Compressed: Ultra-high purity

8.1.14. Helium, compressed: Ultra-high purity

8.1.15. Balance: Sartorius LC 620 S or equivalent

8.1.16. Spatula: Stainless steel or equivalent

8.1.17. Digestion vessel: 50 mL, 100 mL snap seal

8.1.18. Digestion block: Aluminum blocks or equivalent

8.1.19. Thermometer: Range 0 – 110°C

8.1.20. Filter: Whatman #41 or equivalent

8.1.21. Digestate Container: 50 mL polyethylene vessel, 100 mL Corning snap seal or equivalent

8.1.22. Disposable watch glass

8.1.23. ASX press Rapid Sample Introduction System

8.2. **Chemicals and Reagents**

8.2.1. DI water, ASTM Type II or equivalent

8.2.2. Nitric Acid, Trace high purity grade, concentrated

8.2.3. Hydrochloric acid, Trace high purity grade, concentrated

8.2.4. Hydrogen Peroxide, ACS grade (e.g., VW3690-5 from VWR) or equivalent

8.2.5. Matrix Acid Blank (S0) - Prepare matrix acid solution by mixing 3% by volume nitric acid and 1% by volume hydrochloric acid in DI water. Transfer into a clean HDPE bottle and identify the solution as S0. Use this solution for standards or digestate dilutions.

9.0 STANDARDS

9.1. Internal Standard (IS)

9.1.1. Purchase stock internal standard as certified standard at concentration listed below or equivalent.

STANDARD	SOURCE	ELEMENTS	CONC. (mg/L)	MATRIX
IS	Agilent	Li6, Sc, Ge, Rh, In, Tb, Bi, Lu	100	10% HNO_3

9.2. Tune Check Standard

STANDARD	SOURCE	ELEMENTS	CONC. (mg/L)	MATRIX
Tuning Solution	Agilent	Li, Y, Ce, Tl, Co	0.01	2% HNO_3
Tuning Check Standard (TCS)	High Purity Standard	Co, In, Li, Tl	10	2% HNO_3

9.2.1. Prepare tuning check standard at concentration level suggested below.

STANDARD	TCS Aliquot (mL)	Final volume (mL)	Final Concentration (mg/L)
Intermediate tuning check standard (ITCS)	0.5	50	0.100

9.3. Primary Standards

9.3.1. Stock Standards

9.3.1.1. Purchase certified stock standards as listed in the table below or equivalent.

STOCK STANDARD	SOURCE	ELEMENTS	CONC. (mg/L)	MATRIX
ICAL 1	High Purity	As, B, Se, Sr, Tl, Ti, V, Zn, Sb, Mo, Sn	10	2% HNO_3 and Trace HF Acid
ICAL 2	High Purity	Ba, Be, Cd, Cr, Co, Cu, Pb, Li, Mn, Ni, Ag, U	10	2% HNO_3
ICAL 3	High Purity	Al, Fe, K, Ca, Mg, Na	1000	4% HNO_3
ICAL 4	High Purity	Al, AS, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Pb, K	500	2% HNO_3
ICAL 5	High Purity	Ag, Mo, Sb, Si, Sn, Ti	0.50	2% HNO_3 and Trace HF Acid
Phosphorus	AccuStandard	P	100	Water
Silicon	High Purity	Si	1000	Water Trace HF
Tungsten	AccuStandard Ultra Scientific	W	100	Water and trace NH_4OH

STOCK STANDARD	SOURCE	ELEMENTS	CONC. (mg/L)	MATRIX
Zinc	High Purity	Zn	1000	2% HNO_3
Zirconium	AccuStandard	Zr	100	2-5% HNO_3
Individual Elements	AccuStandard	As Ba Be B Cd Cr Co Cu Pb Li Mn Mo Se Ag Sr Tl Ti U V Zn	1000	5% HNO_3
		Al Fe Ca Mg K Na Sn Sb	10000	

9.3.2. Intermediate Standard

9.3.2.1. Prepare intermediate standards using the standards as suggested below and S0. Other concentrations may also be prepared at the discretion of the analyst.

Standard Name	Stock Standard Name	Conc. (mg/L)	Source	Preparation		
				Aliquot (mL)	Final vol. (mL)	Final conc. (µg/L)
Cation Mix	Al	10,000	AccuStandard	2.5	500	50000
	Ca	10,000	AccuStandard	2.5		50000
	Fe	10,000	AccuStandard	2.5		50000
	Mg	10,000	AccuStandard	2.5		50000
	K	10,000	AccuStandard	2.5		50000
	Na	10,000	AccuStandard	1.25		25000
Trace mix	ICAL 1	10	High Purity	5	10	5000
	ICAL 2	10	High Purity	5		
Mix 7A	ICAL 1	10	High Purity	1	10	1000
	ICAL 2	10	High Purity	1		1000
	ICAL 3	1000	High Purity	1		100000
Si-10	Si	1000	AccuStandard	0.1	10	10000
W-1	W	1000	AccuStandard	0.5	50	1000
Zn-1	Zn	1000	High Purity	0.05	50	1000
Zr-1	Zr	100	AccuStandard	0.5	50	1000
Sr-1	Sr	1000	AccuStandard	0.05	50	1000

9.3.3. Initial Calibration Standard

9.3.3.1. The initial calibration consists of a blank (S0) and four standards (S1, S2, S3 and S4). Prepare the standards as suggested in Table 2 unless otherwise specified by the project. Refer to Table 3 for final concentrations for each analyte. Additional standard points may be added at the discretion of the analyst or as required by the project.

9.3.4. Continuing Calibration Verification (CCV) Standard

9.3.4.1. Prepare CCV using the stock standards and S0 as suggested in Table 2 or as required by the project. Refer to Table 3 for final concentrations for each analyte.

9.3.4.2. Prepare intermediate solution for Low Level CCV (LLCCV) using the stock standards and S0 as suggested in Table 2 or as required by the project. Refer to Table 3 for final concentrations of each analyte.

9.4. **P/A Tuning Standard (PATS)**

9.4.1. Using the calibration stock standard from 9.3.1, IS from 9.2.1 and S0, prepare a 50 µg/L and 100 µg/L mixed standard to a final volume of 100 mL.

STANDARD	Aliqout (mL)	CONC. (µg/L)	Aliqout (mL)	CONC. (µg/ L)
ICAL 1	0.5	50	1	100
ICAL 2	0.5		1	
ICAL 3	0.005		0.01	
Phosphorus	0.5		0.1	
Silicon	0.5		0.1	
Tungsten	0.5		0.1	
Zinc	0.05		0.1	
Zirconium	0.05		0.1	
IS	0.05		0.1	

9.5. **Secondary Source Standard**

9.5.1. Secondary Stock Standard

9.5.1.1. Purchase certified secondary stock standards from a different vendor that contains all analytes as listed in primary stock standards or as listed below.

Standard	Source	Elements	Conc. (mg/L)	Matrix
EMAX Mix 2 (ICV1)	High Purity	As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Sr, Tl, Ti, V, Zn, Ag, Sb, Mo, Li, U, Sn	10	5% HNO_3 + Tr HF
EMAX Mix 3 (ICV2)	High Purity	Al, Fe, Ca, Mg, K, Na	10000	5% HNO_3
Phosphorus	CPI	P	100	0.05% HNO_3
Silicon	High Purity	Si	1000	Water Trace HF
Tungsten	Ultra Scientific	W	100	Water
Zirconium	CPI	Zr	100	2% HNO_3
Individual Elements	CPI	Sb As Ba Be B Cd Cr Co Cu Pb Li Mn Mo Se Ag Sr Tl Sn Ti U V Zn	1000	5% HNO_3
		Al Fe Ca Mg K Na	10000	
6020ICS-0A	Inorganic Ventures	Al, Ca, Fe, Mg, Na, P, K, S	1000	1.4% HNO_3
		C	2000	
		Cl	10000	
		Mo, Ti	20	

9.5.2. Secondary Intermediate Standard

9.5.2.1. Prepare a Cation Mix 2 using secondary source individual stock standard and S0 as suggested below to a final volume of 500 mL.

Standard	Element	Stock Conc. (mg/L)	Source	Aliquot (mL)	Final Conc. (µg/L)
Cation Mix 2	Al	10,000	CPI	2.5	50000
	Ca	10,000	CPI	2.5	50000
	Fe	10,000	CPI	2.5	50000
	Mg	10,000	CPI	2.5	50000
	K	10,000	CPI	2.5	50000
	Na	10,000	CPI	1.25	25000
Tungsten	W	1000	Ultra Scientific	0.5	1000

9.6. **Initial Calibration Verification (ICV)**

9.6.1. Prepare ICV using the secondary stock standards, Cation Mix 2 and S0 as suggested in Table 2. Refer to Table 3 for final concentrations for each analyte.

9.7. **Interference Standards (ICSA/ICSAB)**

9.7.1. Prepare Intermediate ICSA and ICSAB standards at concentration levels suggested below. Refer to Table 3 final concentrations.

Standard	Parent Standard	Aliquot (mL)	Final Volume (mL)	Final Concentration (µg/L)
Intermediate ICSA	6020ICS-0A	5	50	Varied
Intermediate ICSAB	6020ICS-0A	5	50	Varied
	ICAL 1	0.10		
	ICAL 2	0.10		
	Zr	0.010		
	W	0.010		

9.8. **LCS/MS Spike Standard**

9.8.1. Prepare LCS/MS standards as suggested below or as required by the project.

STANDARD	SOURCE	ELEMENTS	CONC. (mg/L)	MATRIX
ICV 1	High Purity	B, Sr, As, Ba, Be, Ag, Cd, Cr, Co, Cu, Tl, Pb, Mn, Ni, Se, V, Zn, Ti, Sb, Mo, Li, U, Sn	10	5% HNO_3+ Tr HF
ICV 2	High Purity	Al, Fe, Ca, Mg, K, Na	1000	5% HNO_3
Phosphorus	CPI	P	1000	0.05% HNO_3
Silicon	High Purity	Si	1000	Water Trace HF
Zirconium	CPI	Zr	100	2% HNO_3
Tungsten	Accu Standard	W	100	Water

9.9. Refer to EMAX-QC02 for detailed procedure of standard preparation and labeling.

10.0 PROCEDURES

10.1. Sample Preparation

10.1.1. Water Samples

10.1.1.1. Based from the work order, determine the samples to form a preparative batch (not to exceed 20 samples per preparative batch). Withdraw the sample(s) from the sample control room and bring them to the preparation area. Allow the samples to equilibrate at room temperature.

10.1.1.2. Shake the sample container. Pour a small amount of sample into the sample cap and trickle just enough to wet the pH indicator strip. Compare the color of the wet strip to the indicator chart displayed in the pH indicator box. Record the pH in the digestion log. If the pH value is <2, proceed to 10.1.3. If the pH value is ≥2, check if special instruction is written on the analysis folder or in the COC. Otherwise, fill out an NCR and inform the supervisor immediately. DO NOT PROCEED WITH THE DIGESTION. WAIT FOR FURTHER INSTRUCTION.

10.1.1.3. Line up the samples chronologically under the hood. Check and record the lot number of the digestion vessels if it has been verified for accuracy. Take digestion vessels and label each one corresponding to the samples withdrawn and place them in front of each sample making sure that their labels agree. Take four more vessels and label them as preparation blank, LCS, matrix spike and matrix spike duplicate.

10.1.1.4. Mix the sample thoroughly to achieve homogeneity. Fill each digestion vessel up to the 50 mL mark. (The reduction of the volume is due to waste minimization).

10.1.1.5. Record the volume in the digestion log. Use reagent water for blank and LCS.

10.1.1.6. Take another digestion vessel; fill it with tap water to 50 mL mark. Put a thermometer inside and let it sit on the digestion block. Turn the thermostat to a pre-determined mark to deliver heat at 90-95°C. Record the temperature reading in the digestion log.

10.1.1.7. **Spike Addition**

10.1.1.7.1. Call for a witness for spike addition. Have the witness verify the setting of the micropipette and the expiration dates of the spike standards.

10.1.1.7.2. Spike LCS and MS samples with 0.15 mL of EMAX MIX 2 and 3 (see Section 9.5.1) solutions. Additional elements are listed below to be spiked only when required by the project.

Analyte	Concentration(mg/L)	Spike Amount(mL)
Phosphorus	1000	0.015
Silicon	1000	0.150
Tungsten	1000	0.015
Zirconium	1000	0.01

10.1.1.8. **Acid Digestion for Dissolved Metals**

10.1.1.8.1. Add 0.5 mL of concentrated HNO_3 and 0.25 mL concentrated HCl to each of the digestion vessels.

10.1.1.8.2. Cap the digestion vessels with disposable watch glass.

10.1.1.8.3. Check that the temperature of the digestion block is 90-95°C (covered vessel) and adjust if necessary.

10.1.1.8.4. Place the digestion vessels on the digestion block and reduce volume of sample by continuous heating without boiling for two hours.

10.1.1.8.5. Reflux gently for another 15 minutes. Remove the digestion vessels from the digestion plate and allow the vessels to cool down.

10.1.1.8.6. Using a reagent water wash bottle, rinse the disposable watch glass collecting the rinsate on the same digestion vessel that it covered. Add 1.0 mL concentration HNO_3 and 0.25 mL concentration HCl for matrix matching.

10.1.1.8.7. Dilute the digestate with reagent water to the 50 mL mark of the digestion vessel. Seal the vessel and shake. If the digestate appears to be turbid, pass it through Whatman #41 filter and collect it in a new polyethylene container.

10.1.1.9. **Acid Digestion for Total Recoverable Metals**

10.1.1.9.1. Add 0.5 mL of concentrated HNO_3 and 0.25 mL concentrated HCl to each of the digestion vessels.

10.1.1.9.2. Cap the digestion vessels with disposable watch glass.

10.1.1.9.3. Check that the temperature of the digestion block is 90-95°C (covered vessel) and adjust if necessary.

10.1.1.9.4. Place the digestion vessels on the digestion block and reduce volume of sample by continuous heating without boiling for two hours.

10.1.1.9.5. Reflux gently for another 15 minutes. Remove the digestion vessels from the digestion block and allow the vessels to cool down.

10.1.1.9.6. Using a reagent water wash bottle, rinse the disposable watch glass collecting the rinsate on the same digestion vessel that it covered. Add 1.0 mL concentration HNO_3 and 0.25 mL concentration HCl for matrix matching.

10.1.1.9.7. Dilute the digestate with reagent water to the 50 mL mark of the digestion vessel. Seal the vessel and shake. If the digestate appears to be turbid, pass it through Whatman #41 filter and collect it in a new polyethylene container.

10.1.2. Soil Samples

10.1.2.1. **Sample Handling**

10.1.2.1.1. Based from the work order, determine the samples to form a preparative batch (not to exceed 20 field samples). Withdraw the sample(s) from the

sample control room designated for metals analysis (passing # 10 sieve) and bring them to the weighing area. Allow the samples to equilibrate at room temperature.

Note: *Sample homogeneity is crucial in metals analysis. If sample is not solely designated for metals analysis (i.e., sample is to be used for other analysis) and it is apparent that sample particles contain > #10 sieve, inform the Supervisor for further instruction.*

10.1.2.1.2. Take digestion vessels and label each one corresponding to the samples withdrawn. Take four more vessels and label them as preparation blank, LCS, matrix spike and matrix spike duplicate.

10.1.2.1.3. Check project sub-sampling requirement. If multi-incremental sub-sampling (MIS) is required, refer to EMAX-SM01, section 5.13.2 for details. Otherwise follow the steps described in EMAX-SM01, section 5.13.1.

10.1.2.1.4. Scoop 1-2 g sub-sample and transfer into a properly labeled digestion vessel. Record the weight to the nearest 0.01 g.

10.1.2.2. **Pre-heating the Digestion Block**

10.1.2.2.1. Place a digestion vessel with reagent water and a temperature monitoring thermometer on the digestion block.

10.1.2.2.2. Turn the digestion block on and set the thermostat to 95°C or to a predetermined temperature to obtain 95°C($\pm$5°C) once the digestion vessel is covered with a watch glass.

10.1.2.2.3. When the temperature reading is 95°C($\pm$5°C), the digestion block is now ready for digestion.

10.1.2.3. **Spike Addition**

10.1.2.3.1. Call for a witness for spike addition. Have the witness verify the setting of the micropipette and the expiration dates of the spike standards.

10.1.2.3.2. Spike LCS and MS samples with 4 mL of EMAX MIX 2 & 3 (Sec. 9.5.1) to LCS and MS samples. Additional elements are listed below to be spiked only when required by the project.

Analyte	Concentration(mg/L)	Spike Amount(mL)
Phosphorus	1000	0.5
Tungsten	1000	0.5
Zirconium	1000	0.3

10.1.2.4. **Acid Digestion**

10.1.2.4.1. Add 10 mL of reagent water and 5 mL of concentrated HCL[1] into each vessel, swirl the vessel to mix the acid and the sample. Add same

[1] Addition of 5 mL HCl is a modification from Method 3050B to enhance recovery of antimony. Refer to Appendix 3 for the comparative study done on ICP-MS.

amount of acid into a clean and empty vessel and designate it as blank. Insert the vessels in the digestion block(s). Cap the vessels with conical watch glass.

10.1.2.4.2. Check the temperature of the digestion block 95°C(±5°C), adjust if necessary. If temperature happens to be ≥ 100°C, adjust the thermostat and wait until temperature falls within 95°C(±5°C). Record the temperature reading in the digestion log.

10.1.2.4.3. Place the digestion vessels on the digestion block and reflux for 15 minutes without boiling.

10.1.2.4.4. Transfer the vessels into unheated digestion block and allow the vessels to cool down for at least 5 minutes. Lift the watch glass and add 10-mL of concentrated HNO_3. Place the watch glass back before working on the next vessel.

10.1.2.4.5. Return the vessels to the digestion block and reflux for another 15 minutes.

10.1.2.4.6. Transfer the vessels into unheated digestion blocks and allow the vessels to cool down for at least 5 minutes. Lift the water glass and add 10 mL 1:1 HNO_3. Place the water glass back before working on the next vessel.

10.1.2.4.7. Return the vessels to the digestion block and reflux for another 15 minutes.

10.1.2.4.8. Transfer the vessels into unheated digestion blocks and allow the vessels to cool down for at least 5 minutes.

10.1.2.4.9. Add 2 mL of reagent water. Then add 3 mL of 30% hydrogen peroxide (H_2O_2) to each vessel, swirling each one of them after every addition to initiate peroxide reaction. Continue to add H_2O_2 until the amount added reaches 10 mL.

10.1.2.4.10. Return the vessels to the heated digestion block. Care must be taken to ensure that losses do not occur due to excessive effervescence.

10.1.2.4.11. Continue to reflux the mixture at 95°C(±5°C) for 15 minutes. Remove the digestion vessels from the digestion block and allow the vessel to cool down for at least 5 mins.

10.1.2.4.12. Lift the watch glass, add 5 mL of concentrated HCl. Swirl the vessel until added reagents are properly mixed with the solution. Place the watch glass before working on the next vessel. Return the vessels into the heated digestion block. Reflux for additional 15 minutes. Subsequently, remove the digestion vessels from the digestion block and allow the vessels to cool down and dilute to 100 mL final volume with reagent water.

10.1.2.4.13. Let the digestate settle and centrifuge or filter with Whatman #41 (see 10.1.5) if necessary otherwise digestates are now ready for analysis.

10.1.2.5. **Digestate Filtration**

10.1.2.5.1. Place Whatman #41 filter paper into each funnel resting on holders.

Rinse the filter papers with reagent water.

10.1.2.5.2. Place a pre-labeled digestate container under each funnel making sure that the labels are visible.

10.1.2.5.3. Check the labels to make sure that they agree. Pour the digestate into the filter.

10.1.2.5.4. Filter and collect the digestates in the labeled container. The digestates are now ready for analysis.

10.2. **Instrument Parameters**

10.2.1. Set instrument parameters as suggested below.

10.2.2. Plasma Condition

- RF Power: 1500 W
- RF Matching: 1.68 V
- Sample Depth: 8.0 mm
- Torch Height: -0.4 mm
- Torch Vertical: 0 mm
- Carrier Gas: 0.9 L/min
- Make-up Gas: 0.15 L/min Note: Total Carrier and Make-up gas not to exceed 1.1 L/min.
- Peristaltic pump: 0.1 rps
- Spray Chamber (S/C) Temp: 2°C

10.2.3. Ion Lenses

- Extract 1: 0 V
- Extract 2 : -140V
- Omega Bias-ce: -22 V
- Omega Lens-ce: -1.2 V
- Cell Entrance: -26 V
- QP Focus: 2 V
- Cell Exit: -30 V

10.2.4. Octopole Parameters

- Octopole RF: 150 V
- Octopole Bias: -6 V

10.2.5. Q-Pole Parameters

- AMU Gain: 127
- AMU Offset: 125

- Axis Gain: 0.9996
- Axis Offset: 0.04
- QP Bias: -3 V

10.2.6. Detector Parameters

- Discriminator: 8 mV
- Analog HV: 1630 V
- Pulse HV: 990 V

10.2.7. Reaction Cell

- H_2 Gas: 3.0 mL/min
- He Gas: 4 mL/min

10.2.8. Adjust the instrument parameters to optimize the instrument performance in conformance to the tuning requirement.

10.2.9. Print the most current instrument parameters and place in the appropriate binder for easy reference. Replaced instrument parameter set-up should be archived chronologically for future reference and historical record.

10.3. **Calibration**

10.3.1. Instrument Set-Up

10.3.1.1. Set up the ICP-MS with proper operating parameters. Refer to Section 10.3.

10.3.1.2. Ignite the plasma and allow the instrument to become thermally stable for at least 30 minutes.

10.3.1.3. Check the peristaltic pump to deliver a steady flow.

10.3.2. Tuning the Instrument

10.3.2.1. Tune the instrument according to Normal Mode, Hydrogen Mode and Helium Mode without the internal standard. Refer to Section 10.3 for parameters. On the ICP-MS main Menu, go to Instrument and click Tune and run the tuning solution without the internal standard. After about 60 seconds (making sure the solution is in the system) click start and evaluate the counts of the isotopes according to the table below.

Mode	**Range**		
Normal	Li6 ≥ 6400 counts	Y89 ≥ 16000 counts	Tl205 ≥ 9600 counts
Hydrogen	Ar/Ar78 < 10	Y89 ≥ 3000 counts	
Helium	V51/Co59 < 0.6	Co59 ≥ 7000 counts	ArCl-75 < 10 counts

10.3.2.2. If non-compliant, adjust parameters (e.g. Torch height, Torch vertical, Octopole Bias and QP Bias) and repeat the tune process until the required range is met.

10.3.2.3. Click Generate report for a full scan of the tune. Save all tune values to the current method.

10.3.3. Perform a P/A Factor Evaluation

10.3.3.1. Analyze the 50 µg/L P/A tuning standard.

10.3.3.2. Under "Tune," access "P/A Factor." Select "Load masses from the acquisition method". Subsequently, highlight and delete Ca, $(Cd)^{106}$, $(Cd)^{108}$, $(Pb)^{206}$, $(Pb)^{207}$ from the list of elements, then select "run."

10.3.3.3. Upon completion, accept the changes.

10.3.3.4. Accept the new P/A Factors and save file as "norm.u".

10.3.3.5. Similarly, analyze the 100 ug/L P/A tuning standard as described above, with the check box for "Merge current data" enabled.

10.3.4. Perform Tune Check

10.3.4.1. Analyze the intermediate tune check solution (9.1.1) using 4 replicates.

10.3.4.2. Evaluate the tune check so that the mass calibration differs no more than 0.1 AMU of the true value and the resolution to be less than 0.9 AMU full width at 10% peak height RSD should less than or equal to 5% for the 4 replicate analysis.

10.3.5. Initial Calibration (ICAL)

10.3.5.1. Set the instrument rinse time to 90 seconds between each standard solution.

10.3.5.2. Analyze a calibration blank (S0) and a multi-point calibration standard (Section 9.3.3.1).

10.3.5.3. Refer to Appendix 1 for ICAL acceptance criteria and /or corrective action.

10.3.6. Initial Calibration Verification (ICV)/ Instrument Calibration Blank (ICB)

10.3.6.1. Analyze the ICV (Section 9.5.1) from a second source to verify the concentration of the ICAL.

10.3.6.2. Analyze a low-level ICV (LLICV) from the same source as the calibration standard to verify the lower limit of quantitation (RL).

10.3.6.3. Analyze an ICB after LLICV to demonstrate absence of instrument contamination.

10.3.6.4. Refer to Appendix 1 for ICV, LLICVand ICB acceptance criteria and /or corrective action.

10.3.7. Continuing Calibration Verification (CCV)/ Continuing Calibration Blank (CCB)

10.3.7.1. Analyze CCV to check the validity of the ICAL every 10 samples and at the end of the analytical sequence.

10.3.7.2. Analyze low-level CCV (LLCCV) to check the system stability at low end of ICAL at the end of the analytical sequence.

10.3.7.3. Analyze a CCB every after LLCCV to demonstrate the absence of instrument contamination.

10.3.7.4. Refer to Appendix 1 for CCV, LLCCV and CCB acceptance criteria and/or corrective action.

10.3.8. ICSA and ICSAB

10.3.8.1. Analyze ICSA and ICSAB at the beginning of each analytical run and every 12 hours thereafter**.**

10.3.9. Establishing Instrument Detection Limit (IDL)

10.3.9.1. Analyze a minimum of seven consecutive method blanks.

10.3.9.2. Repeat the process within three non-consecutive days.

10.3.9.3. Calculate the standard deviation of each run.

10.3.9.4. The average of the standard deviation of the three runs determines the IDL for each analyte.

10.3.9.5. Establish IDL at least every 3 months.

10.3.10. Verifying Linear Dynamic Range (LDR)

10.3.10.1. Verify the LDR by preparing a standard at the upper limit of the LDR. Analyze and quantitate against the normal calibration curve. Percent recovery must be within ± 10% of expected value. If non-complaint re-establish LDR.

10.3.10.2. At a minimum perform LDR verification every six months.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. From the main menu of the ICPMS top window, go to Sequence and create the analytical sequence by editing the sample log table. Refer to Table 4.

10.4.1.2. Set QC limits on QC samples for easy verification while analytical samples are running.

10.4.1.3. Using the sample log table, input the standards and the digestates to be analyzed. Samples are analyzed in the order they appear in the scanner.

10.4.1.4. Transfer about 5 mL of its content into the autosampler tubes placing them on the autosampler rack in the same order as the analytical sequence. A dilution of x10 for soil samples is required due to the high acid content of the digestate.

10.4.1.5. Dilution Test sample is prepared at 5 times dilution. Seal the tube with Parafilm and invert the tube several times to ensure adequate mixing.

10.4.1.6. Prepare a Post Digestion Spike test sample. Using the un-spiked sample digestate (preferably the QC sample), add MS standard to maintain the same spike level as the MS sample digestate.

10.4.1.7. A 100 µg/L internal standard shall be spiked into each sample.

10.4.1.8. Set the prepared analytical samples into the auto-sampler and start the analytical run.

10.4.2. Sample Result Evaluation

10.4.2.1. Check QC parameters as soon as the data is available.

- Check the initial calibration verification (ICV, LLICV and ICB) against Appendix 1.
- Check MB, LCS against Appendix 1. Perform specified corrective action if necessary.
- Check the MS, duplicate sample, serial dilution and post digestion spike results. If matrix interference is indicated, dilute the sample and re-analyzed.
- Check intensity of internal standard on each sample.

- If any of the above checkpoints is non-compliant, perform the specified corrective action in Appendix 1. If results indicate digestion problem, order re-digestion for affected samples. If unresolved, consult the Supervisor for further action.

10.4.2.2. Check the sample rack to ensure that the Autosampler did not skip any sample.

10.4.2.3. Check concentration of target analytes. If the response exceeds LDR, dilute and reanalyze the sample at a concentration within the LDR.

10.4.2.4. Check other QC requirements like ICSA, ICSAB, CCV, LLCCV, CCB against Appendix 1.

10.5. **Calculations**

10.5.1. The computer software is designed to calculate the concentration in the digestate, based on the assumption that the initial calibration is linear through the origin. Thus, for aqueous samples, the computer-generated result represent the concentration of the sample.

10.5.2. For water samples

$$C_s = C_i\left(\frac{ExpAmt}{Aliquot}\right)\left(\frac{Vd}{ExpVd}\right)DF \qquad \text{Eq.-10.5.2}$$

where:

C_s – Concentration in the sample, μg/L

C_i – Concentration in the digestate, (based on rawdata), μg/L

V_d – Digestion volume, mL

ExpVd – Expected digestion volume, mL

DF – Dilution factor

ExpAmt – Expected amount for digestion, mL

Aliquot – Amount digested, mL

10.5.3. For solids, use the following equation to calculate the concentration

$$Cs = C_i\left(\frac{ExpAmt}{Aliquot}\right)\left(\frac{V_d}{ExpVd}\right)\left(\frac{100}{100-\%H_2O}\right)\times DF\times 0.1 \qquad \text{Eq.-10.5.3}$$

where:

C_s – Concentration in the sample, mg/Kg

C_I – Concentration in the digestate (based on rawdata), μg/L

V_d – Digestion volume, mL

ExpVd – Expected digestion volume, mL

ExpAmt – Expected amount for digestion, g

Aliquot – Amount digested, g

% H_2O – Percent moisture of the sample

DF – Dilution factor

0.1 – Conversion factor

10.5.4. Calculate the percent recovery (%R)

$$\% \ Recovery = \frac{Cf - C}{Cs} * 100 \qquad \text{Eq.-10.5.4}$$

where:

C_f – Concentration found, µg/ L

C – Concentration of sample, µg/L

C_s – Concentration of spike, µg/L

10.5.5. Relative Percent Difference (%RPD)

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \qquad \text{Eq.-10.5.5}$$

where:

RPD – Relative Percent Difference

C_1 – Measured concentration of the first sample aliquot

C_2 – Measured concentration of the second sample aliquot

10.6. **Data Reduction**

10.6.1. Make a copy of the analytical run log and sample preparation log.

10.6.2. Highlight the data to be reported.

10.6.3. Print a copy of the raw data and the QC report.

10.6.4. Keep all other data generated with the analytical folder marked with “For record only” for traceability purpose.

10.7. **Report Generation**

10.7.1. Print the summary of the analytical run, perform a data transfer into a disk, and convert the instrument electronic output file into a CSV file format.

10.7.2. Run the ICPCHK.exe program for calibration check.

10.7.3. Identify samples that need to be re-analyzed, if any, and report all samples that met the analytical requirements.

10.7.4. Generate the report using the following reporting program:

Executable Files	Required Support Files	Output
ICPMS00.exe	Login File (requires network) Project support files (.met, .QC, .CRF, .DL, .pln, F:\ST\ProjectCode.txt) ICP-MS raw data output (e.g.,IF6A023.CSV) Digestion log (e.g.,IMA027.CSV)	[Analytical Sequence].CAL – this file summarizes ICV, ICSA and ICSAB results. [Analytical Sequence].CCB – this file summarizes calibration blanks (ICB and CCBs relevant to reported SDG. [Analytical Sequence].CCV – this file summarizes CCVs relevant to reported SDG. Limits.CSV – this file contains target analytes and its LOQ,LOD and DL SQ.CSV – this file contains the samples analyzed for the SDG being processed. ARAW.CSV – this file contains the raw data of samples analyzed for the SDG being processed Method.txt [this file integrates the login sample information and the analytical sample information]
MET1F3.exe	Limits.CSV, SQ.CSV and ARAW.CSV	Sample Results (Form1) LCS Summary Forms (Form 3) MS Summary Forms (Form 3) Analytical Spike Summary Forms Serial Dilution Summary Forms
METF1C.exe	Form 1	[SampleID].TXC – Combines sample results with multiple dilutions.
LABCHRNX.exe	method.txt	Lab Chronicle
CN2.exe	Login File, method.txt, Form 1, Form 3	Case Narrative

10.8. **Data Review**

10.8.1. Arrange the analysis package in sequence as detailed below.

- Case Narrative

- Lab Chronicle
- Sample Results
- LCS/LCSD Summary
- MS/MSD Summary
- Sample Duplicate Summary
- Analytical Run Log
- ICAL Summary
- ICV Summary
- CCV Summary
- Sample Preparation Log
- Non-Conformance Report (if any)

10.8.2. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- Check Project Specific Requirement (PSR) or Appendix 1 for acceptance criteria.
- Check frequency of calibration verification. Verify results to be within acceptance limits.
- Check of target analytes concentration to be within linear range.
- Verify interference check results to be within acceptance limits.

10.8.3. If any of the above checkpoints is non-compliant, re-analysis is required.

10.8.4. Review the attached logs that they are properly filled.

10.8.5. Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.8.6. Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.7. Submit the analytical folder for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Instruments shall receive routine preventive maintenance that is properly recorded in the instrument-specific maintenance logs. The list of maintenance is summarized in Form 6020FM. The practice ensures optimum operating condition of the equipment thus reducing the possibility of frequent instrument malfunction.

Maintenance Activity	Description	Frequency
Verification	Verify instrument parameters to ensure normal operating conditions. Change tubings as necessary. Perform system tune check. Check instrument performance (e.g., ICV/ICB)	Daily prior to analysis

Maintenance Activity	Description	Frequency
Vacuum System Maintenance	Inspect vacuum hoses and exhaust tubes for possible problems. Check pump for evidence of leakage	Daily prior to analysis
Documentation	Record all instrument maintenance performed in the instrument maintenance log.	Daily prior to analysis
Ion Lens Cleaning	Remove and clean surfaces of the ion lens. Sonicate ion lens parts	As necessary
System Cleaning	Remove covers and clean dust from fans and vent covers	Every 6 months or as necessary
Pump Maintenance	Replace oil mist filter, drain and replace mechanical pump oil. Verify proper pump operation	Every 6 months
Inspection	Perform general inspection of the complete system	Once a year

11.0 QUALITY CONTROL

11.1. Sample Preparation QC

11.1.1. All labwares used in the sample preparation shall be properly treated as specified in EMAX-QC07.

11.1.2. A preparative batch consists of 20 or fewer samples of the same matrix that are prepared for analysis simultaneously or sequentially, using the same lots of all reagents.

11.1.3. Every preparative batch shall have at least one method blank, one LCS and a set of MS/MSD unless otherwise specified by the project. These QC samples shall be digested together with the field samples.

11.1.4. All reagents shall be subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

11.2. Sample Analysis QC

11.2.1. Perform a tune check before every analytical run, an initial calibration and initial calibration verification (ICV / LLICV). Obtain the ICV standard from a different source from that of the initial calibration and LLICV from the same source as the ICAL. Analyze an instrument calibration blank (ICB) after the LLICV. No further analysis shall be valid unless acceptance criteria are met.

11.2.2. Monitor the intensities of all internal standards for every analysis. Refer to Appendix 1 for acceptance criteria.

11.2.3. Verify inter-element and background correction factors with ICSA and ICSAB standards after ICB every 12 hours.

11.2.4. Verify calibration with continuing calibration verification (CCV) standard and continuing calibration blank (CCB) after every ten samples and at the end of the analytical run. Also verify LLCCV at the end of the analytical run.

11.2.5. Evaluate results of MS/MSD to document matrix interference.

11.2.6. Perform Post Digestion Spike whenever recoveries for MS/MSD failed.

11.2.7. Evaluate Dilution Test result if post digestion spike result failed to meet the acceptance criteria. Failure typically happens when analyte concentrations are high.

11.2.8. Refer to Appendix 1 for acceptance criteria.

11.3. **Method QC**

11.3.1. A valid DL and LOD must exist prior to sample analysis. Refer to EMAX-QA04 for details.

11.3.2. Perform dynamic range study at least every six months or whenever there is a significant change in instrument response unless otherwise specified by the project.

11.3.3. All analysts conducting this analysis must have an established Demonstration of Capability (DOC) as described in EMAX-QA05.

12.0 CORRECTIVE ACTION

12.1. Quality control procedures and corresponding corrective actions are summarized in Appendix 1.

12.2. If tune is non-compliant, consider the following suggestions to correct the problem:

- Check the instrument settings and make sure that the instrument parameters are properly set up.
- Check argon gas flow.
- Perform auto tune or visual optimization
- If the problem persists, inform the Supervisor.

12.3. If correlation coefficient (R) of ICAL is non-compliant, consider the following suggestions to help you correct the problem:

- Check the calibration points for possible presence of out-lier. If out-lier is present, prepare a fresh standard and repeat the calibration.
- Check the connections and make sure that they are air-tight. Perform maintenance as needed.
  - Presence of bubbles is indicative of poor connection between the sipper and the nebulizer.
  - Poor precision to inability to light the plasma is a symptom of a poor drain tube connection
  - Poor precision and carry-over problems are indicative of a dirty spray chamber.
  - Relative increase in the sensitivity ratio of the higher: lower atomic number elements are indicative of stretched pump tubing. The sample flow rate decreases as the tubing stretches.
- Check the argon gas flow. Loss of signal is indicative of low or no argon gas flow.
- Poor precision and a gradual loss of signal is indicative of “salting-out” in the nebulizer and/or spray chamber due to samples with high dissolved or suspended solids. This problem will necessitate nebulizer and spray chamber cleaning.
- If the problem persists, inform the Supervisor.

12.4. If ICV is non-compliant, consider the following suggestions to help you correct the problem:

- If the RSD is high it is indicative that the carry-over might be present in the spray chamber.

- If result is bias high, prepare a fresh standards and repeat calibration.
- If the problem persists, inform the Supervisor.

12.5. If ICB/CCB is non-compliant, consider the following suggestions to correct the problem:

- Prepare a fresh calibration blank solution. Perform instrument rinsing and repeat the ICB/CCB prior to re-analysis of associated sample(s).
- Carry-over problem is indicative of dirty spray chamber, nebulizer and/or torch. Perform instrument maintenance and repeat the calibration.
- If the problem persists, inform the Supervisor.

12.6. If CCV or LLICV or LLCCV is non-compliant, consider the following suggestions to correct the problem:

- Check the connections prior to re-running the ICAL. Refer to Section 12.3.
- Prepare a new standard and repeat the ICAL.

12.7. If the intensity of the Internal Standard is non-compliant, consider the following suggestions to correct the problem:

- Check for drift occurrence by observing the internal standard intensities in the calibration blank.
- If drift has occurred, terminate the analysis, recalibrate, verify the new calibration and reanalyze the affected samples.
- If drift has not occurred, dilute affected samples five fold and reanalyze with the addition of appropriate amounts of internal standards.

12.8. If Method Blank is non-compliant, consider the following suggestions to correct the problem:

- Rule-out instrument contamination by checking the CCBs. Refer to Section 12.5.
- Rule-out reagent contamination by testing each reagent as described in EMAX-QC01.
- Rule-out digestate vessel contamination by adding verified reagents heating the vessels prior to testing.
  - Common environmental contaminants – Ca, Si, Fe, Na, Mg, K, Tl, Cu, Mn, can be minimized by maintaining the lab clean.
- Other sources of contamination:
  - Sweat contains Ca, Mg, Pb, K, NH_4^+ , SO_4^{-2}, PO_4^{-3}, and Cd (for those who smoke).
  - Cosmetics can contain high concentrations of Al, Be, Ca, Cu, Cr, K, Fe, Mn, Ti, and Zn.
  - Some hair dyes contain $Pb(OAC)_2$.
  - Dandruff shampoo can contain significant levels of Se.
  - Eye make-up may contain Hg as a preservative.
  - Calamine lotion is almost pure ZnO.
  - Watches and jewelry contain an assortment of elements and should not be worn in the laboratory.
- Re-digest MB and the associated samples with reagents free of contamination or with newly opened reagents.
- If the problem persists, inform the Supervisor.

12.9. If LCS is non-compliant, consider the following suggestions to correct the problem:

- If result is bias-high, check the LCS standard by analyzing at the spike level.

  If the LCS check is within 80-120 % of the expected value, check the calibration of the micropipette use for spiking. Re-digest and re-analyze the LCS and the associated samples.

  If the LCS check is not within 80-120%, prepare a fresh LCS standard, re-digest and re-analyze LCS and the associated samples.

- Common Problems with Ag, As, Ba, Pb, and Cr, indicating stock standard degradation, are as follows:

  Low Silver (Ag) recovery is indicative of Chloride contamination causing AgCl precipitation

  Low Arsenic (As) recovery is indicative of loss during sample preparation as volatile oxides (AsO_3) or precipitation as $AsCl_3$

  Low Barium (Ba) recovery is indicative of SO_4or CrO_4 contamination. Barium will form precipitates with HF and H_2SO_4.

  High Lead (Pb) recovery is indicative of environmental contamination.

12.10. A Non-Conformance Report (NCR) is required when the following circumstances occur:

- Anomalies other than those specified in Appendix 1 are observed.
- Sample is out of technical holding time.

12.10.1. Refer to EMAX-QA08 for NCR details.

13.0 POLLUTION PREVENTION

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2. Prepare all standards in fume hoods.

14.0 WASTE MANAGEMENT

14.1. No sample may be dumped in the laboratory sink.

14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.

14.3. Place all waste generated during the analytical process in properly labeled satellite waste containers for proper collection.

14.4. Dispose all unused samples, digestates, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. **Definition of Terms**

15.1.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

15.1.1.1 **Preparation Batch** - is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.

15.1.1.1 **Analytical batch -** is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.2. Detection Limit (DL) – The lowest concentration or amount of the target analyte that can be identified, measured and reported with confidence that the analyte concentration is not a false positive.

15.1.3. Limit of Detection (LOD) – An estimate of the minimum amount of substance that an analytical process can reliably detect.

15.1.4. Limit of Quantitation (LOQ) – The minimum levels, concentrations or quantities of target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

15.1.5. Safety Data Sheet (SDS) – is where the physical data, toxicology and safety precaution of a certain substance is listed.

15.1.6. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.7. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.1.8. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.9. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical procedure to monitor contamination.

15.1.10. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.

15.1.11. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.

15.1.12. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

15.1.13. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.14. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.15. Matrix – is a physical state of a sample. Most of environmental samples are classified as water, soil or air.

15.1.16. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.

15.1.17. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

15.1.18. Reagent Water – is purified water free from any target analyte or any other substances that may interfere with the analytical process.

15.1.19. Reagent Soil – organic-free Ottawa sand or equivalent.

15.2. **Application of EMAX QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing metals analysis. In instances where there is a project or program QAPP, the requirements given in the project shall take precedence over this SOP.

15.3. **Department of Defense (DoD) and Department of Energy (DOE) Projects**

15.3.1. Samples from DoD and/or DOE sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD and DOE Consolidated Quality Systems Manual (QSM), latest update, is applied.

16.0 REFERENCES

16.1. "Test Methods for Evaluating Solid Waste, Physical / Chemical Methods", EPA Publication SW-846 Update IV, Method 6020A.

16.2. Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry, Method 200.8 Rev. 5.4, 1994.

16.3. USEPA SW846, Method 3050B, Revision 2, December 1996.

16.4. Title 40 Code of Federal Regulations, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, latest edition.

16.5. EMAX Quality Systems Manual, as updated.

17.0 APPENDICES

17.1. **Tables**

17.1.1. Table 1 ICP-MS Elements & Isotopes

17.1.2. Table 2 Calibration Standard and Verification Preparation

17.1.3. Table 3 Calibration Standards Concentration and Reporting Limit

17.1.4. Table 4 ICP-MS Analytical Sequence

17.1.5. Table 5 DL, LOD, LOQ and Linear Range Concentration Levels

17.2. **Figures**

17.2.1. Figure 1 Typical Sample Report

17.2.2. Figure 2 Typical LCS/LCD Summary

17.2.3. Figure 3 Typical MS/MSD Summary

17.2.4. Figure 4 Typical Case Narrative

17.3. **Appendices**

17.3.1. Appendix 1 Summary of Quality Control Procedures

17.3.2. Appendix 2 Demonstration of Capability

17.3.3. Appendix 3 Comparative Study of Modified 3050B

17.4. **Forms**

17.4.1. 6020FS Sample Preparation Log

17.4.2. 6020FA Analytical Run Log

17.4.3. 6020FM Instrument Maintenance Log

Table 1: ICP-MS ELEMENTS & ISOTOPES

ELEMENT	SYMBOL	MASS	Tune Mode	Internal Standard
Aluminum	Al	27	3	Sc45
Antimony	Sb	121	3	In115
Arsenic	As	75	2	Ge72
Barium	Ba	137	3	In115
Beryllium	Be	9	3	Li6
Boron	B	11	3	Li6
Cadmium	Cd	111	3	In115
Calcium	Ca	43	1	Sc45
Chromium	Cr	53	2	Sc45
Cobalt	Co	59	3	Sc45
Copper	Cu	63	2	Sc45
Iron	Fe	57	1	Sc45
Lead	Pb	208	3	Tb159
Lithium	Li	7	3	Li6
Magnesium	Mg	24	3	Sc45
Manganese	Mn	55	3	Sc45
Molybdenum	Mo	95	3	In115
Nickel	Ni	60	2	Sc45
Phosphorus	P	31	3	Sc45
Potassium	K	39	3	Sc45
Selenium	Se	78	1	Ge72
Silicon	Si	28	1	Sc 45
Silver	Ag	107	3	In115
Sodium	Na	23	1	Sc45
Strontium	Sr	88	3	Y89
Thallium	Tl	205	3	Tb159
Tin	Sn	118	3	In115
Titanium	Ti	47	3	Sc45
Tungsten	W	182	3	Tb159
Uranium	U	238	3	Tb159
Vanadium	V	50	2	Sc45
Zinc	Zn	66	3	Ge72
Zirconium	Zr	90	3	Ge72

Tune Mode: 1=Reaction H_2 Mode; 2=Collision He Mode; 3= Normal Mode

Table 2 : CALIBRATION AND VERIFICATION STANDARDS

Standard	Source		Preparation (Final Vol. (500 mL)	
	Mixed Std. Name	Concentration (mg/L)	Aliquot (mL)	Final Conc. (µg/L)
S1	ICAL 1, 2	10, 10	0.025, 0.025	0.5
	ICAL 3	1000	0.025	50
	Silicon	1000	0.05	100
	Zn-1	1	0.25	0.5
	W-1	1	0. 25	0.5
	Phosphorus	100	0.125	25
	Zr	100	0.025	5
S2	ICAL 4	MIXED	5.0	MIXED
	ICAL 5	MIXED	5.0	MIXED
	W	100	0.025	5
	Zr	100	0.05	10
S3	ICAL 4	MIXED	25	MIXED
	ICAL 5	MIXED	25	MIXED
	W	100	0.125	25
	Zr	100	0.125	25
S4	ICAL 4	MIXED	50	MIXED
	ICAL 5	MIXED	50	MIXED
	W	100	250	50
	Zr	100	250	50
ICV (Individual Spike)	Fe, Al, Ca, K, Mg, Na	10000	1.5	30
	Tl, V, Cr, Ni, Co, Cu, Zn, As, Se, Sr, Mo, Cd, Sn, Ba, Pb, U	1000	0.15	0.3
	Mn	1000	1.0	2
	Sb	1000	0.03	0.06
	Li, Be, B, Ag, W	1000	0.015	0.03
	Zr	100	0.15	0.03
	P	1000	0.15	0.3
	Si	1000	1.5	3.0
	Ti	1000	0.15	0.3
CCV	ICAL 4	MIXED	25	MIXED
	ICAL 5	MIXED	25	MIXED
	W	100	0.125	25
	Zr	100	0.125	25
LLICV / LLCCV (water)	ICAL 1, 2	10, 10	0.1, 0.1	0.001, 0.001
	ICAL 3	1000	0.1	0.1
	Zn	1000	0.009	0.01
	B	1000	0.009	0.01
	Sr	1.0	1.0	0.002
	P	100	0.5	0.05
	Zr	100	0.05	0.005
	W	100	0.02	0.002
	Si	1000	0.100	0.1
LLICV / LLCCV (soil)	ICAL 1	10	0.05	0.0005
	ICAL 2	10	0.05	0.0005
	ICAL 3	1000	0.05	0.05
	Zn	1.0	4.0	0.004

Table 3: CALIBRATION STANDARDS CONCENTRATION AND REPORTING LIMIT

ELEMENT	ICAL (µg/L)				ICV (µg/L)	CCV (µg/L)	ICSA (µg/L)	ICSAB (µg/L)
	S1	S2	S3	S4				
Aluminum	50	5000	25000	50000	30000	25000	100000	100000
Antimony	0.5	10	50	100	60	50	0	20
Arsenic	0.5	50	250	500	300	250	0	20
Barium	0.5	100	500	1000	300	500	0	20
Beryllium	0.5	5	25	50	30	25	0	20
Boron	0.5	10	50	100	30	50	0	20
Cadmium	0.5	50	250	500	300	250	0	20
Calcium	50	5000	25000	50000	30000	25000	100000	100000
Chromium	0.5	50	250	500	300	250	0	20
Cobalt	0.5	50	250	500	300	250	0	20
Copper	0.5	50	250	500	300	250	0	20
Iron	50	5000	25000	50000	30000	25000	100000	100000
Lead	0.5	50	250	500	300	250	0	20
Lithium	0.5	5	25	50	30	25	0	20
Magnesium	50	5000	25000	50000	30000	25000	100000	100000
Manganese	0.5	300	1500	3000	2000	1500	0	20
Molybdenum	0.5	50	250	500	300	250	2000	2000
Nickel	0.5	50	250	500	300	250	0	20
Phosphorus	25	50	250	500	300	250	100000	100000
Potassium	50	5000	25000	50000	30000	25000	100000	100000
Selenium	0.5	50	250	500	300	250	0	20
Silver	0.5	5	25	50	30	25	0	20
Silicon	100	500	2500	5000	3000	2500	0	200
Sodium	25	5000	25000	50000	30000	25000	100000	100000
Strontium	0.5	50	250	500	300	250	0	20
Thallium	0.5	50	250	500	300	250	0	20
Tin	0.5	50	250	500	300	250	0	20
Titanium	0.5	50	250	500	300	250	2000	2000
Tungsten	0.5	5	25	50	30	25	0	20
Uranium	0.5	50	250	500	300	250	0	20
Vanadium	0.5	50	250	500	300	250	0	20
Zinc	1	50	250	500	300	250	0	20
Zirconium	5	10	25	50	30	25	0	20

Table 4: ICP-MS ANALYTICAL SEQUENCE

RUN ID LABEL	SAMPLE DESCRIPTION	SOLUTION ID LABEL
S0	Calibration Standard 1 (blank)	S0
S3, S4, S5	ICAL Standards	S3, S4, S5
ICV	Initial Calibration Verification	ICV
LLICV	Low Level Initial Calibration Verification	LLICV
ICB	Initial Calibration Blank	ICB
ICSA	Initial Interference Solution A	ICSA
ICSAB	Initial Interference Solution A and B	ICSAB
CCV1	Continuing Calibration Verification #1	CCV
CCB1	Continuing Calibration Blank #1	S0
IMSSSSB[2]	Preparation Blank	
IMSSSSL/C	Lab Control Sample	
Sample 1	Sample 1	
Sample 1M	Sample 1 Matrix Spike	
Sample 1S	Sample 1 Matrix Spike Duplicate	
Sample 1J	Sample 1 Serial Dilution(5x dilution sample 1)	
Sample 1A	Sample 1 Post Digestion spike	
Samples 2 to 4	Sample 2 to Sample 5	
CCV2	Continuing Calibration Verification #2	CCV
CCB2	Continuing Calibration Blank #2	S0
Samples 5 to 14	Maximum of 10 Samples	
CCV3	Continuing Calibration Verification #3	CCV
CCB3	Continuing Calibration Blank #3	S0
Samples 15 to 20	Sample 15 to 20 or a maximum of 10 samples (sample 15 to 24)	
ICSA	Initial Interference Solution A	ICSA
ICSAB	Initial Interference Solution B	ICSAB
CCV4	Continuing Calibration Verification #4	CCV
LLCCV	Low Level Continuing Calibration Verification	LLCCV
CCB4	Continuing Calibration Blank #4	S0

[2] where IMSSSS is the digestion batch reference.

Table 5: DL, LOD, LOQ AND LINEAR RANGE CONCENTRATION LEVELS

ELEMENT	WATER (µg/L)			SOIL (mg/kg)			LINEAR RANGE
	DL	LOD	LOQ	DL	LOD	LOQ	µg/L
Aluminum	10	20	100	5	10	100	50000
Antimony	0.25	0.5	1	0.1	0.2	0.5	100
Arsenic	0.1	0.2	1	0.05	0.1	0.5	500
Barium	0.25	0.5	1	0.072	0.1	0.5	1000
Beryllium	0.05	0.1	1	0.05	0.1	0.5	50
Boron	2.5	5	10	2.5	5	10	100
Cadmium	0.1	0.2	1	0.057	0.1	0.5	500
Calcium	13	25	100	17	20	100	50000
Chromium	0.1	0.2	1	0.05	0.1	0.5	500
Cobalt	0.1	0.2	1	0.05	0.1	0.5	500
Copper	0.25	0.5	1	0.1	0.2	0.5	500
Iron	5	10	100	5	10	100	50000
Lead	0.05	0.1	1	0.05	0.1	0.5	500
Lithium	0.25	0.5	2	0.139	0.2	0.5	50
Magnesium	5	10	100	10	20	100	50000
Manganese	0.1	0.2	1	0.153	0.2	0.5	3000
Molybdenum	0.25	0.5	2	0.1	0.2	0.5	500
Nickel	0.1	0.2	1	0.063	0.1	0.5	500
Phosphorus	12.5	25	50	12.5	25	50	500
Potassium	10	20	100	10	20	100	50000
Selenium	0.15	0.3	1	0.05	0.1	0.5	500
Silicon	10	20	50				5000
Silver	0.1	0.2	1	0.05	0.1	0.5	50
Sodium	25	50	100	10	20	100	50000
Strontium	0.5	1	2	0.05	0.1	0.5	500
Thallium	0.1	0.2	1	0.05	0.1	0.5	500
Tin	0.1	0.2	1	5	10	20	500
Titanium	0.25	0.5	2	0.125	0.25	0.5	500
Tungsten	0.5	1	2	0.5	1	2	50
Uranium	0.05	0.1	1	0.05	0.1	0.5	500
Vanadium	0.25	0.5	1	0.19	0.3	0.5	500
Zinc	5	10	20	0.683	1	2	500
Zirconium	1	2	5	1	2	5	50

Figure 1: TYPICAL SAMPLE REPORT

METHOD SW6020A
METALS BY ICP-MS

Client : XYZ, INC.		Date Collected: MM/DD/YY HH:HH	
Project : CLEAN PROJECT		Date Received: MM/DD/YY	
SDG NO. : YYMXXX		Date Extracted: MM/DD/YY HH:HH	
Sample ID: ABCDEF-02		Date Analyzed: MM/DD/YY HH:HH	
Lab Samp ID: MXXX-02		Dilution Factor: 1	
Lab File ID: F6M15094		Matrix: WATER	
Ext Btch ID: IMM006W		% Moisture: NA	
Calib. Ref.: F6M15085		Instrument ID: F6	

PARAMETERS	Result (ug/L)	LOQ (ug/L)	DL (ug/L)	LOD (ug/L)
Aluminum	ND	100	10.0	20.0
Antimony	ND	1.00	0.250	0.500
Arsenic	ND	1.00	0.100	0.200
Barium	ND	1.00	0.250	0.500
Beryllium	ND	1.00	0.0500	0.100
Cadmium	ND	1.00	0.100	0.200
Calcium	61.8J	100	13.0	26.0
Chromium	0.140J	1.00	0.100	0.200
Cobalt	ND	1.00	0.100	0.200
Copper	ND	1.00	0.250	0.500
Iron	5.88J	100	5.00	10.0
Lead	ND	1.00	0.0500	0.100
Magnesium	9.54J	100	5.00	10.0
Manganese	0.194J	1.00	0.100	0.200
Nickel	ND	1.00	0.100	0.200
Potassium	ND	100	10.0	20.0
Selenium	ND	1.00	0.150	0.300
Silver	ND	1.00	0.100	0.200
Sodium	171	100	25.0	50.0
Thallium	ND	1.00	0.100	0.200
Vanadium	ND	1.00	0.250	0.500
Zinc	ND	20.0	5.00	10.0

Note: Detection limits are reported relative to sample result significant figures.

Sample Amount : 50ml	Final Volume:50ml
Prepared by : MCande/LVicto	Analyzed by:SKao\LVicto

Figure 2: TYPICAL LCS/LCD SUMMARY

EMAX QUALITY CONTROL DATA
LAB CONTROL SAMPLE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN PROJECT
BATCH NO. : YYMXXX
METHOD : SW6020A

MATRIX	WATER		% MOISTURE:NA
DILUTION FACTOR	1.00	1.00	1.00
SAMPLE ID	MBLK1W	LCS1W	LCD1W
LAB SAMPLE ID	IMM006WB	IMM006WL	IMM006WC
LAB FILE ID	F6M15087	F6M15088	F6M15089
DATE PREPARED	MM/DD/YY HH:MM	MM/DD/YY HH:MM	MM/DD/YY HH:MM
DATE ANALYZED	MM/DD/YY HH:MM	MM/DD/YY HH:MM	MM/DD/YY HH:MM
PREP BATCH	IMM006W	IMM006W	IMM006W
CALIBRATION REF	F6M15085	F6M15085	F6M15085

ACCESSION:

PARAMETERS	MBResult (ug/L)	SpikeAmt (ug/L)	LCSResult (ug/L)	LCSRec (%)	SpikeAmt (ug/L)	LCDResult (ug/L)	LCDRec (%)	RPD (%)	QCLimit (%)	MaxRPD (%)
Aluminum	ND	3000	3060	102	3000	3170	106	4	84-117	20
Antimony	ND	30	30.2	101	30	30.3	101	0	85-117	20
Arsenic	ND	30	29.0	97	30	28.4	95	2	84-116	20
Barium	ND	30	30.3	101	30	30.4	101	0	86-114	20
Beryllium	ND	30	28.7	96	30	28.1	94	2	83-121	20
Cadmium	ND	30	30.4	101	30	30.2	101	1	87-115	20
Calcium	75.7JE	3000	3150E	105	3000	3140E	105	0	87-118	20
Chromium	ND	30	29.6	99	30	29.4	98	1	85-116	20
Cobalt	ND	30	30.7	102	30	30.5	102	1	86-115	20
Copper	ND	30	29.6	99	30	29.5	98	0	85-118	20
Iron	7.20J	3000	3090	103	3000	3090	103	0	87-118	20
Lead	ND	30	33.6	112	30	32.5	108	3	88-115	20
Magnesium	6.53J	3000	3180	106	3000	3270	109	3	83-118	20
Manganese	0.147J	30	31.5	105	30	30.9	103	2	87-115	20
Nickel	ND	30	29.3	98	30	29.2	97	0	85-117	20
Potassium	ND	3000	3150	105	3000	3140	105	0	87-115	20
Selenium	ND	30	30.4	101	30	29.8	99	2	80-120	20
Silver	ND	30	26.9	90	30	27.4	91	2	85-116	20
Sodium	30.1J	3000	3320	111	3000	3300	110	1	85-117	20
Thallium	ND	30	33.2	111	30	32.2	107	3	82-116	20
Vanadium	ND	30	29.0	97	30	29.0	97	0	86-115	20
Zinc	ND	60	64.5	108	60	62.2	104	4	83-119	20

Figure 3: TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN PROJECT
BATCH NO. : YYMXXX
METHOD : SW6020A

MATRIX : WATER % MOISTURE: NA
DILUTION FACTOR: 1 1 1
SAMPLE ID : ABCDEF-02 ABCDEF-02MS ABCDEF-02MSD
LAB SAMPLE ID : MXXX-02 MXXX-02M MXXX-02S
LAB FILE ID : F6M15094 F6M15091 F6M15092
DATE PREPARED : MM/DD/YY HH:MM MM/DD/YY HH:MM MM/DD/YY HH:MM
DATE ANALYZED : MM/DD/YY HH:MM MM/DD/YY HH:MM MM/DD/YY HH:MM
PREP BATCH : IMM006W IMM006W IMM006W
CALIBRATION REF: F6M15085 F6M15085 F6M15085

ACCESSION:

PARAMETERS	PSResult (ug/L)	SpikeAmt (ug/L)	MSResult (ug/L)	MSRec (%)	SpikeAmt (ug/L)	MSDResult (ug/L)	MSDRec (%)	RPD (%)	QCLimit (%)	MaxRPD (%)
Aluminum	ND	3000	3000	100	3000	3030	101	1	84-117	20
Antimony	ND	30	28.5	95.0	30	29.9	99.7	5	85-117	20
Arsenic	ND	30	27.1	90.3	30	28.5	95.0	5	84-116	20
Barium	ND	30	28.9	96.3	30	30.2	101	4	86-114	20
Beryllium	ND	30	27.2	90.7	30	28.3	94.3	4	83-121	20
Cadmium	ND	30	28.7	95.7	30	30.1	100	5	87-115	20
Calcium	61.8J	3000	3220	105	3000	3120	102	3	87-118	20
Chromium	0.140J	30	28.4	94.2	30	29.3	97.2	3	85-116	20
Cobalt	ND	30	29.3	97.7	30	30.2	101	3	86-115	20
Copper	ND	30	28.3	94.3	30	29.0	96.7	2	85-118	20
Iron	5.88J	3000	2970	98.8	3000	3030	101	2	87-118	20
Lead	ND	30	32.1	107	30	32.6	109	2	88-115	20
Magnesium	9.54J	3000	3110	103	3000	3150	105	1	83-118	20
Manganese	0.194J	30	30.2	100	30	30.6	101	1	87-115	20
Nickel	ND	30	28.1	93.7	30	28.8	96.0	2	85-117	20
Potassium	ND	3000	3060	102	3000	3080	103	1	87-115	20
Selenium	ND	30	29.0	96.7	30	29.5	98.3	2	80-120	20
Silver	ND	30	25.4	84.7*	30	27.2	90.7	7	85-116	20
Sodium	171	3000	3310	105	3000	3360	106	1	85-117	20
Thallium	ND	30	31.8	106	30	32.5	108	2	82-116	20
Vanadium	ND	30	27.9	93.0	30	28.6	95.3	2	86-115	20
Zinc	ND	60	62.8	105	60	63.0	105	0	83-119	20

PSResult - Parent Sample Result
* Out of QC limit

Figure 4: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project: CLEAN PROJECT

SDG : YYMXXX

METHOD SW6020A
METALS BY ICP-MS

A total of five (5) water samples were received on MM/DD/YY to be analyzed for Metals by ICP-MS in accordance with Method SW6020A and project specific requirements.

Holding Time
Samples were digested and analyzed within the prescribed holding time.

Calibration
Initial Calibration was established as prescribed by the method and was verified using a secondary source(ICV). Interference checks were performed and results were within required limits. Continuing calibration verifications and continuing calibration blanks were carried out at the frequency specified by the project. All calibration requirements were satisfied. MRL was analyzed as required by the project.

Method Blank
Method blank was prepared and analyzed at the frequency required by the project. For this SDG, one (1) method blanks were analyzed. Iron(7.20J <1/2 LOQ), Magnesium(6.53J <1/2 LOQ), Manganese(0.147J <1/2 LOQ) and Sodium(30.1J <1/2 LOQ) were detected at trace level in IMM006WB. Refer to sample result summary forms for details.

Lab Control Sample
Lab control sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of LCS/LCD was analyzed. All analytes were within LCS QC limits in IMM006WL/IMM006WC Refer to LCS summary forms for details.

Matrix QC Sample
Matrix spike sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of MS/MSD was analyzed and the following was noted: MXXX-02M/S - Percent recovery for Silver was not within QC limits in MS but was within QC limits in MSD. The rest of the analytes were in control. Analytical spike was analyzed and evaluated as appropriate. Results were within expected values. Refer to Matrix QC summary form for details.

Sample Analysis
Samples were analyzed according to prescribed analytical procedures. Results were evaluated in accordance to project requirements. For this SDG, all quality control requirements were met with the exception of those that were discussed within the associated QC parameter.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURES	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
Tune Check (Mass calibration and resolution check)	Daily before ICAL.	±0.10 AMU (Mass of Isotope) <0.9 AMU full width resolution RSD of 4 replicates : ≤5%	Correct problem and repeat tune check.		
Initial Calibration (multi-point)	Daily initial calibration prior to sample analysis.	r ≥ 0.998	Correct the problem and repeat the initial calibration.		
Initial Calibration Verifications (ICV) Second Source	Daily after the initial calibration.	All analytes within ±10% of expected value RSD of Replicate integrations: < 5%	Correct the problem and repeat the initial calibration.		
Low Level Calibration Verification (LLICV / LLCCV)	LLICV: Daily after initial calibration. LLCCV: At the end of the analysis sequence	All analytes with ± 30% of expected value.	Correct the problem and repeat the initial calibration.		
Calibration Verifications (CCV)	Daily before sample analysis, after every 10 samples and at the end of the analysis sequence.	All analytes within ±10% of expected value. RSD of replicate integrations < 5%.	Repeat calibration and reanalyze all samples since last successful calibration.		
Calibration Blanks (ICB/CCB)	After every calibration verification	All target analytes < ½ LOQ.	Correct problem then reanalyze calibration blank and previous samples.		
Interference Check Sample (ICSA/ICSAB)	Analyze at the beginning of each analytical run or once every 12 hours, whichever is more frequent.	Within ±20% of expected value	Terminate analysis, correct the problem, reanalyze ICS, and reanalyze all affected samples		
Internal Standard (IS)	ICV, LLICV, CCV, LLCCV, CCBs, MB, LCS, every sample	IS Intensities > 70% from Initial Calibration Blank IS Intensity	Correct problem then re-analyze		
Method Blank	One per preparation batch	All target analytes < ½ LOQ.	Re-digest and reanalyze method blank and all samples processed with the contaminated blank.		
Laboratory Control Sample (LCS)	One per preparation batch	% Recovery: 80% - 120%	Re-digest and reanalyze LCS and all associated samples		
Matrix Spikes (MS/MSD)	One MS/MSD every 20 project samples per matrix	% Recovery: 75% - 125% RPD ≤20%	Evaluate post spike and dilution test: • If parent sample result is "ND", evaluate post spike. • If parent sample result is high (i.e., 4x of spike concentration) and post spike failed, evaluate dilution test.		
Post Digestion Spike Addition	When MS fails.	Recovery within 80-120% of expected value	Correct the problem then reanalyze post digestion spike addition		
Dilution Test (SX)	When MS fails.	1:5 dilution must agree within ±10% of the original determination	Evaluate. Discuss in case narrative.		
Instrument Detection Limit (IDL)	Every three months		Correct the problem and repeat the IDL determination.		
Comments: Refer to PSR for flagging criteria.			Reviewed By:		
			Date:		

Revision approved by: 06-06-19
Caspar Pang, Laboratory Director (Sign/Date)

06 06 19
Kenette Pimentel, QA Manager (Sign/Date)

Appendix 2: DEMONSTRATION OF CAPABILITY

MATRIX: WATER

PARAMETER	18F6J16019	18F6J16020	18F6J16023	18F6J16024	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	Accuracy Acceptance Limits (% Rec)	RSD (%) Criteria	COMMENTS
	IMJ017WL	IMJ017WC	IMJ017WX	IMJ017WY								
Aluminum	3164	3137	3258	3033	3000	3148	105	92.6	3	80 - 120	≤ 20	Passed
Antimony	31.1	30.1	32.0	30.1	30	30.8	103	0.890	3	80 - 120	≤ 20	Passed
Arsenic	31.2	30.5	32.2	30.4	30	31.1	104	0.845	3	80 - 120	≤ 20	Passed
Barium	31.3	30.5	32.1	30.3	30	31.0	103	0.812	3	80 - 120	≤ 20	Passed
Beryllium	32.6	31.6	34.6	32.0	30	32.7	109	1.318	4	80 - 120	≤ 20	Passed
Boron	32.3	31.8	34.1	31.5	30	32.4	108	1.166	4	80 - 120	≤ 20	Passed
Cadmium	31.9	30.8	32.6	30.7	30	31.5	105	0.939	3	80 - 120	≤ 20	Passed
Calcium	3193	3144	3353	3115	3000	3201	107	106.2	3	80 - 120	≤ 20	Passed
Chromium	32.8	32.1	33.9	31.9	30	32.7	109	0.924	3	80 - 120	≤ 20	Passed
Cobalt	33.1	33.3	34.6	32.3	30	33.3	111	0.940	3	80 - 120	≤ 20	Passed
Copper	32.9	32.3	34.0	32.0	30	32.8	109	0.904	3	80 - 120	≤ 20	Passed
Iron	3180	3096	3301	3075	3000	3163	105	102.6	3	80 - 120	≤ 20	Passed
Lead	33.7	32.2	34.5	32.4	30	33.2	111	1.079	3	80 - 120	≤ 20	Passed
Lithium	31.9	30.5	33.4	31.0	30	31.7	106	1.248	4	80 - 120	≤ 20	Passed
Magnesium	3133	3104	3253	3007	3000	3124	104	101.3	3	80 - 120	≤ 20	Passed
Manganese	33.5	33.6	35.0	32.6	30	33.7	112	0.981	3	80 - 120	≤ 20	Passed
Molybdenum	30.5	29.3	30.9	29.1	30	29.9	100	0.899	3	80 - 120	≤ 20	Passed
Nickel	32.9	32.4	34.2	32.3	30	32.9	110	0.904	3	80 - 120	≤ 20	Passed
Phosphorous	282	280	292	272	300	281	94	7.97	3	80 - 120	≤ 20	Passed
Potassium	3249	3156	3305	3128	3000	3210	107	82.0	3	80 - 120	≤ 20	Passed
Selenium	31.9	31.0	33.2	31.3	30	31.8	106	0.994	3	80 - 120	≤ 20	Passed
Silicon	3012	3023	3161	3022	3000	3055	102	71.2	2	80 - 120	≤ 20	Passed
Silver	32.2	31.1	32.6	31.1	30	31.7	106	0.753	2	80 - 120	≤ 20	Passed
Sodium	3087	3031	3224	3002	3000	3086	103	98.5	3	80 - 120	≤ 20	Passed
Strontium	33.3	31.9	33.6	32.2	30	32.8	109	0.842	3	80 - 120	≤ 20	Passed
Thallium	33.8	32.3	34.3	32.5	30	33.2	111	0.956	3	80 - 120	≤ 20	Passed
Tin	31.7	30.4	32.3	30.5	30	31.2	104	0.926	3	80 - 120	≤ 20	Passed
Titanium	32.2	31.9	33.3	31.5	30	32.2	107	0.767	2	80 - 120	≤ 20	Passed
Tungsten	26.4	25.6	27.2	25.8	30	26.2	87	0.708	3	80 - 120	≤ 20	Passed
Uranium	32.7	31.4	33.0	31.5	30	32.2	107	0.829	3	80 - 120	≤ 20	Passed
Vanadium	31.3	30.6	32.5	30.7	30	31.3	104	0.881	3	80 - 120	≤ 20	Passed
Zinc	68.7	67.3	71.3	65.5	60	68.2	114	2.443	4	80 - 120	≤ 20	Passed
Zirconium	29.5	29.1	30.6	28.7	30	29.5	98	0.830	3	80 - 120	≤ 20	Passed

Appendix 2 (Cont.): DEMONSTRATION OF CAPABILITY

MATRIX: SOIL

PARAMETER	18F6J09022	18F6J09023	18F6J09024	18F6J09025	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	Accuracy Acceptance Limits (% Rec)	RSD (%) Criteria	COMMENTS
	IMJ014SL	IMJ014SC	IMJ014SX	IMJ014SY								
Aluminum	1924	1983	1952	1938	2000	1949	97	25.2	1	80 - 120	≤ 20	Passed
Antimony	19.3	19.7	19.6	19.5	20	19.5	97	0.178	1	80 - 120	≤ 20	Passed
Arsenic	19.1	19.7	19.3	19.1	20	19.3	97	0.308	2	80 - 120	≤ 20	Passed
Barium	19.7	20.3	20.0	20.0	20	20.0	100	0.226	1	80 - 120	≤ 20	Passed
Beryllium	20.3	20.9	20.7	20.3	20	20.5	103	0.268	1	80 - 120	≤ 20	Passed
Boron	20.7	21.5	21.3	21.0	20	21.1	106	0.335	1.6	80 - 120	≤ 20	Passed
Cadmium	19.8	20.1	20.0	20.0	20	20.0	100	0.145	1	80 - 120	≤ 20	Passed
Calcium	2031	2086	2048	2053	2000	2054	103	23.0	1	80 - 120	≤ 20	Passed
Chromium	20.2	20.8	20.3	21.2	20	20.6	103	0.463	2	80 - 120	≤ 20	Passed
Cobalt	20.9	21.6	21.1	21.1	20	21.2	106	0.263	1	80 - 120	≤ 20	Passed
Copper	22.0	22.4	22.0	21.9	20	22.1	110	0.221	1	80 - 120	≤ 20	Passed
Iron	2032	2090	2074	2071	2000	2067	103	24.83	1	80 - 120	≤ 20	Passed
Lead	20.2	20.8	20.4	20.2	20	20.4	102	0.284	1	80 - 120	≤ 20	Passed
Lithium	20.4	21.1	20.6	20.3	20	20.6	103	0.349	2	80 - 120	≤ 20	Passed
Magnesium	1924	1990	1940	1938	2000	1948	97	28.6	1	80 - 120	≤ 20	Passed
Manganese	21.3	21.7	21.4	21.9	20	21.6	108	0.282	1	80 - 120	≤ 20	Passed
Molybdenum	19.1	19.6	19.4	19.3	20	19.3	97	0.205	1	80 - 120	≤ 20	Passed
Nickel	20.5	20.9	20.6	21.9	20	21.0	105	0.655	3	80 - 120	≤ 20	Passed
Phosphorous	202	209	206	207	200	206	103	2.90	1	80 - 120	≤ 20	Passed
Potassium	2049	2113	2068	2096	2000	2082	104	28.5	1	80 - 120	≤ 20	Passed
Selenium	19.3	20.0	19.5	19.6	20	19.6	98	0.293	1	80 - 120	≤ 20	Passed
Silver	21.0	21.5	21.2	21.2	20	21.2	106	0.202	1	80 - 120	≤ 20	Passed
Sodium	2018	2091	2069	2061	2000	2060	103	30.8	1	80 - 120	≤ 20	Passed
Strontium	20.5	21.1	20.6	20.5	20	20.7	103	0.308	1	80 - 120	≤ 20	Passed
Thallium	20.4	20.9	20.8	20.6	20	20.7	103	0.205	1	80 - 120	≤ 20	Passed
Tin	21.8	22.6	22.2	22.1	20	22.2	111	0.309	1	80 - 120	≤ 20	Passed
Titanium	20.1	20.6	20.3	20.3	20	20.3	102	0.179	1	80 - 120	≤ 20	Passed
Tungsten	18.9	19.9	19.9	19.7	20	19.6	98	0.461	2.3	80 - 120	≤ 20	Passed
Uranium	20.4	21.0	20.8	20.4	20	20.6	103	0.270	1	80 - 120	≤ 20	Passed
Vanadium	19.7	20.3	19.8	19.9	20	19.9	100	0.264	1	80 - 120	≤ 20	Passed
Zinc	40.0	41.8	40.8	40.5	40	41	102	0.745	2	80 - 120	≤ 20	Passed
Zirconium	14.3	14.9	14.6	14.7	15	14.6	97	0.250	2	80 - 120	≤ 20	Passed

Appendix 3: COMPARATIVE STUDY OF MODIFIED 3050B

ANALYTICAL METHOD: SW 6020A
PREPARATION BATCH: IMG031S - Modified Method 3050B
IMG032S - Reference Method 3050B
ANALYTICAL BATCH: I98H01
QC STANDARD: 09G281-02

Preparation Date: 7/27/2010
Extracted by: M. Mendoza
Analytical Run Date: 8/3/2010
Analyzed by: C. Capulong

COMPOUND	TRUE VALUES (mg/Kg)	ACCEPTANCE LIMITS (mg/Kg)	RECOVERY LIMITS (%)	REFERENCE METHOD 3050B				MODIFIED METHOD 3050B			
				Concentration (mg/Kg)		Recovery		Concentration (mg/Kg)		Recovery	
				98H01041 G281-02	98H01043 G281-02D	98H01041 G281-02	98H01043 G281-02D	98H01030 G281-02	98H01032 G281-02D	98H01030 G281-02	98H01032 G281-02D
Aluminum	7320	4940 - 16400	67 - 224	10900	10700	149%	146%	10700	10700	146%	146%
Antimony	110	11.0 - 121	10 - 110	20.0	19	18%	17%	61.4	66.6	56%	61%
Arsenic	84.2	42.8 - 92.7	51 - 110	61.2	61.7	73%	73%	63.6	65.3	76%	78%
Barium	247	186 - 318	75 - 129	255	260	103%	105%	275	273	111%	111%
Beryllium	49.0	29.0 - 53.9	59 - 110	39.7	39.6	81%	81%	42	41.7	86%	85%
Boron	130	61.8 - 149	48 - 115	104	104	80%	80%	126	126	97%	97%
Cadmium	76.5	48.7 - 84.4	64 - 110	65.3	66.7	85%	87%	69.9	69.8	91%	91%
Calcium	10200	7860 - 12900	77 - 126	9360	10700	92%	105%	10200	10300	100%	101%
Chromium	116	74.8 - 140	64 - 121	99.5	98.4	86%	85%	107	110	92%	95%
Cobalt	64.6	47.2 - 79.7	73 - 123	62.1	61.3	96%	95%	63.8	64	99%	99%
Copper	143	99.7 - 166	70 - 116	116	115	81%	80%	127	126	89%	88%
Iron	23300	11500 - 36200	49 - 155	23900	23700	103%	102%	24800	24800	106%	106%
Lead	82.0	41.9 - 90.2	51 - 110	63.3	64.9	77%	79%	67.5	67.6	82%	82%
Magnesium	7400	5300 - 9060	72 - 122	6770	6760	91%	91%	6930	7100	94%	96%
Manganese	475	428 - 724	90 - 152	546	562	115%	118%	584	571	123%	120%
Molybdenum	66.1	36.0 - 72.7	54 - 110	54.9	53.5	83%	81%	60.1	59.4	91%	90%
Nickel	144	88.8 - 158	62 - 110	112	113	78%	78%	122	122	85%	85%
Potassium	3740	2360 - 4990	63 - 133	3620	3600	97%	96%	3690	3560	99%	95%
Selenium	189	97.2 - 208	51 - 110	142	148	75%	78%	154	157	81%	83%
Silver	40.6	21.2 - 44.6	52 - 110	30.9	32.7	76%	81%	34.2	34.9	84%	86%
Sodium	200	31.4 - 324	16 - 162	292	197	146%	99%	192	200	96%	100%
Strontium	135	88.1 - 162	65 - 120	123	121	91%	90%	129	128	96%	95%
Thallium	127	73.2 - 144	58 - 113	110	114	87%	90%	117	112	92%	88%
Tin	250	39.5 - 275	16 - 110	71.2	73.2	28%	29%	84.7	85.2	34%	34%
Titanium	609	426 - 792	70 - 130	737	744	121%	122%	772	760	127%	125%
Uranium	1.12	0.784 - 1.46	70 - 130	0.93	1.1	83%	98%	1.03	1.01	92%	90%
Vanadium	61.8	55.6 - 123	90 - 199	90.2	89.7	146%	145%	92.8	93.1	150%	151%
Zinc	208	145 - 270	70 - 130	208	207	100%	100%	225	214	108%	103%

** Note: Modified 3050B procedure is specified in EMAX-3050 Rev. 4*

6020FS: SAMPLE PREPARATION LOG

EMAX LABORATORIES, INC.

DIGESTION LOG
for
ICP-MS

Page 1

Note: For samples, relevant QCs/Standards digested, refer to attached digestion sequence.

Comments:

Book #: EIM-033
Batch:
Matrix:
Digestor ID:

SOP #	Rev. #
☐ EMAX-200.8	
☐ EMAX-6020	
☐ EMAX-	

Start	Temp °C	End	Temp °C

Standards	ID	Amount Added (ml)
LCS-1		
LCS-2		
MS		
Reagent	**Lot# / ID**	**Amount Added (ml)**
HNO_3		
HCl		
H_2O_2		
HNO3(1:1)		

Digestate Location	
Extract Location	

☐ Soil Samples: -diluted 1 ml of digestate to 10 ml reagent water (DF=100)

☐ Reagent Water ID:

☐ Thermometer ID:

☐ Pipette ID:

☐ Pipette ID:

☐ Pipette ID:

☐ HNO_3 dispenser checked @ 5.0 ml with Class A volumetric flask

☐ HCl dispenser checked @ 5.0 ml with Class A volumetric flask

Prepared By:

Standard Added By: Witnessed By:

Extract Rcvd By: Checked By:

6020FA: ANALYSIS RUN LOG

EMAX LABORATORIES, INC.

ANALYSIS RUN LOG
for
ICP-MS

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Start Date:

End Date:

Comments:

Book #: A98-030

Instrument No.: 98

Analytical Batch:

Analytical Sequence:

Method File:

SOP #	Rev. #
☐ EMAX-6020	
☐ EMAX-200.8	
☐ EMAX-	

STANDARDS ID	
S0	
S1	
S2	
S3	
S4	
S5	
S6	
S7	
ICV	
CCV	
ICSA	
ICSAB	
6020 TUNE SOLN.	
200.8 TUNE SOLN.	
MRL1	
MRL2	
MRL3	

Analyzed By:

Date:

6020FM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

ICP-MS MAINTENANCE LOG

Instrument No: 98 Month: Year: 201_ Book # M98-007

Date							
Argon, Hydrogen, Helium							
Instrument Discharge							
Pump Tubings							
Rinse Bottle							
Printer Paper							
Autosampler							
Cooling Water							
Peri Pump Rate (0.08-0.12 RPS)							
Nebulizer Gas (0.8-1.30 L/min)							
Makeup Gas (0.1-0.3 L/min)							
ICP-MS TUNE							
Li - 7 (≥ 6400 Counts)							
Y - 89 (≥ 16000 Counts)							
Tl - 205 (≥ 9600 Counts)							
Oxides (MO^+) ≤ 2%							
Double Charge (M^{++}) ≤ 2%							
Analyst Initials							
ICP-MS TUNE							
Problem(s) Encountered							
Corrective Action							
Return To Specs (Analyst Initials)							

Code	Problems Encountered	Code	Corrective Action

EMAX
LABORATORIES, INC.
1835 W. 205th Street
Torrance, CA 90501
Tel: (310) 618-8889

Page 1 of 1

ADDENDUM TO	
Document	ALL ANALYTICAL SOPS
Revision Number	CURRENT REVISIONS
Section	10.2 Instrument Parameters
Date	11 September 2019
Reference Number	AA.7

This applies to all GC/MS, GC, HPLC-MS, HPLC, ICP-MS, ICP and IC Method SOPs

Section 10.2 shall include:

10.2. **Instrument Parameters**

10.2.1. Instrument parameters setup stipulated in the SOPs, are instrument suggested parameters. Fine tune the instrument to obtain optimum instrument condition.

10.2.2. Print and staple a copy of the current instrument parameter on the instrument log for easy access when performing daily instrument routine check.

10.2.3. In the event that instrument parameters necessitate a change, replace the instrument parameter printout with the new parameter setup. Archive the previous instrument parameters at the back of the instrument maintenance log.

PREPARED BY: _____ Date: 9/11/19

Name Farina Madamba

Title QA / QC Coordinator

APPROVED BY: _____ Date: 09.11.19

Name Kenette Pimentel

Title QA Manager

APPROVED BY: _____ Date: 09-11-19

Name Caspar Pang

Title Laboratory Director

STANDARD OPERATING PROCEDURES

MERCURY IN LIQUID WASTE

SOP No.:	EMAX-7470	Revision No.	8	Effective Date:	13-Jul-16
Prepared By:	Mary Jane Mendoza			Date:	07,12,16
Approved By:	Kenette Pimentel QA Manager			Date:	07·12·16
Approved By:	Caspar Pang Laboratory Director			Date:	07-12-16

Control Number: 7470-08-

1.0 SCOPE AND APPLICATION

1.1. This procedure applies to the measurement of Mercury in aqueous wastes, leachates, and wastewater samples by Cold Vapor Absorption Technique.

1.2. This SOP is an adaptation of SW846 Methods 7470A.

2.0 SUMMARY OF METHOD

2.1. A representative amount of sample is digested in nitric and sulfuric acids, followed by oxidation with potassium permanganate and potassium persulfate.

2.2. Organic mercurial are broken down and converted into mercuric ions in order to respond to the cold vapor atomic absorption technique. Persulfate oxidation step, followed by addition of permanganate ensures that organo-mercury compounds are oxidized.

2.3. Absorption of radiation by mercury vapor at 253.7 nm is then measured in the digested samples.

2.4. **Interferences**

2.4.1. Sulfides, as sodium sulfide, Copper and Chloride at high concentrations are known to interfere with the recovery of mercury. Samples containing such interference may require additional permanganate (about 12.5 ml).

2.4.2. Care must be taken to ensure that free chlorine is absent before the mercury is swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent.

2.4.3. Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL), Limit of Detection (LOD) and Limit of Quantitation (LOQ)**

3.1.1. Refer to EMAX-QA04 for generation, validation and verification of DL, LOD and LOQ.

3.2. **Established DL, LOD & LOQ**

PARAMETER	DL	LOD	LOQ	Unit
Water	0.054	0.1	0.50	µg/L

4.0 DYNAMIC RANGE

4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (see Section 9.6). All samples analyzed above this range are considered "over-range" and requires dilution to properly quantitate.

4.2. The lowest quantifiable range of diluted samples is equal to the concentration of the lowest calibration point. All diluted samples analyzed below this range are considered as "under-range" and requires lower dilution factor to properly quantitate.

5.0 SAMPLE HOLDING TIME AND PRESERVATION

5.1. **Sample Preservation**

5.1.1. Samples are expected to be contained in HDPE pre-cleaned containers and preserved with HNO3 to pH < 2.

5.2. **Holding Time**

5.2.1. Samples must be analyzed within 28 days from date of collection.

6.0 ASSOCIATED SOPs

6.1. EMAX-DM01 Data Flow and Review

6.2. EMAX-QA04 Detection Limit (DL)

6.3. EMAX-QA05 Training

6.4. EMAX-QA08 Corrective Action

6.5. EMAX-QC01 Quality Control of Chemicals

6.6. EMAX-QC02 Analytical Standard Preparation

6.7. EMAX-QC06 Volumetric Labware and Micropipette Verification

6.8. EMAX-QC07 Glassware Cleaning

6.9. EMAX-SM03 Waste Disposal

6.10. EMAX-SM04 Analytical and QC Sample Labeling

7.0 SAFETY

7.1. Read all SDS of chemicals listed in this SOP.

7.2. Treat reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves at all times when performing this procedure. Perform preparation and analysis of mercury in a fume hood equipped with an exhaust fan or blower.

7.3. If for any reason, sample and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amounts of water. If irritations persist inform your supervisor immediately so that proper action can be taken.

7.4. Do not look directly at the Mercury Lamp while lit. The radiation may cause damage to your eyes.

7.5. Perform all reagent additions under a fume hood.

7.6. Mercury Analyzers are to be used by trained personnel only.

8.0 INSTRUMENTS, CHEMICALS AND REAGENTS

8.1. **Instruments and Supplies**

8.1.1. Mercury Analyzers

8.1.1.1. Leeman PS-200 Automated Mercury Analyzer with Autosampler, Computer, Printer and PS 200 Software, Win Hg Runner 1.3

8.1.1.2. Leeman Hydra AA Automated Analyzer with Autosampler, Computer, Printer and PS200 Software, Win Hg Runner 1.3

8.1.2. 100 ml Digestion vessel

8.1.3. Digestion block or equivalent

8.1.4. Micropipettes and tips

8.1.5. Thermometer

8.2. **Chemicals and Reagents**

8.2.1. Where available, purchase reagent grade chemicals and reagents

8.2.2. Sulfuric Acid, concentrated

8.2.3. Nitric Acid, concentrated

8.2.4. Sodium chloride - hydroxylamine hydrochloride solution: Dissolve 120 g of sodium chloride and 120 g of hydroxylamine hydrochloride in reagent water and dilute to 1 L. *(Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.)*

8.2.5. Potassium Permanganate, 5% solution: Dissolve 50 g of potassium permanganate in 1 L reagent water.

8.2.6. Potassium Persulfate, 5% solution: Dissolve 50 g of potassium persulfate in 1 L reagent water.

8.2.7. Stannous Chloride – 10% solution: Dissolve 200 g of $SnCl_2$ in reagent water, add 200 ml HCl and volume to 2 L.

8.2.8. Reagent water – Mercury-free water

9.0 STANDARDS

9.1. Refer to EMAX-QC02 for proper analytical standard preparations.

9.2. Other concentration levels may be prepared to meet the data quality objective of a project.

9.3. **Stock Standard**

9.3.1. Purchase stock standards as certified solutions from two different vendors. Use one as primary standard and the other as secondary standard.

Stock Std	Name	Source	Conc.	Used for
Primary	Mercury	ERA or equivalent	1000 mg/L	ICAL, CCV
Secondary	Mercury	AccuStandard or equivalent	1000 mg/L	ICV/LCS/MS

9.3.2. Transfer standards on a properly labeled inert vial with minimal headspace and store it at -10°C to -20°C.

9.4. **Intermediate Standard Solution**

9.4.1. Primary Intermediate Standard, 10.0 mg/L: Dilute 1.0 mL of 1000 mg/L Hg primary stock standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.4.2. Secondary Intermediate Standard, 10.0 mg/L: Dilute 1.0 mL of 1000 mg/L Hg secondary stock standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.4.3. Post Spike Standard, 100 µg/L: Dilute 1.0 mL of 10 mg/L Hg primary intermediate standard to 100 mL using reagent water. Digest prior to use.

9.5. **Working Standard**

9.5.1. Primary Working Standard, 50 µg/L: Dilute 0.50 mL of 10 mg/L primary intermediate standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.5.2. Secondary Working Standard, 50 µg/L: Dilute 0.50 mL of 10 mg/L secondary intermediate standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.6. **Initial Calibration Standards**

9.6.1. Initial calibration consists of five standards and a reagent water blank. Using the primary working standard, prepare the ICAL points in reagent water, as follows:.

Level	Primary Working Standard, 100 µg/L Aliquot (ml)	Final Volume (mL	Concentration (µg/L)
S0	0	50	0
S1	0.2	50	0.2
S2	0.5	50	0.5
S3	1.0	50	1.0
S4	2.0	50	2.0
S5	5.0	50	5.0

Note: Other concentration levels may be prepared as appropriate to meet project quality objectives.

9.7. **Initial Calibration Verification Standard (ICV)**

9.7.1. Prepare ICV at 2 µg/L by diluting 2mL of secondary working standard (50 µg/L) to final volume of 50 mL using reagent water

9.8. **Continuing Calibration Verification Standard (CCV)**

9.8.1. Prepare CCV at 2 µg/L by diluting 2 mL of primary working standard (50 µg/L) to final volume of 50 mL using reagent water.

9.9. **Laboratory Control Standard (LCS) / Matrix Spike (MS) Standard**

9.9.1. Spike 2.5mL of secondary working standard (50 µg/L) to 50 mL reagent water (for LCS) or designated sample (for MS).

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Transfer 50 mL of sample into 100 ml digestion vessel. Use reagent water for method blank, LCS, and calibration standards. For STLC and TCLP extracts, use 5 ml sample volume diluted with reagent water to 50 ml. (The reduction of the volume is due to waste minimization.)

10.1.2. Prepare LCS/MS (Sec. 9.9.1). Spike appropriate standards for ICAL standards (Sec. 9.6.1), ICV (Sec. 9.7.1) and CCV (Sec. 9.8.1).

10.1.2.1. **Spike Addition**. Call a witness. Have the witness verify the setting of the micropipette and the expiration date of the spike standard.

10.1.3. Add 2.5 mL of concentrated H_2SO_4 and 1.25 mL concentrated HNO_3 with mixing after each addition.

10.1.4. Add 7.5 mL of 5% $KMnO_4$ solution to each vessel.

10.1.5. Swirl each vessel to mix and let it stand by for 15 min. Check each vessel if purple color persist. If not, add permanganate solution at 2.5 mL increments (maximum total amount of 12.5 mL) swirling the Digestion vessel at every addition, until purple color persists.

Add the same amount of permanganate solution added to a sample, to the method blank, LCS, calibration standards and calibration verification standards.

10.1.6. Add 4 mL of 5% potassium persulfate. Heat for 2 hours in hot block maintained at 95 ± 3°C.

10.1.7. Allow the samples to cool.

10.1.8. Add 3 mL NaCl-hydroxylamine hydrochloride solution and wait for the sample to decolorize. Additional NaCl-hydroxylamine hydrochloride solution may be added to reduce excess permanganate in 1 mL increments up to a maximum total amount of 5 mL.

Add the same amount of NaCl-hydroxylamine hypochloride solution added to the method blank, LCS, calibration standards and calibration verification standards.

10.1.9. Upon decolorization, dilute to 80 mL final volume using reagent water.

10.1.10. Properly fill up the sample preparation log.

10.2. **Instrument Parameters**

10.2.1. PROTOCOL

10.2.1.1. Set Values

Instrument ID:	PS200	HYDRA AA
Number of Integration	1	
Uptake time	20 sec.	18 sec.
Weight	N	N
Dilution	N	N
On/Off, times, gains		
On	Y	Y
Time	10	10
Gas	0.35 LPM	0.15 LPM
Pump Rate	5 ml/min	7 ml/min
AUTOSAMPLER – Setup		
Station 1 (rack1)	From cup 1 to cup 44	From cup 1 to cup 44
Station 2 (rack 2)	From cup 1 to cup 44	From cup 1 to cup 44
Rinse time	50 sec.	60 sec.

CALIBRATION S0, S1, S2, S3, S4, S5	Concentration, µg/L 0 0,.20, 0.50, 1.0, 2.0, 5.0	Concentration, µg/L 0 0,.20, 0.50, 1.0, 2.0, 5.0

10.2.2. DATA OUTPUT – Specify Report

Data Output	Real Time	Post Run
Samples	Y	Y
Standards	Y	Y
Updates	Y	Y
Peaks	N	N
IEC Stds.	N	N
Check Stds.	Y	Y
Dups and % Diff.	Y	Y
Wavelength	N	N
Rel. Absorbances	N	N
% RSD	Y	Y
Scans to PRN		N
Detail		Y
Summary		N
Post Run Copies		1
Post Run Report Order [1-sorted; 2- sequential]	2	

10.3. **Calibration**

10.3.1. Instrument Set-up

10.3.1.1. Set up PS200 or Hydra AA to proper operating parameters. Refer to Sec. 10.2.1.

New pump tubing, must be ran with rinse for 45 minutes to break in the tubing.

10.3.1.2. Turn on the lamp and allow to warm up for at least 5 minutes.

10.3.1.3. Check the peristaltic pump to deliver a steady flow.

10.3.1.4. Check that the reductant solution, 10% $SnCl_2$, is sufficient. If not, prepare solution as described in Section 8.2.7.

10.3.2. Initial Calibration (ICAL)

10.3.2.1. Prepare initial calibration solution as described in Sec. 9.6.1.

10.3.2.2. Digest the ICAL standards as described in Sec. 10.1.

10.3.2.3. Analyze them as described in Sec. 10.4.

10.3.2.4. Refer to Sec. 10.5 for calculation.

10.3.2.5. Initiate initial calibration as described in the instrument operations manual. After calibration is completed, acquire the calibration data and review.

10.3.2.6. Refer to Appendix 1 for acceptance criteria.

10.3.2.7. Verify the initial calibration by a secondary source standard (ICV).

10.3.3. Initial Calibration Verification (ICV)

10.3.3.1. Prepare ICV as described in Sec. 9.7.1. Prepare ICB using reagent water.

10.3.3.2. Digest ICV and ICB as described in Sec. 10.1.

10.3.3.3. Analyze the ICV sample to verify the concentration of the ICAL. Analyze the ICB after the ICV.

10.3.3.4. Refer to Appendix 1 for acceptance criteria.

10.3.4. Continuing Calibration Verification (CCV)

10.3.4.1. Prepare CCV as described in Sec. 9.8.1. Prepare CCB using reagent water.

10.3.4.2. Digest the CCV and CCB as described in Sec. 10.1.

10.3.4.3. Analyze CCV sample to verify the validity of ICAL. Analyze the CCB after the CCV.

10.3.4.4. Refer to Appendix 1 for acceptance criteria.

10.4. **Analysis**

10.4.1. Calibration

10.4.1.1. Refer to the instrument operations manual for proper calibration and analytical sequence setup (autosampler setup).

10.4.1.2. Analytical batch ID naming convention: MIIMSSS

where:

"M" – is for Mercury and is always the first character

II – is the instrument number

M – is the month code (A for January, B for February, and so on)

SSS – is a sequential number (resets to 001 for the first folder created each month)

10.4.1.3. **Typical Calibration Sequence**

S0	0.00000
S1	0.20000
S2	0.50000
S3	1.00000
S4	2.00000
S5	5.00000
S6	10.0000

10.4.2. Analytical Sequence

10.4.2.1. ICV

10.4.2.2. ICB

10.4.2.3. CCV1

10.4.2.4. CCB1

10.4.2.5. Method Blank (MB)

10.4.2.6. Lab Control Sample (LCS)

10.4.2.7. Lab Control Sample Duplicate (LCSD)

10.4.2.8. Post Digestion Spike (Sec. 10.4.4)

10.4.2.9. Parent Sample

10.4.2.10. Serial Dilution (Sec. 10.4.5)

10.4.2.11. Matrix Spike (MS)

10.4.2.12. Matrix Spike Duplicate (MSD)

10.4.2.13. Maximum of 2 samples

10.4.2.14. CCV2

10.4.2.15. CCB2

10.4.2.16. Maximum of 10 samples

10.4.2.17. CCV3

10.4.2.18. CCB3

10.4.3. Using the analytical sequence, arrange the digested standards and samples to be analyzed chronologically.

10.4.4. Transfer about 6 ml of the digestates into the autosampler tubes placing them on the autosampler rack in the same order as the analytical sequence.

10.4.5. Prepare a Dilution Test sample at 5x dilution. Pipette 2 mL of sample, add 8 mL of S0 into a sample tube. Seal the tube with parafilm and invert the tube several times to ensure adequate mixing.

10.4.6. Prepare a Post Digestion Spike test sample. Using 10 mL unspiked sample digestate (preferably the QC sample), add 0.3 mL Post Spike standard (Sec. 9.4.3).

10.4.7. Set the prepared samples into the autosampler and start the analytical run.

10.4.8. Sample Result Evaluation

10.4.8.1. Check the QC data as soon as available.

- ✓ Check the ICAL and ICV prior to sample analysis.
- ✓ Check the method blank and LCS against project specific requirements.
- ✓ Check the matrix QC sample that it meets the acceptance criteria or note possible matrix interference in the run log.
- ✓ Check that all samples are analyzed within the calibration range. Dilute the samples that measured over the absorbance of the highest calibration point.
- ✓ Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.
- ✓ Check the calibration checks to make sure that the instrument is functioning properly.

10.4.8.2. Refer to Sec. 11 for quality control and Sec. 12 for corrective action.

10.4.8.3. Refer to Appendix 1 for acceptance criteria. If any of the acceptance criteria is not met, perform the necessary corrective action. If problem persists, inform the Supervisor for further action.

10.4.8.4. Properly fill up the analytical run log.

10.4.8.5. Upload the electronic data to the network.

10.4.9. Dealing with Carryover

10.4.9.1. Check the sample analyzed preceded by another sample found to have target analyte concentrations exceeding the calibration range.

10.4.9.2. If the target analyte is not detected, as found in the preceding high concentration sample, proceed with data reduction.

10.4.9.3. If the target analyte is detected, as found in the preceding high concentration sample, re-analyze the sample to rule-out carryover. If carryover is confirmed, proceed with data reduction and report the data from re-analysis.

10.4.10. Method of Standard Addition (MSA)

10.4.10.1. Perform MSA for all EP extracts, samples for de-listing petition, whenever a new matrix is encountered and/or as indicated above.

10.4.10.2. Prepare three sample solutions (Ms1, Ms2, Ms3) to objectively produce equal increments of concentration in the final solution without diluting the sample more than 50% of its original volume and expected concentrations falls within the linear range.

Example: Sample concentration is tentatively determined at 2 µg/L.

Ms1 – take 10 ml of digestate and add 0.2 ml of 100 µg/L spike standard (≈ 6 µg/L)

Ms2 – take 10 ml of digestate and add 0.4 ml of 100 µg/L spike standard (≈ 7 µg/L)

Ms 3 – take 10 ml of digestate and add 0.6 ml of 100 µg/L spike standard (≈ 8 µg/L)

10.4.10.3. Analyze Ms1, Ms2 and Ms3 and calculate the results using Eq.-10.5.5.

10.5. **Calculations**

10.5.1. Calibration Factor

Plot the absorbance (y-axis) versus the known concentration of the calibration standards (x-axis) using Excel spreadsheet or equivalent. The reciprocal of the slope shall be the calibration factor.

10.5.2. Calculate for Mercury Concentration

$$C = [(A + CF) - Y] * DF \qquad \text{Eq.-10.5.2}$$

where:

C – Concentration of Mercury, µg/L
A – Absorbance
CF – Calibration Factor = 1/slope
Y – Y-intercept / slope
DF – Dilution Factor

10.5.3. Calculate for Percent Recovery

$$\%\,\mathrm{Recovery} = \frac{(C_f - C)}{C_s} \times 100 \qquad \text{Eq.-10.5.3}$$

where:

C_f – Concentration found
C – Concentration of the sample (use 0 for LCS)
C_s – Concentration of spike

10.5.4. Calculate for Relative Percent Difference

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \qquad \text{Eq.-10.5.4}$$

where:

RPD – Relative Percent Difference
C_1 – Measured concentration of the first sample aliquot
C_2 – Measured concentration of the second sample aliquot

10.5.5. Calculation for MSA

$$C_x = \frac{(S_2)(V_s)(C_s)}{(S_2 - S_1)V_x} \qquad \text{Eq.-10.5.5}$$

where:

C_x – Concentration of the sample
C_s – Concentration of spike
S_1 – Analytical signal of MS1
S_2 – Analytical signal of MS2
V_x – Volume of sample aliquot
V_s – Volume of spike or reagent water

10.6. **Data Reduction**

10.6.1. Make a copy of the analytical run and sample preparation log.

10.6.2. Print a copy of raw data and the QC report.

10.6.3. Highlight the data to be reported.

10.6.4. Collate the reportable data separating the QC results from the sample results.

10.6.5. Keep all other data generated with the analytical folder marked with “For record only”.

10.7. **Report Generation**

10.7.1. Generate the method.txt file and sample results using HGF1.exe.

10.7.2. Generate the QC summaries using HGF3.exe.

10.7.3. Generate the case narrative using CN00.exe.

10.7.4. Arrange the analysis package in sequence as detailed below.

10.7.4.1. Case Narrative

10.7.4.2. Lab Chronicle

10.7.4.3. Sample Results

10.7.4.4. LCS/LCSD Summary

10.7.4.5. MS/MSD Summary

10.7.4.6. Post Digestion Spike Summary

10.7.4.7. Dilution Test Result Summary

10.7.4.8. Analytical Run Log

10.7.4.9. Raw Data

10.7.4.10. Sample Preparation Log

10.7.4.11. Non-Conformance Report (if any)

10.8. **Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- ✓ Check method blank is compliant to Project Specific Requirements (PSR) criteria.
- ✓ Check LCS/LCSD, MS/MSD and Dilution test against QC limits.
- ✓ Check analytical spike test if dilution test failed.
- ✓ Check for possible carry-over and if confirmation is performed.
- ✓ Review the attached logs that they are properly filled.
- ✓ Check the generated reports against the raw data, analytical run log and sample preparation log. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.
- ✓ Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.2. Submit the analytical folder for secondary review

10.9. **Preventive Maintenance**

10.9.1. Daily routine maintenance must be observed religiously. Observe manufacturer's notes regarding DOs and DON'Ts:

- System preparation is a ***MUST*** before instrument startup.
- Make certain that drying tube has been packed loosely. If drying tube is blocked, liquid may backflow into the optical cell; this will require disassembly and leaning.
- Do not shutdown the instrument when operational, abort the run first if interruption is needed.

10.9.2. Daily routine maintenance including checking of reductant solution, 10% $SnCl_2$, troubleshooting and major repairs must be recorded in the maintenance log. Refer to Form 7470FM.

10.9.3. Maintain the instrument clean at all times.

10.9.4. For troubleshooting, consult the Operations Manual, Section 4.

11.0 QUALITY CONTROL

11.1. **Sample Preparation QC**

11.1.1. Pipettes must be calibrated prior to its use. Refer to EMAX-QC06.

11.1.2. Reagents are subjected to QC check prior to its use. Refer to EMAX-QC01.

11.1.3. Properly treat all lab wares used in the sample preparations as specified in EMAX-QC07.

11.1.4. A preparative batch consists of 20 or fewer samples of the same matrix, that are prepared for analysis simultaneously or sequentially, using the same lots of all reagents.

11.1.5. Every preparative batch must have at least one method blank, one LCS and a set of MS/MSD unless otherwise specified by the project. Digest QC samples together with the field samples.

11.2. **Sample Analysis QC**

11.2.1. Every analytical run is preceded by an Initial Calibration (ICAL) and Initial Calibration Verification. The ICV standard should be obtained from a different source from that of the initial calibration. Analyze an instrument calibration blank (ICB) after the ICV. No further analysis is valid unless acceptance criteria are met.

11.2.2. Verify calibration with Continuing Calibration Verification (CCV) standard and Continuing Calibration Blank (CCB) after every ten samples and at the end of the analytical run.

11.2.3. Evaluate MS/MSD to document matrix interference.

11.2.4. Dilution Test shall be performed whenever a new or unusual sample matrix is encountered.

11.2.5. Perform Post Digestion Spike result if the recoveries of MS/MSD fail and if dilution test failed.

11.2.6. Use Method of Standard Addition (MSA) technique for analysis of all EP extracts and whenever a new sample matrix is being analyzed.

11.2.7. Refer to Appendix 1 for acceptance criteria.

11.3. **Method QC**

11.3.1. Detection Limit study must be established before the analytical procedure can be used. Quarterly verification must be performed. Refer to EMAX-QA04.

11.3.2. All analysts conducting this analysis must have an established Demonstration of Capability (DOC) as described in EMAX-QA05.

12.0 CORRECTIVE ACTION

12.1. Corrective actions for each Quality Control Procedure is summarized in Appendix 1.

12.2. **Calibration**

12.2.1. Initial Calibration (ICAL) - if ICAL is non-compliant, consider the following suggestions to correct the problem:

- Replace the sample tubing, prepare fresh rinsate and re-prepare fresh $SnCl_2$. Rinse the system for at least 15 minutes prior to calibration.
- If problem persist, run the latest calibration standard that passed to check for possible instrumentation problem. If it passes, this is an indication that no instrumentation problem exist, re-digest the calibration standards. If it failed, clean the lamp, prior to re-calibration.
- If problem persist, inform the supervisor for further action

12.2.2. Initial Calibration Verification (ICV) - if the ICV is non-compliant, consider the following suggestions to correct the problem:

- Run the latest ICV standard that passed to check for possible standards preparation error. If it passes, this is an indication of standards preparation error. Re-digest the ICV and re-analyze. If it fails, refer to Sec. 12.1.1 prior to re-calibration.

12.2.3. Continuing Calibration Verification (CCV) - If CCV is non-compliant, consider the following suggestion to correct the problem:

- Run the latest CCV standard that passed to check for possible standards preparation error. If it passes, this is an indication of standards preparation error. Re-digest the CCV and re-analyze. If it fails, refer to Sec. 12.1.1 prior to re-calibration.

12.3. **Sample Prep QC**

12.3.1. For insufficient amount of sample(s), inform the supervisor immediately for further action.

12.3.2. Method Blank (MB) - if MB is non-compliant, consider the following suggestion to correct the problem:

- Check the sample results. If sample results are non-detected, you may report the result upon concurring with the PM, otherwise perform the corrective action as specified in the PSR.

12.4. **Sample Analysis QC**

12.4.1. Lab Control Sample (LCS) - If LCS is non-compliant, consider the following suggestions to correct the problem:

- Check for errors in calculation and concentration of the analyte solution.
- Check instrument performance to determine if it is within acceptable guidelines.
- Re-calculate the data and/or re-analyze the extract if any of the above checks reveals a problem.
- If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate digestion problem, fill-up an NCR and order re-digestion to include the associated sample(s).

12.4.2. Matrix Spike / Matrix Spike Duplicate (MS/MSD) - If MS is non-compliant, consider the following suggestions to correct the problem:

- If recovery failed to meet the acceptance criteria and sample result is > 5X the LOQ and the spike amount is > 4X the parent sample concentration, evaluate the post digestion spike sample result. Refer to Appendix 1 for acceptance criteria. If it fails to meet the acceptance criteria, perform MSA.
- If recovery failed to meet the acceptance criteria and sample result is ~ 5X the LOQ and the spike amount is > 4X of the parent sample concentration, evaluate the serial dilution sample result. Refer to Appendix 1 for acceptance criteria. If it fails to meet the acceptance criteria, perform MSA.

12.5. A Non-Conformance Report (NCR) is required when the following circumstances occur:

- Anomalies other than specified in Appendix 1 is observed.
- Sample is out of technical holding time.

12.5.1. Refer to EMAX-QA08.

13.0 POLLUTION PREVENTION

13.1. Mercury is a very volatile element, dangerous levels are readily attained in air. Mercury vapour should not exceed 0.1 mg/m^{-3} in air. Air saturated with the vapor at 20°C contains mercury in a concentration far greater than that limit. The danger increases at higher temperatures. It is therefore important that

mercury be handled with care. Containers of mercury must be securely covered and spillage must be avoided. Mercury must only be handled under in a well-ventilated area. Prepare all standards in the fume hoods.

13.2. Because of the toxic nature of mercury vapor, precaution must be taken to avoid its inhalation. A bypass must be included on the system to vent the mercury vapor into an exhaust hood.

13.3. Small amounts of mercury spillage can be cleaned up by addition of sulphur powder. The resulting mixture must be properly labeled and turned over to the waste disposal unit for proper disposal.

13.4. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.5. To prevent internal pollution, the entire sample preparation process and analytical standard preparation must be conducted in a properly functioning fume hood.

14.0 WASTE MANAGEMENT

14.1. No samples may be dumped on the laboratory sink.

14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.

14.3. Place all wastes generated during analytical process in properly labeled satellite waste containers for proper collection.

14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. **Definition of Terms**

15.1.1. Mercury – is also known as quicksilver, is a chemical (element) that occurs naturally in the environment in several forms. One form of mercury is used in thermometers. This form is called “metallic mercury”. Mercury is also used in barometers and other common consumer products. Mercury can also be combined with other chemicals, such as chlorine, carbon or oxygen to form either “inorganic” or “organic” mercury compounds.

15.1.2. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.

15.1.3. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

15.1.3.1. **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.

15.1.3.2. **Analytical Batch** – is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.4. Detection Limit (DL) – is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

15.1.5. Limit of Detection (LOD) – is defined as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

15.1.6. Limit of Quantitation (LOQ) – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

15.1.7. Safety Data Sheet (SDS) – is a written information concerning a chemical physical properties, toxicity, health standards, fire hazard and reactivity data including storage, spill and handling precautions.

15.1.8. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.9. Calibration Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.10. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.1.11. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.

15.1.12. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyze to monitor the accuracy of the analytical system.

15.1.13. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.

15.1.14. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

15.1.15. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.16. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.17. Matrix – is a component or form of sample.

15.1.18. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.

15.1.19. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

15.1.20. Reagent Water – Purified water free from any target analyte or any other substances that may interfere with the analytical process.

15.2. **Application of EMAX QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing Mercury analysis by Cold Vapor Absorption Technique. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

15.3. **Department of Defense (DoD) and Department of Energy (DOE) Projects**

15.3.1. Samples from DoD sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD ELAP Quality Systems Manual (QSM), latest update, is applied.

16.0 REFERENCES

16.1. Method 7470A, Test Methods for Evaluating Solid Wastes, USEPA SW-846, 1992.

16.2. EMAX Quality Systems Manual, as updated.

17.0 APPENDICES

17.1. **Figures**

17.1.1. Figure 1 Autosampler Layout

17.1.2. Figure 2 Typical Calibration Curve

17.1.3. Figure 3 Typical Sample Result Summary

17.1.4. Figure 4 Typical LCS/LCSD Summary

17.1.5. Figure 5 Typical MS/MSD Summary

17.1.6. Figure 6 Typical Analytical Spike Summary

17.1.7. Figure 7 Typical Dilution Test Summary

17.1.8. Figure 6 Typical Case Narrative

17.2. **Appendices**

17.2.1. Appendix 1 Summary of Quality Control Procedures

17.2.2. Appendix 2 Demonstration of Capability

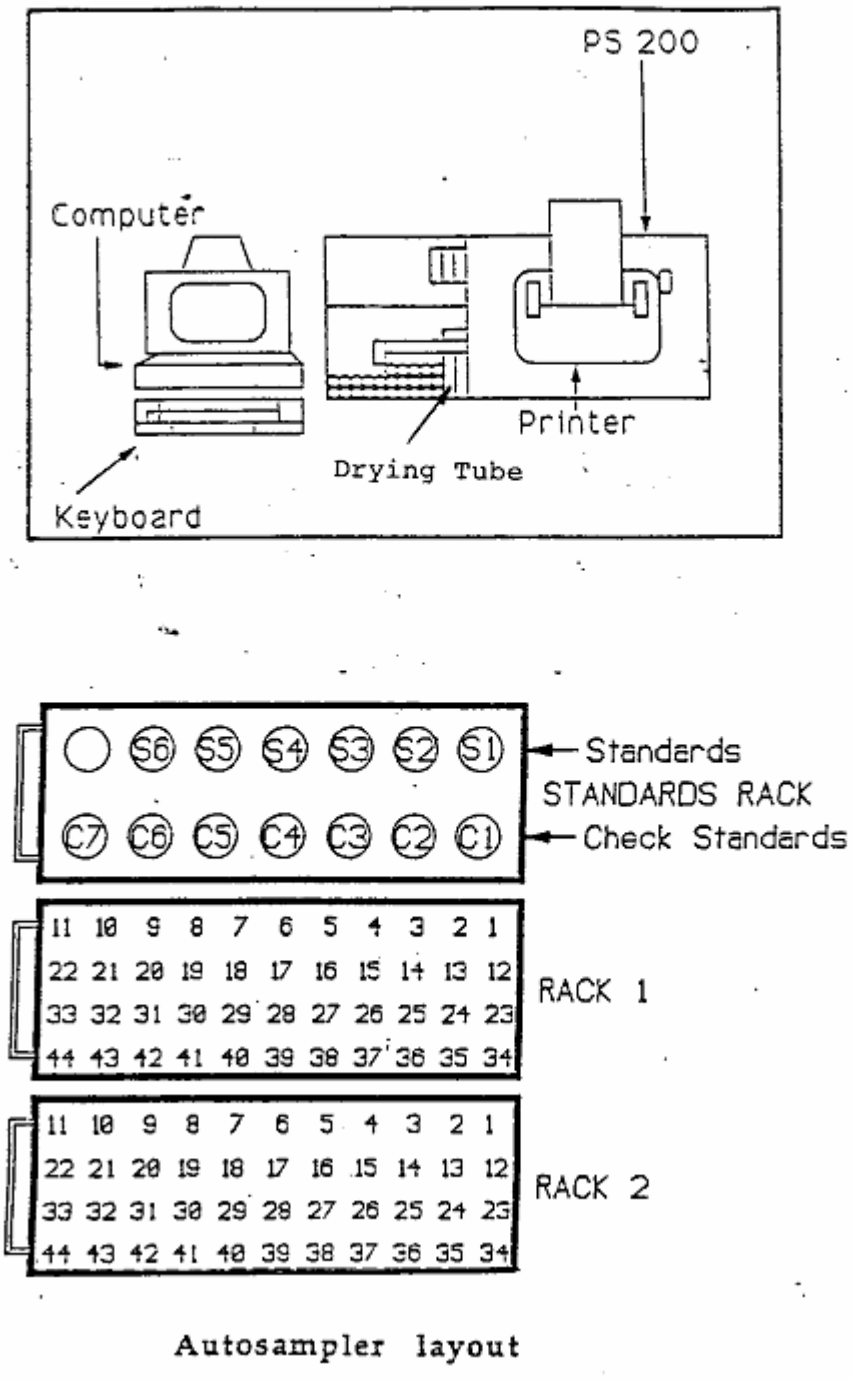
17.3. **Forms**

17.3.1. 7470FS Sample Preparation Log

17.3.2. 7470FA Analytical Run Log

17.3.3. 7470FM Instrument Maintenance Log

Figure 1: **AUTOSAMPLER LAYOUT**



Autosampler layout

Figure 2: TYPICAL CALIBRATION CURVE

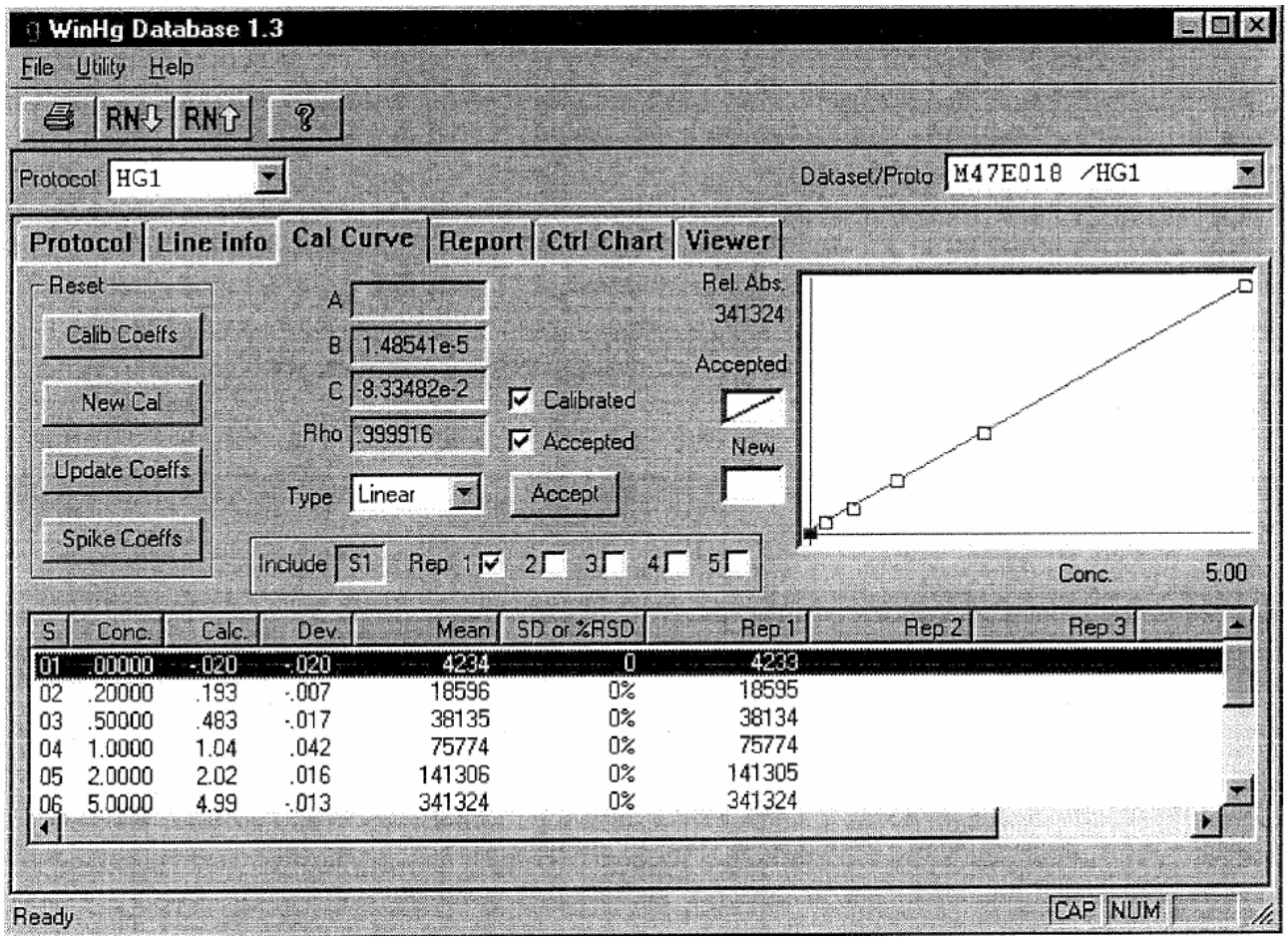


Figure 3: **TYPICAL SAMPLE RESULT SUMMARY**

METHOD SW7470A
MERCURY BY COLD VAPOR

Client : XYZ, INC.
Project : CLEAN WATER
Batch No. : 16E175
Matrix : WATER
InstrumentID : 47

CLIENT SAMPLE ID	EMAX SAMPLE ID	RESULTS (ug/L)	DIL'N FACTOR	MOIST (%)	LOQ (ug/L)	DL (ug/L)	LOD (ug/L)	ANALYSIS DATETIME	PREPARATION DATETIME	DATA FILE ID	CAL REF	PREP BATCH	COLLECTION DATETIME	RECEIVED DATETIME
MBLK1W	HGE027WB	ND	1	NA	0.50	0.054	0.10	05/31/1620:45	05/31/1614:00	M47E018043	M47E018	HGE027W	NA	NA
LCS1W	HGE027WL	2.48	1	NA	0.50	0.054	0.10	05/31/1620:47	05/31/1614:00	M47E018044	M47E018	HGE027W	NA	NA
LCD1W	HGE027WC	2.46	1	NA	0.50	0.054	0.10	05/31/1620:54	05/31/1614:00	M47E018047	M47E018	HGE027W	NA	NA
03M08-2016S	E175-08	ND	1	NA	0.50	0.054	0.10	05/31/1620:58	05/31/1614:00	M47E018049	M47E018	HGE027W	05/18/1614:28	05/20/16
03M08-2016SMS	E175-08M	2.59	1	NA	0.50	0.054	0.10	05/31/1621:03	05/31/1614:00	M47E018051	M47E018	HGE027W	05/18/1614:28	05/20/16
03M08-2016SMSD	E175-08S	2.59	1	NA	0.50	0.054	0.10	05/31/1621:05	05/31/1614:00	M47E018052	M47E018	HGE027W	05/18/1614:28	05/20/16
03M12B-2016S	E175-01	ND	1	NA	0.50	0.054	0.10	05/31/1621:07	05/31/1614:00	M47E018053	M47E018	HGE027W	05/18/1611:16	05/20/16
03M12B-2016S-9	E175-02	ND	1	NA	0.50	0.054	0.10	05/31/1621:10	05/31/1614:00	M47E018054	M47E018	HGE027W	05/18/1611:16	05/20/16
03M04B-2016S	E175-03	ND	1	NA	0.50	0.054	0.10	05/31/1621:12	05/31/1614:00	M47E018055	M47E018	HGE027W	05/18/1612:56	05/20/16
03M11B-2016S	E175-04	ND	1	NA	0.50	0.054	0.10	05/31/1621:14	05/31/1614:00	M47E018056	M47E018	HGE027W	05/18/1614:42	05/20/16
03M10-2016S	E175-05	ND	1	NA	0.50	0.054	0.10	05/31/1621:21	05/31/1614:00	M47E018059	M47E018	HGE027W	05/18/1616:22	05/20/16
03MW20-2016S	E175-06	ND	1	NA	0.50	0.054	0.10	05/31/1621:23	05/31/1614:00	M47E018060	M47E018	HGE027W	05/18/1611:28	05/20/16
03M18-2016S	E175-07	ND	1	NA	0.50	0.054	0.10	05/31/1621:25	05/31/1614:00	M47E018061	M47E018	HGE027W	05/18/1611:35	05/20/16
03M19-2016S	E175-09	ND	1	NA	0.50	0.054	0.10	05/31/1621:27	05/31/1614:00	M47E018062	M47E018	HGE027W	05/19/1611:17	05/20/16

Figure 4: **TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA
LAB CONTROL SAMPLE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER
BATCH NO. : 16E175
METHOD : SW7470A

MATRIX	: WATER		% MOISTURE: N/A
DILUTION FACTOR	: 1	1	1
SAMPLE ID	: MBLK1W	LCS1W	LCD1W
LAB SAMPLE ID	: HGE027WB	HGE027WL	HGE027WC
LAB FILE ID	: M47E018043	M47E018044	M47E018047
DATE PREPARED	: 05/31/1614:00	05/31/1614:00	05/31/1614:00
DATE ANALYZED	: 05/31/1620:45	05/31/1620:47	05/31/1620:54
PREP BATCH	: HGE027W	HGE027W	HGE027W
CALIBRATION REF	: M47E018	M47E018	M47E018

ACCESSION:

PARAMETER	MB RESULT (ug/L)	SPIKE AMT (ug/L)	BS RESULT (ug/L)	BS REC (%)	SPIKE AMT (ug/L)	BSD RESULT (ug/L)	BSD REC (%)	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Mercury	ND	2.50	2.48	99	2.50	2.46	98	1	80-120	20

Figure 5: **TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER
BATCH NO. : 16E175
METHOD : SW7470A

MATRIX	: WATER		% MOISTURE: NA
DILUTION FACTOR	: 1	1	1
SAMPLE ID	: 03M08-2016S	03M08-2016SMS	03M08-2016SMSD
LAB SAMPLE ID	: E175-08	E175-08M	E175-08S
LAB FILE ID	: M47E018049	M47E018051	M47E018052
DATE PREPARED	: 05/31/1614:00	05/31/1614:00	05/31/1614:00
DATE ANALYZED	: 05/31/1620:58	05/31/1621:03	05/31/1621:05
PREP BATCH	: HGE027W	HGE027W	HGE027W
CALIBRATION REF	: M47E018	M47E018	M47E018

ACCESSION:

PARAMETER	PARENT RESULT (ug/L)	SPIKE AMT (ug/L)	MS RESULT (ug/L)	MS REC (%)	SPIKE AMT (ug/L)	MSD RESULT (ug/L)	MSD REC (%)	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Mercury	ND	2.50	2.59	104	2.50	2.59	104	0	80-120	20

Figure 6: TYPICAL ANALYTICAL SPIKE SUMMARY

EMAX QUALITY CONTROL DATA
ANALYTICAL SPIKE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER
BATCH NO. : 16E175
METHOD : SW7470A

MATRIX : WATER % MOISTURE: NA

DILUTION FACTOR:	1	1
SAMPLE ID :	03M08-2016S	03M08-2016SMS
LAB SAMPLE ID :	E175-08	E175-08A
LAB FILE ID :	M47E018049	M47E018048
DATE PREPARED :	05/31/1614:00	05/31/1614:00
DATE ANALYZED :	05/31/1620:58	05/31/1621:03
PREP BATCH :	HGE027W	HGE027W
CALIBRATION REF:	M47E018	M47E018

ACCESSION:

PARAMETER	SMPL RESULT (ug/L)	SPIKE AMT (ug/L)	AS RSLT (ug/L)	MS REC (%)	QC LIMIT (%)
Mercury	ND	2.50	2.58	103	85-115

Figure 7: TYPICAL DILUTION TEST SUMMARY

EMAX QUALITY CONTROL DATA
SERIAL DILUTION ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER
BATCH NO. : 16E175
METHOD : SW7470A

MATRIX : WATER % MOISTURE: NA
DILUTION FACTOR: 1 1
SAMPLE ID : 03M08-2016S 03M08-2016SMS
LAB SAMPLE ID : E175-08 E175-08J
LAB FILE ID : M47E018049 M47E018050
DATE PREPARED : 05/31/1614:00 05/31/1614:00
DATE ANALYZED : 05/31/1620:58 05/31/1621:01
PREP BATCH : HGE027W HGE027W
CALIBRATION REF: M47E018 M47E018

ACCESSION:

PARAMETER	SMPL RESULT (ug/L)	SERIAL DIL RSLT (ug/L)	DIL RST (%)	QC LIMIT (%)
Mercury	ND	ND	0	10

Figure 8: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ,INC.

Project: CLEAN WATER

SDG : 16E175

METHOD SW7470A
MERCURY BY COLD VAPOR

A total of nine (9) water samples were received on 05/20/16 to be analyzed for Mercury by Cold Vapor in accordance with Method SW7470A and project specific requirements.

Holding Time
Samples were digested and analyzed within the prescribed holding time.

Calibration
Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source (ICV). Continuing calibration (CCV) verifications were carried on a frequency specified by the project. All calibration requirements were within acceptance criteria.

Method Blank
Method blank was prepared and analyzed at the frequency required by the project. For this SDG, one (1) method blank was analyzed. Mercury was not detected in HGE027WB. Refer to sample result summary form for details.

Lab Control Sample
Lab control sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of LCS/LCD was analyzed. HGE027WL/HGE027WC was within LCS limits. Refer to LCS summary form for details.

Matrix QC Sample
Matrix spike sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of MS/MSD was analyzed. Mercury was within MS QC limits in E175-08M/S. Refer to Matrix QC summary form for details. Analytical spike and serial dilution were analyzed for matrix interference evaluation. Results were within method acceptance criteria.

Sample Analysis
Samples were analyzed according to prescribed analytical procedures. Results were evaluated in accordance to project requirements. For this SDG, all quality control requirements were met.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
Initial multipoint calibration	Daily initial calibration prior to sample analysis	Correlation coefficient r≥ 0.995 for linear regression	Correct the problem then repeat initial calibration		
Initial calibration verification (second source)	Daily after initial calibration	Analyte within ± 10% of expected value	Correct the problem then repeat initial calibration		
Calibration verification (CCV)	Daily, before sample analysis, every 10 samples and at the end of analysis sequence	Analyte within ± 10% of expected value	Repeat calibration and re-analyze all samples since last successful calibration		
Calibration blank (CCB)	After every calibration verification	No analyte detected > LOD	Correct the problem then re-analyze calibration blank and previous samples		
Method blank (MB)	One per preparation batch	No analyte detected > ½ LOQ	Re-prep and re-analyze method blank and all samples processed with the contaminated blank		
Lab Control Sample (LCS)	One LCS per preparation batch	%Rec.: 80-120%	Re-prep and re-analyze the LCS and all associated samples		
MS/MSD or MS/Dup	One set MS/MSD or MS/Dup in every preparatory batch	%Rec. 80-120%, RPD≤ 20%	Perform Post-Digestion Spike.		
Post-Digestion Spike	When MS/MSD or MS/Dup fails	%Rec. 85-115%	Perform dilution test if analyte concentration is sufficiently high (~5x the LOQ after dilution), otherwise perform MSA.		
Dilution Test	When Post-Digestion Spike fails and analyte concentration is sufficiently high (~5x the LOQ after dilution)	Within ± 10% of the parent sample result	Perform MSA.		
Comments: Refer to PSR for flagging criteria LOQ = lowest calibration point			Reviewed By:		
			Date:		

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
MERCURY
EPA 7470A

EMAX-7470 Rev. 7

Conc Unit: mg/L

Sample Amount(mL): 50

Extraction dates: 1/11/2016

Analysis dates: 1/11/2016

Extracted and Analyzed by: N. Tan

PARAMETER	HGA006WL	HGA006WC	HGA007WL	HGA007WC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	M47A004012	M47A004013	M47A004043	M47A004044							
Mercury	2.56	2.56	2.47	2.51	2.5	2.53	101	0.044	2	80 - 120	PASSED

7470FS: SAMPLE PREPARATION LOG

EMAX LABORATORIES, INC.

DIGESTION LOG
for
MERCURY

Page 1

Note: For samples, relevant QCs/Standards digested, refer to attached digestion sequence.

Comments:

Digestion Vessel Lot #:

Book #: E47-102

Batch No.:

Matrix:

SOP #	Rev. #
☐ EMAX-7470	8
☐ EMAX-7471	9
☐ EMAX-245.1	4
☐ EMAX-	

Standards	ID	Conc. (µg/L)	Amount Added (ml)
ICAL			
CCV			
ICV			
LCS/MS			

Reagent	ID / Lot #
HNO_3	
HCl	
H_2SO_4	
$KMnO_4$	
$K_2S_2O_8$	
$NH_2OH•HCl•NaCl$	
$SnCl_2$	
Silica Sand	
Reagent Water	
pH strip 0-14	

Digestor ID/ Temp (°C)

Thermometer ID/LOC:

Thermometer ID/LOC:

Pipette ID:

☐ H_2SO_4 dispenser checked @ 2.5 ml with Class A graduated cylinder

☐ HCl dispenser checked @ ___ ml with Class A graduated cylinder

☐ HNO_3 dispenser checked @ ___ ml with Class A graduated cylinder

Prepared By:

Standard Added By: Witnessed By:

7470FA: ANALYTICAL RUN LOG

EMAX LABORATORIES, INC.

ANALYSIS RUN LOG
for
MERCURY

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Start Date: **Start Time:**

End Date: **End Time:**

Comments:

Book #:	A47-085
Instrument No.:	47
Analytical Sequence/Batch:	
Method File:	
Micropipette ID:	☐ ICP DOE-1
Micropipette ID:	☐ HG-03
Micropipette ID:	☐ HG-04
Micropipette ID:	☐

SOP #	Rev. #
☐ EMAX-7470	
☐ EMAX-7471	
☐ EMAX-245.1	
☐ EMAX-	

STANDARDS ID
S1
S2
S3
S4
S5
S6
CCV
ICV
LCS
Analytical Spike

Analyzed By:

Date:

7470FM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: 47 **Month:** **Year: 201_** **Book #: M47-010**

DAQ TEST							
Sample: 700,000 - 1,720,000							
Hg Lamp Volt: 8.0-9.5							
GAS CONTROL TEST							
Argon Input Pressure: 75-90 psi							
Power Supply Voltage: 5V+/-0.25, +12V+/-0.5, -12V+/-0.5							
Gas: 0-1.0 LPM							
Pump Rate: 2-15 ml/min							
Instrument Warm-up: Minimum 5 mins							
Peristaltic Pump							
Tubings Pump/Capillary							
Fume Hood							
Reductant Bottle							
Rinse Solution							
Computer Date/Time							
Printer Paper							
Analyst Initial							
Trouble Shooting							
Problem(s) Encountered							
Return to Specs (Analyst Initial)							

Code	Problems Encountered	Code	Corrective Action

STANDARD OPERATING PROCEDURES

DIESEL RANGE ORGANICS

SOP No.:	EMAX-8015D	Revision No. 7	Effective Date: 20-Sep-17
Prepared By:	Larisa Ermolova		Date: 9/20/17
Approved By:	Kenette Pimentel QA Manager		Date: 09.20.17
Approved By:	Caspar Pang Laboratory Director		Date: 09-20-17

Control Number: 8015D-07-

1.0 SCOPE AND APPLICATION

1.1 This method is used to analyze extractable fuel hydrocarbons in water, soil and other sediment samples. In the vast field of petroleum hydrocarbons, this method is limited to provide semi-quantitative results on those extractable hydrocarbons with a comparable aliphatic hydrocarbon range from C_{10} to C_{34}.

1.2 This SOP is an adaptation of SW846 Method 8015C. This SOP is also applicable to SW846 Method 8015B and Method 8015D.

2.0 SUMMARY OF METHOD

2.1 Petroleum hydrocarbons are extracted in methylene chloride, analyzed by flame ionization detector (FID) in gas chromatograph and quantified as diesel fuel at C_{10} to C_{28} range. The hydrocarbons that fall in this range are defined as Diesel Range Organics (DRO).

2.2 Other fuel standards that fall in the range of C_{10} to C_{34} can also be quantitated by this method.

2.3 **Interference**

2.3.1 Glassware can be a potential source of contamination. They must be scrupulously cleaned prior to its use.

2.3.2 Carry-over from a highly concentrated sample can be potential source of contamination. Instrument performance must be observed keenly for possible carry-over. If this is apparent, inject solvent blank until no trace of carry-over is observed.

2.3.3 Deposits may adhere in the injection port/glass liner over a period of time and can cause interference. The injection port and glass liner must be routinely cleaned.

3.0 DETECTION LIMITS

3.1 **Detection Limit (DL), Limit of Detection (LOD) and Limit of Quantitation (LOQ)**

3.1.1 Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.

3.1.2 Refer to Table 1 for established limits.

4.0 DYNAMIC RANGE

4.1 The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (refer to Section 9.5.1). Dilute and re-analyze all samples having results above this range to properly quantitate.

4.2 The lowest quantifiable range of diluted samples is equal to the lowest calibration point (refer to Section 9.5.1). Lower the dilution factor and re-analyze all diluted samples analyzed below this range to properly quantitate.

5.0 SAMPLE HOLDING TIME & PRESERVATION

5.1 Sample Collection

5.1.1 Water samples received in the lab are expected to be contained in an amber bottle with Teflon-lined cap and cooled to ≤ 6°C without freezing.

5.1.2 Soil samples received in the lab are expected to be contained in a jar or Shelby tube and cooled to ≤ 6°C without freezing.

5.2 Holding Time

5.2.1 Water samples must be extracted within 7 days from sampling date.

5.2.2 Soil samples must be extracted within 14 days from sampling date.

5.2.3 All extracts must be analyzed within 40 days from extraction.

5.3 Preservation

5.3.1 Store water samples at ≤ 6°C without freezing, store in dark.

5.3.2 Store soil samples and extracts at ≤ 6°C without freezing.

6.0 ASSOCIATED SOPs

6.1	EMAX-3520	Extraction, Continuous Liquid/Liquid
6.2	EMAX-3540	Extraction, Soxhlet
6.3	EMAX-3550	Extraction, Pulse Sonication
6.4	EMAX-3580	Waste Dilution
6.5	EMAX-DM01	Data Flow & Review
6.6	EMAX-QA04	Detection Limit (DL)
6.7	EMAX-QA05	Training
6.8	EMAX-QA08	Corrective Action
6.9	EMAX-QC01	Quality Control for Chemicals
6.10	EMAX-QC02	Analytical Standard Preparation
6.11	EMAX-QC07	Glassware Cleaning
6.12	EMAX-SM01	Sample Management

6.13 EMAX-SM03 Waste Disposal

6.14 EMAX-SM04 Analytical and QC Sample Labeling

7.0 SAFETY

7.1 Read all SDS for chemicals listed in this SOP.

7.2 Treat reagents, standards and samples as potential hazard. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.

7.3 If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amounts of water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENT, CHEMICALS & REAGENTS

8.1 Instruments and Supplies

8.1.1 Gas Chromatography

Instrument ID	**Inst. D5**	**Inst. F2**
Gas Chromatography and Autosampler	Agilent 6890 with 7683B autosampler	Perkin Elmer Clarus 680
Detector	FID	FID
Column	HP-5 30m x 0.32 x 0.25µm	HP-5 30m x 0.32 x 0.25µm
Data Acquisition	EZChrom Elite 3.3.1	EZChrom Elite 3.3.2

8.1.2 Syringes: 10, 50, 100, 250, 500, 1000 µl microsyringe

8.1.3 Volumetric Flask: 10, 50, 100 and 1000 ml

8.1.4 Vials: 2, 10 and 40 ml, amber

8.1.5 Bottle: 125, 250 ml (amber)

8.2 Chemicals and Reagents

8.2.1 Where available, purchase reagent-grade chemicals and reagents.

8.2.2 Methylene Chloride

8.2.3 Acetone, Methanol

8.2.4 High purity He, H_2, Air, N_2

9.0 STANDARDS

9.1 Standard Preparation

9.1.1 Refer to EMAX-QC02 for proper analytical standard preparation.

9.1.2 Other concentration levels may be prepared as long as it complies with the method and project requirements.

9.2 **Stock Standard**

9.2.1 Diesel

Purchase stock standards as certified solutions from certified vendors as suggested below or equivalent:

Standard Name	Source	Concentration (mg/L)	Solvent	Intended Use
Diesel	Restek	50,000	$MeCl_2$	ICAL, DCC
Diesel	CPI	50,000	Acetone	ICV, LCS/LCSD, MS/MSD
Bromobenzene (C_6H_5Br) Hexacosane ($C_{26}H_{54}$)	CPI	2000 500	Acetone/$MeCl_2$ 1:1	Surrogates

9.2.2 JP5 and Motor Oil

Purchase stock standards as certified solutions from certified vendors as suggested below or equivalent:

Standard Name	Source	Concentration (mg/L)	Solvent	Intended Use
JP-5 Military Fuel	Supelco	10,000	$MeCl_2$	ICAL, DCC
JP-5	AccuStandard	20,000	Methanol	ICV, LCS/LCSD, MS/MSD
Motor Oil 5W30	Restek	50,000	$MeCl_2$	ICAL, DCC
SAE 5W30 Motor Oil	AccuStandard	Neat	NA	ICV, LCS/LCSD, MS/MSD

9.3 **Intermediate Standard**

9.3.1 Prepare SAE 5W30 Motor Oil Standard with Methylene Chloride as suggested below:

Standard Name	Concentration	Amount	Final Conc.	Solvent	Final Volume
SAE 5W30 Motor Oil	Neat	1.0 g	100,000 mg/L	$MeCl_2$	10 ml

9.3.2 Prepare Motor Oil working standard as suggested below:

Standard Name	Concentration	Amount	Final Conc.	Solvent	Final Volume
Motor Oil Spike	100,000 mg/L	5.0 ml	5,000 mg/L	Acetone	100 ml

9.4 **Calibration Standard**

9.4.1 Initial Calibration Standard

9.4.1.1 **Diesel**

Prepare a minimum of 5-point calibration standard (ideally 6-point) from the primary stock standard in $MeCl_2$ and store in Teflon-sealed vial with minimal headspace; suggested concentration and injection volume are as follows:

ICAL Pt.	Diesel (50,000 mg/L) Aliquot (µl)	Bromobenzene/ Hexacasone (2000/500 mg/L) Aliquot (µl)	Final Volume (ml)	Final Concentration (mg/L)		
				Diesel	Bromobenzene	Hexacosane
1	0.1	0	1	5	0	0
2	0.4	10	1	20	20	5
3	1	20	1	50	40	10
4	10	40	1	500	80	20
5	20	50	1	1000	100	25
6	40	60	1	2000	120	30
7	60	110	1	3000	220	55

9.4.1.2 **JP5**

Prepare a minimum of 5-point calibration standard (ideally 6-point) from the primary stock standard in $MeCl_2$ and store in Teflon-sealed vial with minimal headspace

ICAL Pt.	Stock Standard (10,000 mg/L) Aliquot (µl)	Final Volume (ml)	Final Concentration (mg/L)
1	1	1	10
2	5	1	50
3	10	1	100
4	50	1	500
5	150	1	1500
6	300	1	3000

9.4.1.3 **Motor Oil**

Prepare a minimum of 5-point calibration standard (ideally 6-point) from the primary stock standard in $MeCl_2$ and store in Teflon-sealed vial with minimal headspace

ICAL Pt.	Stock Standard (50,000 mg/L) Aliquot (µl)	Final Volume (ml)	Final Concentration (mg/L)
1	0.2	1	10
2	1	1	50
3	2	1	100
4	10	1	500
5	30	1	1500

ICAL Pt.	Stock Standard (50,000 mg/L) Aliquot (µl)	Final Volume (ml)	Final Concentration (mg/L)
6	60	1	3000

9.4.2 Initial Calibration Verification Standard

9.4.2.1 **Diesel**

Prepare initial calibration verifications standard using secondary source standard in $MeCl_2$.

Diesel Std. (50,000 mg/L) Aliquot (µl)	Bromobenzene / Hexacosane (2000 / 500 mg/L) Aliquot (µl)	Final Volume (ml)	Final Conc. Analyte/Surrogates (mg/L)
10	40	1	500 / 80/ 20

9.4.2.2 **JP5/Motor Oil**

Prepare initial calibration verifications standard using secondary source standard in $MeCl_2$.

JP5 Std. (20,000 mg/L) Aliquot (µl)	Motor Oil Std. (Intermediate Std.) (5,000 mg/L) Aliquot (µl)	Final Volume (ml)	Final Conc. JP5/Motor Oil (mg/L)
25	100	1	500 / 500

9.4.3 Continuing Calibration Standard

9.4.3.1 **Diesel**

Prepare daily calibration standards using primary-source standards in $MeCl_2$.

Diesel Std. (50,000 mg/L) Aliquot (µl)	(2000 / 500 mg/L) Aliquot (µl)	Final Volume (ml)	Final Conc. Diesel/Surrogates (mg/L)
100	400	10	500 / 80/20

9.4.3.2 **JP5/Motor Oil**

Prepare daily calibration standards using primary-source standards in $MeCl_2$.

JP5 Std. (10,000 mg/L) Aliquot (µl)	Motor Oil Std. (50,000 mg/L) Aliquot (µl)	Final Volume (ml)	Final Conc. JP5/Motor Oil/Surrogates (mg/L)
500	100	10	500 / 500

9.5 **LCS/MS Spike Standard**

9.5.1 Use secondary source standards or primary source standards for laboratory control standard (LCS) and matrix spike (MS) standards.

9.6 **Retention Time Window Standard**

9.6.1 Purchase stock n-alkane standards as suggested below or equivalent.

Standard Name	Source	Concentration	Solvent
Calibration Window Defining Hydrocarbon ($C_8 – C_{40}$)	AccuStandard	1000	Chloroform
Hydrocarbon Window Defining Standard ($C_9 – C_{39}$)	AccuStandard	500	Hexane

9.6.2 Prepare working n-alkane standards with $MeCl_2$ as follows:

Standard Name	Concentration	Amount	Final Volume	Final Concentration
C_8-C_{40} RTW Std.	1000 mg/L	0.2 ml	10 ml	20 mg/L
C_9-C_{39} RTW Std.	500 mg/L	0.4 ml		20 mg/L

10.0 PROCEDURES

10.1 **Sample Preparation**

10.1.1 Aqueous Samples

10.1.1.1 Prepare aqueous samples in accordance with EMAX-3520, unless otherwise specified by the project.

10.1.2 Soil Samples

10.1.2.1 Prepare soil samples in accordance with any of the following extraction procedures: EMAX-3540, EMAX-3550 or EMAX-3580 as specified by the project. Where project does not specify extraction procedure, default to EMAX-3550.

10.2 **Instrument Parameters**

10.2.1 Fine tune the instrument guided by parameter conditions as listed below:

Injector Temperature – 280°C

Detector Temperature – 320°C

Head Pressure – 21 psi

Temperature Program

Instrument	D5	F2
Initial Temperature	55°C, hold for 0.5 min	60°C, hold for 0.5 min
Temperature 1	320°C, hold for 5 min	310°C, hold for 4 min

Rate 1	60°C /min	55°C /min
Temperature 2	-	320°C, hold for 2 min
Rate 2	-	60°C /min
Total Run Time	10 min	12 min

10.3 **Calibration**

10.3.1 Initial Calibration

10.3.1.1 Analyze the initial calibration standards prepared. Sum the area of all peaks eluting between C_{10} to C_{28} for each of the calibration point. Generate this area by rejoining a horizontal baseline between the retention time of C_{10} to C_{28}. Check that the highest point does not have saturated peak(s).

Note: The lowest calibration determines the limit of quantitation (LOQ). Therefore, check that the LOQ is in conformance to the current projects where the ICAL will be used.

10.3.2 Initial Calibration Verification (ICV)

10.3.2.1 After establishing ICAL, analyze the ICV standard (Refer to Section 9.4.2) to verify the validity of the ICAL. Refer to Appendix 1 for acceptance criteria. If non-compliant, refer to Section 12 for corrective action.

10.3.3 Retention Time Window Check (RTW)

10.3.3.1 Analyze the RTW standard after every ICAL to set carbon cut-off ranges (e.g., C_{10}-C_{24}, C_{10}-C_{28}). Refer to Section 9.6.

10.3.4 Daily Continuing Calibration (DCC)

10.3.4.1 Analyze a continuing calibration (Refer to 9.4.3) at the beginning of a 12-hour shift or as specified by the project.

10.3.4.2 Refer to Appendix 1 for acceptance criteria and corrective action.

10.4 **Analysis**

10.4.1 Extract Preparation

10.4.1.1 Allow the extracts to equilibrate to room temperature.

10.4.1.2 Transfer about 1 ml of extracts into autosampler vials.

10.4.2 Analytical Sequence

10.4.2.1 Analyze instrument blank to ensure that the instrument is free from contamination.

10.4.2.2 Analyze DCC to check ICAL validity.

10.4.2.3 Analyze Method Blank to check for preparation batch contamination.

10.4.2.4 Analyze Lab Control Sample to check accuracy.

10.4.2.5 Analyze Lab Control Sample Duplicate (if required by the project).

10.4.2.6 Analyze samples to a maximum of 12-hour runs or as specified by the project.

10.4.2.7 Analyze matrix spikes (MS/MSD) per project requirement.

10.4.2.8 Record the analytical sequence in the Analytical Run Log.

10.4.2.9 Print instrument sequence before and after the analysis run and attach to the analytical run. Document any changes that occurred during the process.

10.4.3 Identification and Quantitation

10.4.3.1 Identification is based on pattern recognition. Hence, compare sample chromatograms to reference hydrocarbons standard chromatograms for their response hydrocarbon range and peak distribution to determine the most probable petroleum product.

10.4.3.2 All peaks eluting within the established RT window identifies the DRO, JP5 and Motor Oil.

10.4.3.3 When the elution profile of a sample does not match that of diesel standard, JP5 or motor oil, but falls within the retention time window, quantitate results as diesel range organics (DRO) and denote the observed deviation in case narrative.

10.4.3.4 Quantitation is achieved by the summation of all peaks in the chromatogram minus the solvent peak, and the sample result is calculated using Eq.-10.5.3.

10.4.3.5 Integrate the total peak area response and quantitate the total area by using the ACF of Diesel, JP5 or Motor Oil (refer to Eq.-10.5.1.2).

10.4.3.6 When manual integration is necessary follow the procedures described in EMAX-DM01 (Section for manual integration).

10.4.4 Retention Time Window (RTW)

10.4.4.1 **Establishing RTW**

10.4.4.1.1 Run RTW standard over a period of 72 hours.

10.4.4.1.2 Calculate the Standard Deviation (SD) of absolute retention time obtained for each analyte (use Eq.-10.5.1.3).

10.4.4.1.3 The width of RTW is defined by ±3XSD.

10.4.4.2 **Evaluating RTW**

10.4.4.2.1 If the SD is equal to 0.00, default to the previous study until historical data is obtained to define the RTW for the current instrument.

10.4.4.2.2 For new instruments, use the established retention time from another instrument having the same instrument parameters (e.g. detector, temperature program and column). If there are no instruments with the same instrument parameter, use 0.03 minutes as the default RTW until historical data is obtained to define the RTW for the current instrument parameters condition.

10.4.4.3 **Application of RTW**

10.4.4.3.1 Establish the center of absolute retention time for each analyte to include the surrogate(s) from the daily calibration check at the beginning of the analytical shift then apply the established RTW.

10.4.4.3.2 Whenever the observed retention is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing analyses.

10.4.4.4 **Updating RTW**

10.4.4.4.1 Re-establish the RTW as described in Section 10.4.4.1 when any of the following conditions occur.

- Yearly RTW update
- Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of the RTW)
- Major instrument maintenance (e.g. replacement of detector).

10.4.4.4.2 If the calculated new RTW is significantly narrower than the previously established RTW, default to the previously established RTW.

10.4.5 Sample Result Evaluation

10.4.5.1 Check QC Criteria as soon as available.

- Check LCS, MS surrogate recoveries against Project Specific Requirement (PSR). In the absence of PSR, default to in-house QC limits. Refer to Appendix 1.

10.4.5.2 **Qualitative Identification**

- Compare the sample chromatogram to the pattern established by the calibration standard.
- When peaks other than diesel pattern are detected within the DRO, take note of it and discuss in the case narrative.

10.4.5.3 **Quantitation**

- Assign the appropriate hydrocarbon range for quantitation (e.g. $C_{10} – C_{28}$; $C_{18} – C_{34}$; C_8-C_{18}; etc.).
- Integrate all peaks bracketed by the hydrocarbon range baseline to the baseline. Set the integration range at least 95% within of the total spectrum of the expected DRO range.
- Calculate the result as specified in Figure 2 based on the appropriate fuel standard (e.g. Diesel: $C_{10} – C_{28}$; Motor Oil: $C_{18} – C_{34}$; JP5 C_8-C_{18}). In the absence of alkane range specification for the project, request for directive from the PM.
- Check that all positively identified target analytes are quantitated within the calibration range.
- Dilute and re-analyze all positively identified target analytes exceeding calibration range.
- When peaks other than diesel pattern are detected within the DRO range, quantitate using the DRO calibration factor and discuss it in the case narrative (e.g. peaks within the DRO range does not resemble diesel pattern or motor oil pattern).

When saturated peaks are present, dilute the extract appropriately until peaks are eluted properly.

10.5 **Calculations**

10.5.1 Initial Calibration

10.5.1.1 **Calculate for Calibration Factor (CF)**

$$CF = \frac{R_t}{C_v} \qquad \text{Eq.10.5.1.1}$$

where:

R_t – Total response of the integrated peaks

C_v – Known value of the standard concentration, mg/L

10.5.1.2 **Calculate for the Average Calibration Factor (ACF)**

$$ACF = \frac{\sum CF}{n} \qquad \text{Eq.-10.5.1.2}$$

where:

$\sum CF$ – Summation of Calibration Factors

n – Number of measurements

10.5.1.3 **Calculate for Standard Deviation (SD)**

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2}{n-1}} \qquad \text{Eq.-10.5.1.3}$$

where:

X_i – Result at the i^{th} measurement

$\bar{x}$ – Mean

n – Number of measurements

10.5.1.4 **Calculate for Percent Relative Standard Deviation (%RSD)**

$$\%RSD = \frac{SD}{ACF} x100 \qquad \text{Eq.-10.5.1.4}$$

where:

SD – Standard deviation

ACF – Average Calibration Factor

10.5.2 Calculate the Percent Difference of DCC from ACF

$$\%D = \frac{C_f - C_k}{C_k} * 100$$ Eq.-10.5.2

where:

$\%D$ – Percent Difference DCC from known concentration

C_k – Known concentration of the analyte, in mg/L

C_f – Found concentration, in mg/L

10.5.3 Calculate for Sample Concentration

10.5.3.1 **Water Samples**

$$C = \frac{(R_t)(V_e)(DF)}{(ACF)(A_s)}$$ Eq.-10.5.3.1

where:

C – Concentration of the sample, mg/L

R_t – Total response of the integrated peaks

V_e – Volume of Extract, ml

A_s – Sample amount, ml

DF – Dilution Factor

ACF – Average Calibration Factor

10.5.3.2 **Soil Samples**

$$C = \frac{(R_t)(V_e)(DF)}{(ACF)(A_s)(\%S)}$$ Eq.-10.5.3.2

where:

C – Concentration of the sample, mg/Kg

R_t – Total response of the integrated peaks

V_e – Volume of Extract, ml

A_s – Sample amount, g

DF – Dilution Factor

ACF – Average Calibration Factor

$\%S$ – Percent solids (%S of water = 1)

10.5.4 Percent Recovery

$$\%\,\mathrm{Recovery} = \frac{(C_f - C)}{C_s} \times 100$$ Eq.-10.5.4

where:

C_f – Concentration found

C – Concentration of the sample (use 0 for LCS)

C_s – Concentration of spike

10.5.5 Relative Percent Difference (RPD)

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} x100 \qquad \text{Eq.-10.5.5}$$

where:

C_1 – Measured concentration of the first sample aliquot

C_2 – Measured concentration of the second sample aliquot

10.6 **Data Reduction**

10.6.1 Make a copy of the analytical run log.

10.6.2 Print a copy of the raw data and the QC report.

10.6.3 Highlight the data to be reported.

10.6.4 Collate the reportable data separating the QC results from the sample results.

10.6.5 Keep all other data generated with the analytical folder marked with "For record only".

10.7 **Report Generation**

10.7.1 Generate the method.txt file using WDBX[1]CN.exe.

10.7.2 Generate the sample results using F1NVX[1]C.exe or F1NVX[1]C4.exe.

10.7.3 Generate the QC summary using QCVX[1]CN.exe or QCVX[1]CN4.exe.

10.7.4 Generate the Lab Chronicle using LABCHRN1.exe.

10.7.5 Generate the Case Narrative using CN00.exe.

10.8 **Data Review**

10.8.1 Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analysis.

10.8.1.1 Case Narrative

10.8.1.2 Lab Chronicle

10.8.1.3 Sample Results

10.8.1.4 Method Blank Results

10.8.1.5 LCS/LCSD Summary

[1] X – version number

10.8.1.6 MS/MSD Summary

10.8.1.7 ICAL Summary

10.8.1.8 ICV Summary

10.8.1.9 DCC Summary

10.8.1.10 Analytical Run Log

10.8.1.11 Sample Preparation Log

10.8.1.12 Non-Conformance Report (if any)

10.8.2 Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

10.8.2.1 If any of the checkpoints below indicate a problem, re-analysis is required.

- ✓ Check that qualitative identification is done properly.
- ✓ Check surrogate recoveries against Project Specific Requirements (PSR). In the absence of PSR, default to in-house QC limits.
- ✓ Check that all samples results are integrated properly and results over calibration range are diluted and re-analyzed within the calibration range.
- ✓ Where manual integration was performed, check that it was done properly and documentation was retained in accordance to EMAX-DM01 (Section for manual integration).
- ✓ Check that saturated peak(s) are diluted and quantitated properly.
- ✓ Check that suspected carry-overs are re-analyzed and results are reported accordingly.
- ✓ Check that discrete peaks (other than column bleeds) are reported according to project requirement.

10.8.2.2 Review the attached logs that they are properly filled.

10.8.2.3 Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.8.2.4 Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.3 Submit the analytical folder for secondary review.

10.9 **Preventive Maintenance**

10.9.1 Perform daily routine check and record it in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back in control. Refer to Form 8015DFM for daily routine maintenance check points. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction that may affect data quality.

10.9.2 The table below is a list of preventive maintenance activities that are essential to consider in performing this SOP.

Maintenance Activity	Description	Frequency
Autosampler	Inspect and clean syringe. Check autosampler response.	Daily prior to analysis
Verification	Check instrument parameters to ensure normal operating conditions. Change liner as necessary. Check instrument performance (e.g., Daily calibration check, instrument blank).	Daily prior to analysis
Documentation	Record all instrument maintenance performed in the instrument maintenance log.	Daily prior to analysis
System Cleaning	Remove dust from fans and vent covers, inspect and clean inlet and detector. Check septa and replace as necessary.	Every 6 months or as necessary
Complete Inspection	Perform general inspection of the complete system Inspect autosampler cabling and configuration setting. Inspect column, change if necessary (6 mo. or as needed)	Once a year

10.9.3 Maintain an inventory of instrument parts and supplies for routine maintenance.

11.0 QUALITY CONTROL

11.1 Analytical Batch QC

11.1.1 Initial Calibration must be established and verified by daily continuing calibration as described in Appendix 1.

11.1.2 Analytical batch must consist of a valid ICAL, QC Samples and field samples bracketed with opening and closing DCC every 12-hour analytical sequence, unless other frequency is prescribed by the project.

11.1.3 A record must be established that the analytical instrument is free from contamination prior to any analysis. This can be achieved by analyzing a solvent blank and identifying its result as instrument blank.

11.2 Preparation Batch QC

11.2.1 A preparation batch consists of a MB, LCS, MS/MSD and ≤ 20 field samples.

11.2.2 For water samples, use organic-free water for MB and LCS.

11.2.3 For soil samples, use organic-free sand for MB and LCS.

11.2.4 Prepare, analyze and control QC samples as required by the project. In the absence of PSR, refer to Appendix 1 for Quality Control Procedures.

11.2.5 Surrogate standard must be added to all samples, including quality control samples (e.g., method blank, LCS and MS). Check the PSR for QC control Limits. In the absence of PSR default to EMAX-QC Limits.

11.2.6 Solvents and reagents must undergo quality control check prior to use. Refer to EMAX-QC01 for details.

11.2.7 Properly treat all lab wares used in the sample preparation as specified in EMAX-QC07.

11.3 **Method QC**

11.3.1 All analytes reported must have a valid DL, LOD and LOQ as described in EMAX-QA04.

11.3.2 Instrument performance must be checked prior to analysis.

11.3.3 Retention Time Window must be established and updated as prescribed.

11.3.4 All analysts conducting this analysis must demonstrate capability (IDOC/DOC) as described in EMAX-QA05.

11.4 Refer to Appendix 1 for all related Quality Control parameters, frequency and acceptance criteria.

12.0 CORRECTIVE ACTION

12.1 Corrective action for each Quality Control procedure is summarized in Appendix 1.

12.2 **Calibration**

12.2.1 If initial calibration is non-compliant, consider the following suggestions:

12.2.1.1 If RSD > 20%, check each calibration point. If an outlier exists, re-analyze that calibration point.

12.2.1.2 If ICV is not within the expected recovery range, review the chromatogram.

- Bias low results are indicative of bad injection or standard degradation.
- Bias high is indicative of inaccurate standard injection of instrument or contamination.
- Consider preparing a fresh ICV standard and re-analyze the ICV.

12.2.1.3 If problem persists, inform the Supervisor.

12.2.2 If the continuing calibration is non-compliant, consider the suggestions described in correcting the ICV. Consider also the following suggestions to correct the problem:

- Change the liner
- Clean the injection port
- Cut or replace the column
- Clean the detector

- Rule-out leaks by checking all connections
- If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL

12.2.3 If the instrument blank is non-compliant, consider the following suggestions to correct the problem:

12.2.3.1 Check the solvent blank source, e.g. check if same source was used by a similar analysis on a different instrument to rule out solvent contamination.

12.2.3.2 Bake the GC column for at least 15 min.

12.2.3.3 Re-calculate the data and/or re-analyze the extract, if any of the above checks reveal a problem.

12.2.3.4 If problem persists, inform the Supervisor prior to re-analysis.

12.3 **Surrogates**

12.3.1 If surrogates are non-compliant, and are not due to matrix effects, consider the following suggestions to correct the problem:

12.3.1.1 Check that the surrogate peak is properly integrated.

12.3.1.2 Check for calculation errors and that the concentrations of the surrogate solutions are correct.

- High recoveries may be due to co-eluting matrix interference, examine the sample chromatogram.
- Low recoveries may be due to bad injection during preparation process and/or analytical process.

12.3.1.3 Check instrument performance to determine if it is within acceptable guidelines.

12.4 **Preparation Batch QC**

12.4.1 For insufficient amount of sample(s), inform the supervisor immediately for further action.

12.4.2 if method blank is non-compliant, consider the following suggestion to possibly correct the problem:

- Check the sample results. If sample results are non-detected, you may report the result upon concurring with the PM.
- If sample results are not reportable, determine the source of contamination and correct the problem. Re-analyze method blank and all samples processed with the contaminated blank.

12.4.3 If LCS is non-compliant, consider the following suggestions to possibly correct the problem:

- Check the results, if standard degradation is apparent, prepare a fresh standard and perform the corrective action as described in Appendix 1.
- If the LCS result is bias high, the solvent of the standard may have evaporated. Prepare a fresh standard and perform the corrective action as described in Appendix 1.
- Check instrument performance to determine if it is within acceptable guidelines.
- Re-analyze the extract if any of the above checks a reveal a problem.

- Otherwise, re-extract all samples associated with the non-compliant LCS with a new set of QC samples.

12.5 A Non-Conformance Report (NCR) is required when any of the following circumstances occur.

- Anomalies, other than specified in Appendix 1, are observed.
- Sample is out of technical holding time.

12.5.1 Refer to EMAX-QA08 for NCR details.

13.0 POLLUTION PREVENTION

13.1 Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2 Prepare all standards in fume hoods.

14.0 WASTE MANAGEMENT

14.1 No samples may be dumped on the laboratory sink.

14.2 Separate and properly identify all unused expired analytical standards for proper disposal.

14.3 Place all wastes generated during analytical process in properly labeled satellite waste containers for proper collection.

14.4 Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1 **Definition of Terms**

15.1.1 Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belongs to the same chemical family, and which are analyzed together.

15.1.2 Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

15.1.2.1 **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.

15.1.2.2 **Analytical Batch –** is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.3 Detection Limit (DL) – is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

15.1.4 Limit of Detection (LOD) – is defined as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

15.1.5 Limit of Quantitation (LOQ) – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

15.1.6 Safety Data Sheet (SDS) – is a written information concerning a chemical physical properties, toxicity, health hazards, fire hazard and reactivity data including storage, spill and handing precautions.

15.1.7 Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.8 Calibration Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.9 Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.1.10 Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.

15.1.11 Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.

15.1.12 Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.

15.1.13 Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

15.1.14 Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.15 Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.16 Matrix – is a component or form of a sample.

15.1.17 Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.

15.1.18 Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

15.1.19 Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard; used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.

15.1.20 Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.

15.2 **Application of EMAX QC Procedures**

15.2.1 The procedures and QC criteria summarized in this SOP applies to all projects when performing Diesel Range Organics Analysis by GC. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

15.3 **Department of Defense (DoD) Projects**

15.3.1 Samples from DoD sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, is applied.

15.4 **Department of Energy (DoE) Projects**

15.4.1 Samples from DoE sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoE Quality Systems for Analytical Services (QSAS), latest update, is applied.

16.0 REFERENCES

16.1 US EPA Method 8015C, Revision 3, February 2007

16.2 US EPA Method 8015D, Revision 4, June 2003

16.3 US EPA Method 8015B, Revision 2, December 1996

16.4 US EPA Method 8000B, Revision 2, December 1996

16.5 EMAX Quality Systems Manual, as updated

17.0 APPENDICES

17.1 **Tables**

17.1.1 Table 1 Established DL, LOD and LOQ

17.2 **Figures**

17.2.1 Figure 1 Peak Evaluation Technique

17.2.2 Figure 2A Typical DRO Chromatogram

17.2.3 Figure 2B Typical JP5 & Motor Oil Chromatogram

17.2.4 Figure 2C Typical n-Alkane Chromatogram

17.2.5 Figure 3A Typical Diesel Initial Calibration Summary

17.2.6 Figure 3B Typical JP5 & Motor Oil Initial Calibration Summary

17.2.7 Figure 4A Typical Diesel Continuing Calibration Summary

17.2.8 Figure 4B Typical JP5 & Motor Oil Continuing Calibration Summary

17.2.9 Figure 5 Typical Raw Data

17.2.10 Figure 6 Typical Sample Result Summary

17.2.11 Figure 7 Typical LCS/LCSD Summary

17.2.12 Figure 8 Typical MS/MSD Summary

17.2.13 Figure 9 Typical Case Narrative

17.3 **Appendices**

17.3.1 Appendix 1 Summary of Quality Control Procedures

17.3.2 Appendix 2 Demonstration of Capability

17.4 **Forms**

17.4.1 8015DFS Sample Preparation Log

17.4.2 8015DFA Analytical Run Log

17.4.3 8015DFM Instrument Maintenance Log

Table 1: ESTABLISHED DL, LOD AND LOQ

Parameter	Water (mg/L)			Soil (mg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ
Diesel (Total)	0.025	0.05	0.1	2.5	5	10
Diesel (C10-C24)	0.025	0.05	0.1	2.5	5	10
Diesel (C10-C25)	0.025	0.05	0.1	2.5	5	10
Diesel (C10-C28)	0.025	0.05	0.1	2.5	5	10
JP5	0.025	0.05	0.1	2.8	5	10
5W30	0.025	0.05	0.1	2.5	5	10
10W30	0.025	0.05	0.1	-	-	-
Bromobenzene	0.05	0.1	0.2	5.0	10	20
Hexacosane	0.0125	0.025	0.05	1.25	2.5	5

Figure 1: PEAK EVALUATION TECHNIQUE

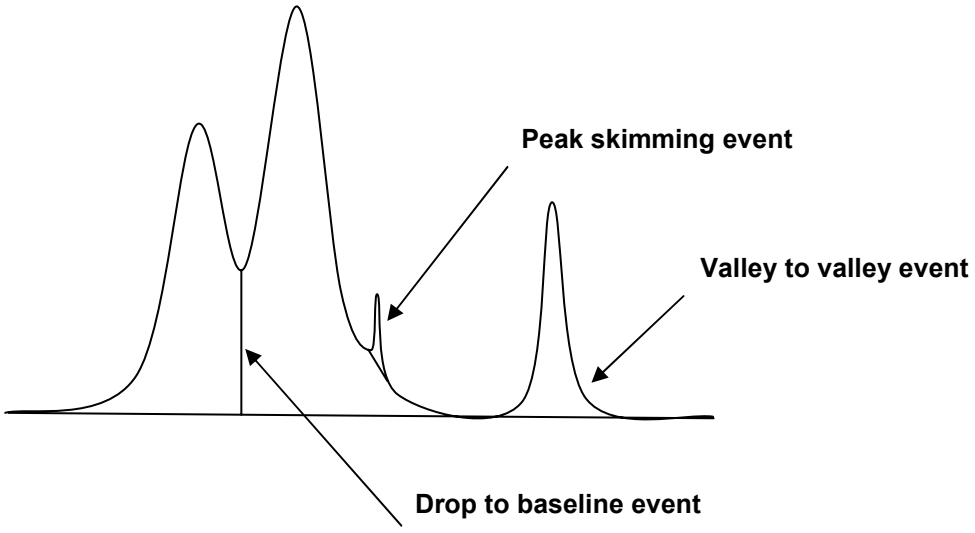


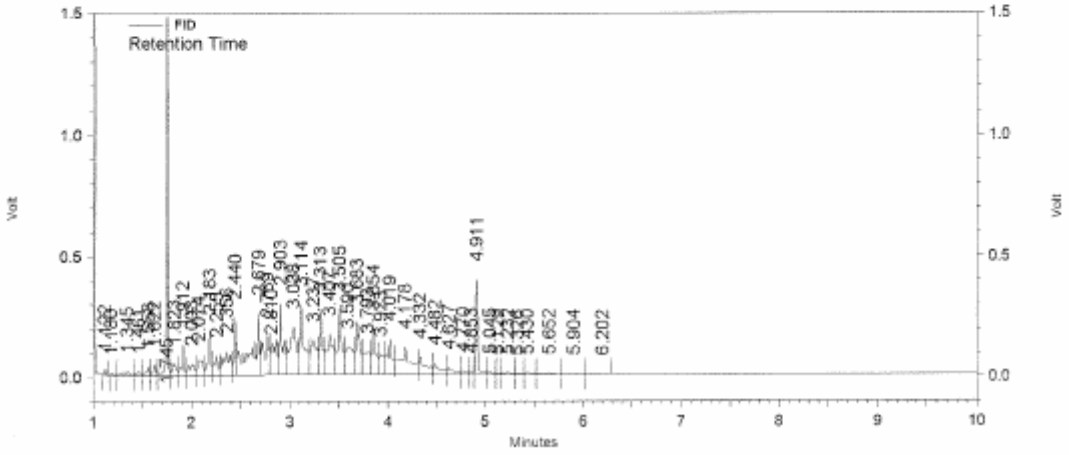
Figure 2A: TYPICAL DRO CHROMATOGRAM

METHOD 8015 by GC/FID
EMAX Laboratories, Inc.

Inst. Name: : D5 (Offline)
File : D:\Projects\EZC331\Data\LC28\LC28011.dat
Method : D:\Projects\EZC331\Method\2017 METHODS\DSD5C28.met
Sequence: : D:\Projects\EZC331\Sequence\LC28.seq
Sample ID : IDSD5C28001 DSL 500/80/20PPM
Acquired : 03/29/17 21:11:22
Printed : 06/19/17 15:17:28
User : CZhao

FID Results

Name	Retention Time	Area	Average RF	ESTD conc. [ppm]
BROMOBENZENE	1.745	1550952	19006.39231	81.602
HEXACOSANE	4.911	496166	22321.13616	22.229
DIESEL(TOTAL)		14542060	26778.65690	543.047
DIESEL(C10-C24)		13913905	25127.20014	553.739
DIESEL(C10-C28)		14024454	25377.61786	552.631
DIESEL(C10-C25)		13958042	25248.85905	552.819
DIESEL(C9-C24)		14132869	26015.56633	543.247
DIESEL(C9-C25)		14177006	26137.22524	542.407
DIESEL(C10-C36)		14134943	25410.79243	556.257
DIESEL(C10-C40)		14134943	25410.79243	556.257
Totals		115065340		4504.233



Software Version: Version 3.3.1

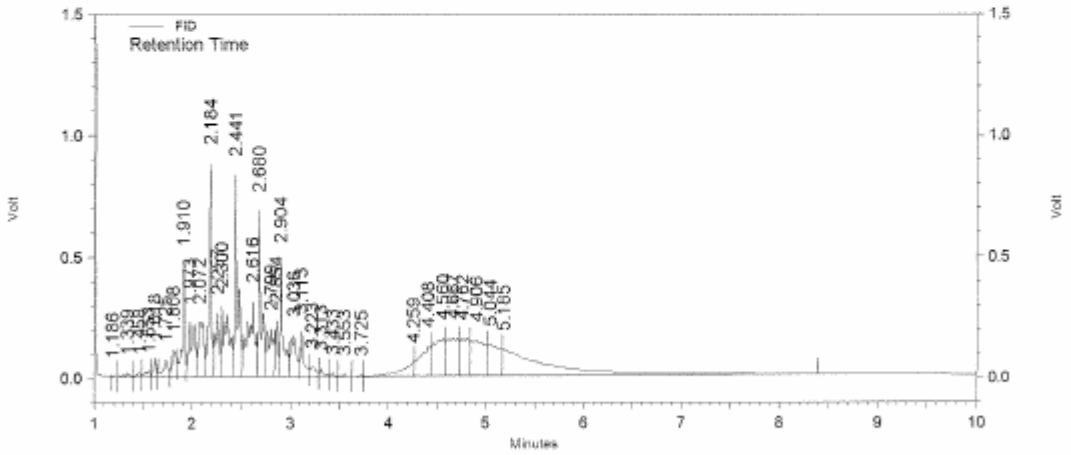
Figure 2B: TYPICAL JP5 & MOTOR OIL CHROMATOGRAM

METHOD 8015 by GC/FID
EMAX Laboratories, Inc.

Inst. Name: : D5 (Offline)
File : D:\Projects\EZC331\Data\LC28\LC28020.dat
Method : D:\Projects\EZC331\Method\2017 METHODS\DSD5C28.met
Sequence: : D:\Projects\EZC331\Sequence\LC28.seq
Sample ID : IDSD5C28003 JP5/5W30 500/500PPM
Acquired : 03/29/17 23:42:06
Printed : 06/19/17 15:18:11
User : CZhao

FID Results

Name	Retention Time	Area	Average RF	ESTD conc. [ppm]
JP5(C8-C18)		14662745	33414.61889	438.812
M.OIL(C18-C36)		10941015	23856.69344	458.614
M.OIL(C24-C36)		8321980	20641.25678	403.172
M.OIL(C24-C40)		8321980	20641.25678	403.172
Totals		42247720		1703.771



Software Version: Version 3.3.1

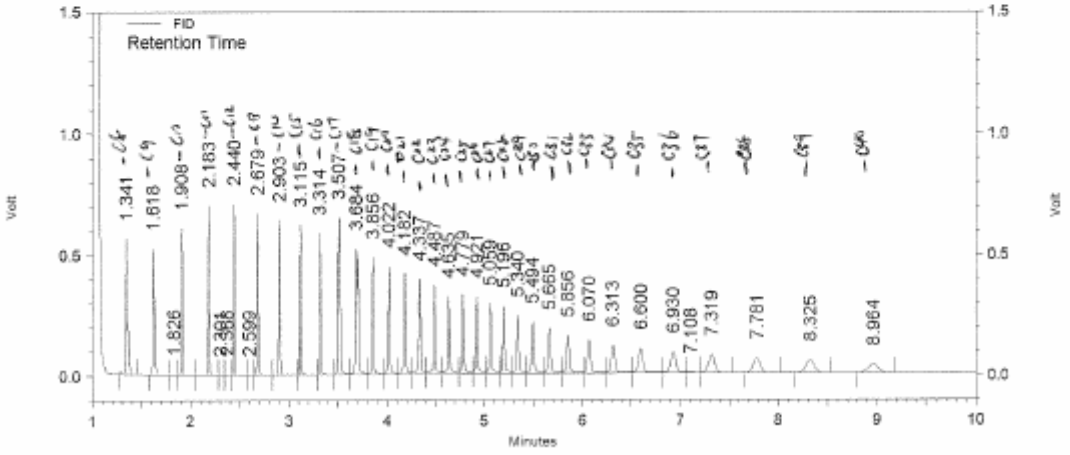
Figure 2C: TYPICAL n-ALKANE CHROMATOGRAM

METHOD 8015 by GC/FID
EMAX Laboratories, Inc.

Inst. Name: : D5 (Offline)
File : D:\Projects\EZC331\Data\LC28\LC28003.dat
Method : D:\Projects\EZC331\Method\2017 METHODS\DSD5C28.met
Sequence: : D:\Projects\EZC331\Sequence\LC28.seq
Sample ID : DRO-01
Acquired : 03/29/17 18:57:32
Printed : 06/19/17 15:18:26
User : CZhao

FID Results

Name	Retention Time	Area	Average RF	ESTD conc. [ppm]
BROMOBENZENE				0.000 BDL
HEXACOSANE	4.921	424588	22321.13616	19.022
DIESEL(TOTAL)		15467068	26778.65690	577.589
DIESEL(C10-C24)		9016646	25127.20014	358.840
DIESEL(C10-C28)		10257523	25377.61786	404.196
DIESEL(C10-C25)		9016646	25248.85905	357.111
DIESEL(C9-C24)		9020885	26015.56633	346.750
DIESEL(C9-C25)		9020885	26137.22524	345.136
DIESEL(C10-C36)		12767078	25410.79243	502.427
DIESEL(C10-C40)		13988721	25410.79243	550.503
Totals		88980040		3461.573



Software Version: Version 3.3.1

Figure 3A: TYPICAL DIESEL INITIAL CALIBRATION SUMMARY

INITIAL CALIBRATION
METHOD M8015

Lab Name : EMAX Inc
Instrument ID : D5
GC Columm : HP5
Column size ID : 30MX0.32MM 0.25UM
LFID & Datetime: LC28004A 03/29/17 19:14
LFID & Datetime: LC28005A 03/29/17 19:30
LFID & Datetime: LC28006A 03/29/17 19:47
LFID & Datetime: LC28007A 03/29/17 20:04
LFID & Datetime: LC28008A 03/29/17 20:21
LFID & Datetime: LC28009A 03/29/17 20:37
LFID & Datetime: LC28010A 03/29/17 20:54
CONC UNIT: ppm

COMPOUND	CONC X	CALIBRATION FACTORS (AREA)/UNIT 1.00X	2.00X	10.00X	20.00X	100.00X	300.00X	600.00X	MEAN	%RSD
DIESEL(TOTAL)	5.00	24541	22722	22488	25778	30053	30402	31466	26778.7	14.2
DIESEL(C10-C24)	5.00	22416	21360	21504	24511	28250	28432	29417	25127.2	14.0
DIESEL(C10-C28)	5.00	23159	21722	21578	24538	28379	28631	29636	25377.6	13.6
DIESEL(C10-C25)	5.00	22753	21501	21542	24521	28322	28552	29550	25248.9	13.9
DIESEL(C9-C24)	5.00	23365	22195	22149	25234	29254	29449	30463	26015.6	14.0
DIESEL(C9-C25)	5.00	23702	22336	22187	25244	29327	29569	30596	26137.2	13.8
DIESEL(C10-C36)	5.00	23159	21722	21578	24538	28415	28726	29737	25410.8	13.7
DIESEL(C10-C40)	5.00	23159	21722	21578	24538	28415	28726	29737	25410.8	13.7
SURROGATE	X	0.00X	1.00X	2.00X	3.00X	4.00X	5.00X	11.00X	MEAN	%RSD
BROMOBENZENE	20.00	0	18130	17939	18670	20717	19114	19469	19006.4	5.4
HEXACOSANE	5.00	0	21767	20750	22349	24398	21621	23042	22321.1	5.7

DSD5C28.MET

Figure 3B: TYPICAL JP5 & MOTOR OIL INITIAL CALIBRATION SUMMARY

INITIAL CALIBRATION
METHOD M8015

Lab Name : EMAX Inc
Instrument ID : D5
GC Column : HP5
Column size ID : 30MX0.32MM 0.25UM
LFID & Datetime: LC28013A 03/29/17 21:44
LFID & Datetime: LC28014A 03/29/17 22:01
LFID & Datetime: LC28015A 03/29/17 22:18
LFID & Datetime: LC28016A 03/29/17 22:35
LFID & Datetime: LC28017A 03/29/17 22:51
LFID & Datetime: LC28018A 03/29/17 23:08
CONC UNIT: ppm

COMPOUND	CONC X	CALIBRATION FACTORS (AREA)/UNIT 2.00X	10.00X	20.00X	100.00X	200.00X	300.00X	MEAN	%RSD
JP5(C8-C18)	5.00	36729	31862	32627	33929	33996	31345	33414.6	5.8
M.OIL(C18-C36)	5.00	26134	24905	22519	23869	23691	22023	23856.7	6.3
M.OIL(C24-C36)	5.00	23315	21831	19343	20291	20233	18835	20641.3	8.0
M.OIL(C24-C40)	5.00	23315	21831	19343	20291	20233	18835	20641.3	8.0

DSD5C28.MET

Figure 4A: TYPICAL DIESEL CONTINUING CALIBRATION SUMMARY

CONTINUE CALIBRATION
METHOD M8015

Lab Name : EMAX Inc
Instrument ID : D5
GC Column : HP5
Column size ID : 30MX0.32MM 0.25UM
Mid Conc Init LFID & Datetime: LC28007A 03/29/2017 20:04
Conc Cont LFID & Datetime: LC28046A 03/30/2017 06:59
CONC UNIT : ppm

COMPOUND	RT MINUTES	RT WINDOW FROM	RT WINDOW TO	TRUE CONC	AVERAGE CF	RESULT AREA	RESULT CONC	%D	QL	%D LIMITS
DIESEL(TOTAL)	NA	NA	NA	500.0	26778.7	14051854	524.74	5		20
DIESEL(C10-C24)	NA	NA	NA	500.0	25127.2	13195089	525.13	5		20
DIESEL(C10-C28)	NA	NA	NA	500.0	25377.6	13300006	524.08	5		20
DIESEL(C10-C25)	NA	NA	NA	500.0	25248.9	13268804	525.52	5		20
DIESEL(C9-C24)	NA	NA	NA	500.0	26015.6	13618860	523.49	5		20
DIESEL(C9-C25)	NA	NA	NA	500.0	26137.2	13692575	523.87	5		20
DIESEL(C10-C36)	NA	NA	NA	500.0	25410.8	13312960	523.91	5		20
DIESEL(C10-C40)	NA	NA	NA	500.0	25410.8	13312960	523.91	5		20
SURROGATE	MINUTES	FROM	TO	TRUECON	CF	AREA	CONC	%D	QL	LIMITS
BROMOBENZENE	1.745	1.735	1.755	80.0	19006.4	1627262	85.62	7		20
HEXACOSANE	4.963	4.769	5.157	20.0	22321.1	454848	20.38	2		20

Figure 4B: TYPICAL JP5 & MOTOR OIL CONTINUING CALIBRATION SUMMARY

CONTINUE CALIBRATION
METHOD M8015

Lab Name : EMAX Inc
Instrument ID : D5
GC Column : HP5
Column size ID : 30MX0.32MM 0.25UM
Mid Conc Init LFID & Datetime: LC28016A 03/29/2017 22:35
Conc Cont LFID & Datetime: LC28047A 03/30/2017 07:16
CONC UNIT : ppm

COMPOUND	RT MINUTES	RT WINDOW FROM	RT WINDOW TO	TRUE CONC	AVERAGE CF	RESULT AREA	RESULT CONC	%D	QL	%D LIMITS
JP5(C8-C18)	NA	NA	NA	500.0	33414.6	15383110	460.37	-8		20
M.OIL(C18-C36)	NA	NA	NA	500.0	23856.7	10916024	457.57	-8		20
M.OIL(C24-C36)	NA	NA	NA	500.0	20641.3	9346513	452.81	-9		20
M.OIL(C24-C40)	NA	NA	NA	500.0	20641.3	9346513	452.81	-9		20

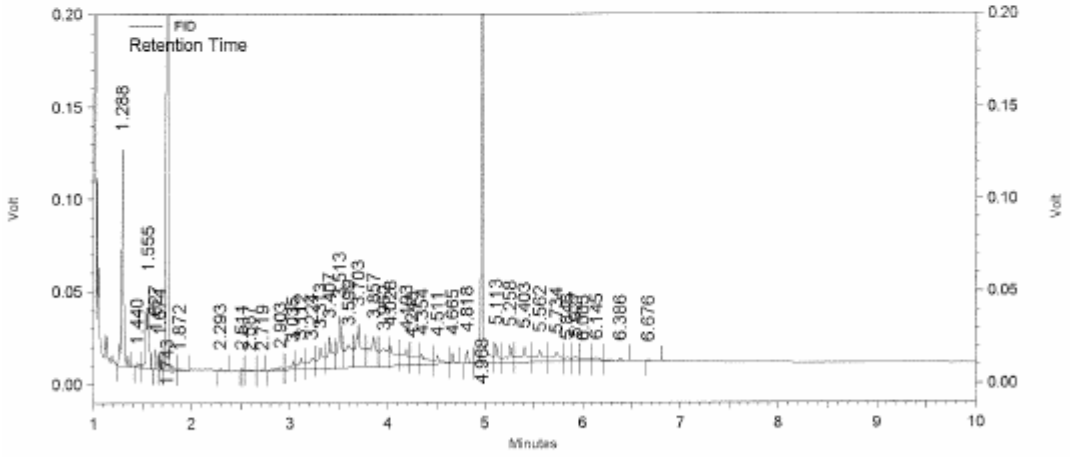
Figure 5: TYPICAL RAW DATA

METHOD 8015 by GC/FID
EMAX Laboratories, Inc.

Inst. Name: : D5 (Offline)
File : D:\Projects\EZC331\Data\LC28\LC28043.dat
Method : D:\Projects\EZC331\Method\2017 METHODS\DSD5C28M.met
Sequence: : D:\Projects\EZC331\Sequence\LC28.seq
Sample ID : 17C173-03
Acquired : 03/30/17 06:09:05
Printed : 06/30/17 11:17:41
User : CZhao

FID Results

Name	Retention Time	Area	Average RF	ESTD conc. [ppm]
BROMOBENZENE	1.743	1461045	19006.39231	76.871
HEXACOSANE	4.968	493024	22321.13616	22.088
DIESEL(TOTAL)		1113398	26778.65690	41.578
DIESEL(C13-C22)		620701	25127.20014	24.702
M.OIL(C23-C32)		178785	20641.25678	8.662
Totals		3866953		173.901



Software Version: Version 3.3.1

Figure 6: TYPICAL SAMPLE RESULT SUMMARY

METHOD 3550B/8015B
PETROLEUM HYDROCARBONS BY EXTRACTION

Client	: XYZ, INC.	Date Collected:	03/23/17
Project	: CLEAN PROJECT	Date Received:	03/25/17
Batch No.	: YYMXXX	Date Extracted:	03/28/17 10:02
Sample ID:	ABCD-03	Date Analyzed:	03/30/17 06:09
Lab Samp ID:	MXXX-03	Dilution Factor:	1
Lab File ID:	LC28043A	Matrix :	SOIL
Ext Btch ID:	DSC041S	% Moisture :	3.3
Calib. Ref.:	LC28034A	Instrument ID :	D5

PARAMETERS	RESULTS (mg/kg)	LOQ (mg/kg)	DL (mg/kg)	LOD (mg/kg)
DIESEL	26	10	2.6	5.2
MOTOR OIL	9.0J	21	2.6	5.2

SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
BROMOBENZENE	79.5	103.4	76.9	50-130
HEXACOSANE	22.8	25.85	88.4	40-160

Parameter	H-C Range"
Diesel	C13-C22
Motor Oil	C23-C32

Figure 7: **TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN PROJECT
BATCH NO.: YYMXXX
METHOD: METHOD 3550B/8015B

MATRIX:	SOIL			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	MBLK1S				
LAB SAMP ID:	DSC041SB	DSC041SL	DSC041SC		
LAB FILE ID:	LC28036A	LC28037A	LC28038A		
DATE EXTRACTED:	03/28/1710:02	03/28/1710:02	03/28/1710:02	DATE COLLECTED:	NA
DATE ANALYZED:	03/30/1704:11	03/30/1704:27	03/30/1704:44	DATE RECEIVED:	03/28/17
PREP. BATCH:	DSC041S	DSC041S	DSC041S		
CALIB. REF:	LC28034A	LC28034A	LC28034A		

ACCESSION:

PARAMETER	BLNK RSLT (mg/kg)	SPIKE AMT (mg/kg)	BS RSLT (mg/kg)	BS % REC	SPIKE AMT (mg/kg)	BSD RSLT (mg/kg)	BSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Diesel	ND	500	467	93	500	465	93	1	38-132	30

SURROGATE PARAMETER	SPIKE AMT (mg/kg)	BS RSLT (mg/kg)	BS % REC	SPIKE AMT (mg/kg)	BSD RSLT (mg/kg)	BSD % REC	QC LIMIT (%)
Bromobenzene	100	85.0	85	100	83.1	83	50-130
Hexacosane	25.0	23.4	93	25.0	22.6	90	60-130

Figure 8: **TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN PROJECT
BATCH NO.: YYMXXX
METHOD: METHOD 3550B/8015B

MATRIX:	SOIL			% MOISTURE:	18.2
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	ABCD-01				
LAB SAMP ID:	MXXX-12	MXXX-12M	MXXX-12S		
LAB FILE ID:	LC28049A	LC28050A	LC28051A		
DATE EXTRACTED:	03/28/1710:02	03/28/1710:02	03/28/1710:02	DATE COLLECTED:	03/24/17
DATE ANALYZED:	03/30/1707:50	03/30/1708:07	03/30/1708:24	DATE RECEIVED:	03/25/17
PREP. BATCH:	DSC041S	DSC041S	DSC041S		
CALIB. REF:	LC28046A	LC28046A	LC28046A		

ACCESSION:

PARAMETER	SMPL RSLT (mg/kg)	SPIKE AMT (mg/kg)	MS RSLT (mg/kg)	MS % REC	SPIKE AMT (mg/kg)	MSD RSLT (mg/kg)	MSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Diesel	ND	611	472	77	611	542	89	14	38-132	30

SURROGATE PARAMETER	SPIKE AMT (mg/kg)	MS RSLT (mg/kg)	MS % REC	SPIKE AMT (mg/kg)	MSD RSLT (mg/kg)	MSD % REC	QC LIMIT (%)
Bromobenzene	122	95.8	78	122	99.4	81	50-130
Hexacosane	30.6	27.1	89	30.6	28.4	93	40-160

Figure 9: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project: CLEAN PROJECT

SDG : YYMXXX

METHOD 3550B/8015B
PETROLEUM HYDROCARBONS BY EXTRACTION

A total of eight (8) soil samples were received on 03/25/17 to be analyzed for Petroleum Hydrocarbons by Extraction in accordance with Method 3550B/8015B and project specific requirements.

Holding Time
Samples were analyzed within the prescribed holding time.

Calibration
Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source (ICV). Continuing calibration (CCV) verifications were carried out on a frequency specified by the project. All calibration requirements were within acceptance criteria. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank
Method blank was prepared and analyzed at the frequency required by the project. For this SDG, one (1) method blank was analyzed. DSC041SB - result was compliant to project requirement. Refer to sample result summary form for details.

Lab Control Sample
Lab control sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of LCS/LCD was analyzed. DSC041SL/DSC041SC were within LCS limits. Refer to LCS summary form for details.

Matrix QC Sample
Matrix spike sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of MS/MSD was analyzed. Diesel was within MS QC limits in MXXX-12M/S. Refer to Matrix QC summary form for details.

Surrogate
Surrogates were added on QC and field samples. All surrogate recoveries were within QC limits. Refer to sample result summary forms for details.

Sample Analysis
Samples were analyzed according to prescribed analytical procedures. Results were evaluated in accordance to project requirements. For this SDG, all quality control requirements were met.
Samples MXXX-02, -03 and -05 displayed heavier fuel pattern.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC Procedure	Frequency	Acceptance Criteria	Corrective Action	1st Rvw	2nd Rvw
Minimum five-point initial calibration	Initially; as needed	Linear - mean RSD ≤ 20%	Correct the problem then repeat initial calibration		
Second-source calibration verification	After initial calibration	Within ± 25% of expected value	Correct the problem then repeat initial calibration		
Initial calibration verification	Daily, before sample analysis	Within ± 20% of expected value	Correct the problem then repeat initial calibration		
Calibration verification	Every 12 hours of analysis time and at the end of analysis sequence	Within ± 20% of expected value	Correct the problem then repeat initial calibration verification and re-analyze all samples since last successful calibration verification		
Method Blank (MB)	One MB per preparation batch	No analyte detected > ½ LOQ	Re-prep and re-analyze method blank and all samples processed with the contaminated blank		
Lab Control Sample (LCS)	One LCS per preparation batch	Within EMAX QC Limits	Re-prep and re-analyze the LCS and all associated samples		
Surrogate spike	Every sample, spiked sample, standard, and method blank	Within EMAX QC Limits	Correct the problem then re-extract and re-analyze sample		
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every 20 project samples per matrix	Refer to EMAX QC Limits	None		
Chromatogram	All sample results	Within calibration range NO SATURATED PEAK(s)	Dilute and re-analyze all samples over the calibration range Diluted and re-analyzed all samples demonstrating saturated peak(s) even if the total integrated peaks do not exceed the calibration range.		
Comments: Refer to PSR for flagging criteria.			Reviewed By:		
			Date:		

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
DIESEL
METHOD: EPA 8015B

MATRIX: SOIL

Analytical SOP: EMAX-8015D Rev. 6
Sample Preparation SOP: EMAX-3550 Rev. 5
Conc Unit: mg/Kg
Sample Amount(g): 10
Volume Extracted(ml): 10

Date Extracted: 01/16/17
Extracted by: J. Villena
Date Analyzed: 01/18/17
Analyzed by: W. Zhao

PARAMETER	LA18007A DSA009SL	LA18008A DSA009SC	LA18009A DSA009SX	LA18010A DSA009SY	TV	Ave. Conc	Ave. %Rec	SD	RSD	QC Criteria	Comments
DIESEL(TOTAL)	507	503	485	485	500	495	99	11.8	2	20 - 160	Passed
DIESEL(C10-C24)	502	502	484	485	500	493	99	10.5	2	20 - 160	Passed
DIESEL(C10-C28)	503	503	484	485	500	494	99	10.5	2	20 - 160	Passed
DIESEL(C10-C25)	503.7	503.4	485.0	485.5	500	494.4	99	10.5	2	20 - 160	Passed
BROMOBENZENE	86	88	86	89	100	87	87	1.4	2	60 - 130	Passed
HEXACOSANE	21.5	22.3	21.7	22.8	25	22.1	88	0.6	3	60 - 140	Passed
PARAMETER	LA18011A J5A009SL	LA18012A J5A009SC	LA18013A J5A009SX	LA18014A J5A009SY	TV	Ave. Conc	Ave. %Rec	SD	RSD	QC Criteria	Comments
JP5	444.63	454.83	446.34	477.02	500	455.70	91	14.90	3	30 - 160	Passed
5W30	436.98	452.77	437.68	451.82	500	444.81	89	8.66	2	30 - 160	Passed

8015FS: SAMPLE PREPARATION LOG (Page 1)

EMAX LABORATORIES, INC.

EXTRACTION LOG
for
TPH

Page 1

SOP	Rev. #
☐ EMAX-3520	
☐ EMAX-3540	
☐ EMAX-3550	
☐ EMAX-3580	
☐ EMAX-8015AZ	
☐ EMAX-AK 102/103	
☐ EMAX-LUFT E	
☐ EMAX-	

Note: For samples and relevant QCs/Standards extracted, refer to attached extraction sequence.

Book #: EDS-062
Preparation Batch:
Matrix:
Micropipette ID:
Micropipette ID:

Standards	ID	Amount Added (ml)
Surrogate		
Surrogate		
LCS/MS		
LCS/MS		

Reagent	Lot # / ID
CH_2Cl_2	
Na_2SO_4	
HCl	
Silica Sand	
Silica Gel	
Reagent Water	
pH strip	

TUNING

Sonicator #	Reading

Lab Sample ID	Sonicator #	Concentrator #

Concentrator	Water Bath Temperature Setting (°C)	Thermometer Reading (°C)
1		
2		
3		
4		
5		
6		

Comments:

Test thermometer = SVOC-T1

Prepared By: Standard Added By:
Witnessed By: Checked By:
Extract Received By: Extraction Location:
Disposal Date: Disposed By:

8015FS: SAMPLE PREPARATION LOG (Page 2)

Prep BatchID	Lab SampleID	Aliquot	Unit	DateTime	Ve(ml)	Exp Amt	Exp Ve(ml)	PrepFctr	Comments

Balance ID: 10304418. Calibration check was verified prior to use. Ve=extract volume PrepFctr=(ExpAmt/Aliquot)(Ve/ExpVe)

Extraction Started @ Prepared By:

Extraction Ended @ Checked By:

8015FA: **ANALYTICAL RUN LOG**

EMAX LABORATORIES, INC.

ANALYSIS RUN LOG
for
TPH

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Comments:

Book #:	AD5-
Instrument No.:	D5
Analytical Sequence:	
Method File:	
Analytical Batch:	

SOP #	Rev. #
☐ EMAX-8015D	5
☐ EMAX-AK102/AK103	3
☐ EMAX-	

STANDARDS ID	Conc (mg/L)
ICAL	
☐ Diesel	
☐ Motor Oil ☐ JP5	
CH_2Cl_2	
DSL DCC	
JP5/5W30 DCC	
Alaska DCC	
Arizona DCC	

ELECTRONIC DATA ARCHIVAL	
Location	Date
☐ EZCHROM_GC6890N	
☐ External Hard Drive	

Analyzed By:

Date:

8015FM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: D5 Month Year: 201_ Book # MD5-004

Date							
Column Head Pressure, N_2 (21.09 psi)							
Column Temperature							
FID lit up							
FID signal within specs (4.5 - 6)							
Autosampler							
Check make up gas flow, N_2 (25 ml/min)							
Run instrument blank							
Computer Date/Time							
Computer Paper							
Analyst Initial							

Trouble Shooting							
Problem(s) Encountered							
Return to Control (Analyst Initial)							

Code	Problems Encountered	Code	Corrective Action

STANDARD OPERATING PROCEDURES

GASOLINE RANGE ORGANICS

SOP No.:	EMAX-8015G	Revision No.	5	Effective Date:	24-Jan-14
Prepared By:	Lucita Arzadon			Date:	01-24-14
Approved By:	Kenette Pimentel QA Manager			Date:	01.24.14
Approved By:	Caspar Pang Laboratory Director			Date:	01-24-14

Control Number: 8015G-05-

1.0 SCOPE AND APPLICATION

1.1. This method is applicable for analyzing purgeable petroleum hydrocarbons bracketed by the range of alkanes from C_6 to C_{10} as gasoline range organics (GRO) in samples of various matrices (i.e. soils, water, sludge). Other range of alkanes may be applied (e.g., C_5 to C_{10}, C_6 to C_{12}, etc.) or other purgeable fuel patterns such as JP4 and Stoddard may be analyzed provided that qualitative identification and quantitative determination is properly established.

1.2. This SOP is an adaptation of SW846 Method 8015C. This SOP is also applicable to SW846 Method 8015B and Method 8015D.

2.0 SUMMARY OF METHOD

2.1. A known amount of sample is purged by inert gas into a trap and retains the purgeable organic compounds. The trap is back flushed into the GC system equipped with flame ionization detector (FID). The instrument is calibrated with a gasoline standard. Hydrocarbon markers (C_5 to C_{12}) are analyzed to determine elution time. Quantitation of typical GRO is based on C_6 to C_{10} alkane range.

2.2. **Interferences**

2.2.1. Glassware can be a potential source of contamination. They must be scrupulously cleaned prior to use.

2.2.2. Carry-over from a highly concentrated sample can be a potential source of contamination. Instrument performance must be observed keenly for possible carry-over. If this is apparent, inject solvent blank until no trace of carry-over is observed.

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.

3.1.2. Established DL, LOD and LOQ are as follows:

Analyte	Water (µg/L)			Soil 1 gm to 5 mL (µg/Kg)			Soil 5 gm to 5 mLMeOH (µg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ	DL	LOD	LOQ
Gasoline	5	10	20	20	40	100	350	500	1000
JP4	8.4	10	20	-	-	-	250	500	1000

Analyte	Water (µg/L)			Soil 1 gm to 5 mL (µg/Kg)			Soil 5 gm to 5 mLMeOH (µg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ	DL	LOD	LOQ
Stoddard	10	20	50	-	-	-	250	500	1000
Bromofluorobenzene	2.5	5	20	10	20	100	50	100	1000
1,1,1-Trifluorotoluene	2.5	5	20	10	20	100	50	100	1000

4.0 DYNAMIC RANGE

4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (See Section 9.3.1). Dilute and re-analyze all samples having results above this range for proper quantitation.

4.2. The lowest quantifiable range of diluted samples is equal to the lowest calibration point (See Section 9.3.1). Lower the dilution factor and re-analyze all diluted samples analyzed below this range for proper quantitation.

5.0 SAMPLE HOLDING TIME & PRESERVATION

5.1. **Holding Time**

5.1.1. Aqueous Samples

5.1.1.1. Analyze preserved samples within 14 days from sampling date.

5.1.1.2. If pH > 2, analyze samples within 7 days from sampling date.

5.1.2. Soil Samples

5.1.2.1. Samples received in encore are extracted with methanol within 48 hours from sampling time and analyzed within 14 days from sampling date unless otherwise specified by the project.

5.1.2.2. Sample received in jars are extracted with methanol and analyzed within 14 days from sampling date.

5.1.2.3. Analyze soil samples (1gram direct purge) within 14 days from sampling date.

5.2. **Preservation**

5.2.1. Water samples received are expected to be contained in 40 ml vial with teflon-lined septa preserved at pH < 2 with HCl with zero head space.

Note: The size of any bubble caused by degassing upon cooling the sample should not exceed 6 mm.[1]

5.2.2. Store samples and extracts at ≤ 6°C.

[1] Referenced from SW846 Method 5030C, Section 8.1.

6.0 ASSOCIATED SOPs

6.1. EMAX-5030 Purge and Trap

6.2. EMAX-5035 Purge and Trap, Closed System

6.3. EMAX-DM01 Data Flow and Review

6.4. EMAX-QA04 Detection Limit

6.5. EMAX-QA05 Training

6.6. EMAX-QA08 Corrective Action

6.7. EMAX-QC01 Quality Control of Chemicals

6.8. EMAX-QC02 Analytical Standard Preparation

6.9. EMAX-QC07 Glassware Cleaning

6.10. EMAX-SM03 Waste Disposal

6.11. EMAX-SM04 Analytical and QC Sample Labeling

7.0 SAFETY

7.1. Read all SDS of chemicals listed in this SOP.

7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.

7.3. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amounts of water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS & REAGENTS

8.1. **Instruments and Supplies**

Purge and Trap System (Concentrator)	LSC 2000 Tekmar (GC39)/OI4560 or equivalent (GC55)
Autosampler	Archon or equivalent
Gas Chromatography	HP 5890 Series II, GC with FID
Column	DB5 – 30m x .53 mm, 1.5 µm thickness, or equivalent
Gas	ultra-high purity helium, ultra-high purity hydrogen, hydrogen generator unit, compressed air
Syringes	5 ml Luerlok hypodermic gas-tight
Microsyringes	Hamilton or equivalent
Data System	EZ-Chrom

Purge Trap	Supelco, Trap "G" or equivalent

8.2. **Chemicals and Reagents**

8.2.1. Methanol, purge and trap

8.2.2. Organic-free Reagent Water

8.2.3. Organic-free Ottawa Sand

9.0 STANDARDS

9.1. **Stock Standard**

9.1.1. Purchase stock standards or equivalent as certified solutions traceable to NIST standards.

Name	Source	Solvent	Conc. (µg/mL)	Intended Use
Unleaded Gas Comp. Standard	Restek	Methanol	2,500	Calibration
Stoddard Solvent Standard	Restek	Methanol	10,000	Calibration
JP4 Standard	Restek	Methanol	50,000	Calibration
Cert. BTEX Unleaded Gas	AccuStandard	Methanol	5,000	ICV/LCS/MS
Stoddard Solvent	AccuStandard	Methanol	20,000	ICV/LCS/MS
JP4 Jet Fuel	AccuStandard	Methanol	20,000	ICV/LCS/MS

9.2. **Intermediate Standards**

9.2.1. Intermediate and working standards are prepared according to EMAX-QC02.

9.2.2. All standards should be transferred in inert vials labeled with ID from standard preparation logbook, date of expiration, concentration, and stored with minimal headspace at -10°C to -20°C.

9.2.3. Intermediate standards are prepared only for Stoddard and JP4 as follows:

Stock Standard			Solvent	Intermediate Standard	
Standard Name	Conc. (µg/ml)	Aliquot (µl)		Final Vol. (ml)	Final Conc. (mg/L)
Stoddard Solvent (1st source)	10,000	500	Methanol	2	2,500
Stoddard Solvent (2nd source	20,000	250	Methanol	2	2,500
JP4 Standard (1st source)	50,000	100	Methanol	2	2,500
JP4 Standard (2nd source)	20,000	250	Methanol	2	2,500

9.3. **Calibration Standards**

9.3.1. Initial Calibration Standards

9.3.1.1. Gasoline

Prepare initial calibration standards in 5 ml organic- free water with concentrations as suggested below:

ICAL Standard	Aliquot (µl)		Final Conc. (µg/L) Gasoline /Surrogate
	Gasoline Std. (2,500 mg/L)	Surrogate BFB/TFT (100 mg/L)	
1	0.04	0.5	20/10
2	0.1	1	50/20
3	0.2	1.5	100/30
4	1	2	500/40
5	2	2.5	1000/50
6	4	3.75	2000/75
7	5 µl	5	2500/100

9.3.1.2. Stoddard

Prepare initial calibration standards in 5 ml organic- free water with concentrations as suggested below:

ICAL Standard	Aliquot (µL)		Final Conc. (µg/L) Stoddard / Surrogate
	Stoddard Std. (2,500 mg/L)	Surrogate BFB/TFT (100 mg/L)	
1	0.04	0.5	20/10
2	0.1	1	50/20
3	0.2	1.5	100/30
4	1	2	500/40
5	2	2.5	1000/50
6	3	3	1500/60

9.3.1.3. JP4

Prepare initial calibration standards in 5 ml organic- free water with concentrations as suggested below:

ICAL Standard	Aliquot (µL)		Final Conc. (µg/L) JP4/ Surrogate
	JP4 Std. (2,500 mg/L)	Surrogate BFB/TFT (100 mg/L)	
1	0.04	0.5	20/10
2	0.1	1	50/20
3	0.2	1.5	100/30

ICAL Standard	Aliquot (µL)		Final Conc. (µg/L) JP4/ Surrogate
	JP4 Std. (2,500 mg/L)	Surrogate BFB/TFT (100 mg/L)	
4	1	2	500/40
5	2	2.5	1000/50
6	4	3.75	2000/75
7	5	5	2500/100

9.3.2. Initial Calibration Verification Standards

Prepare initial calibration verifications standards (using second-source standards) in 5 ml organic-free water with concentrations as suggested below:

ICV Standard	Intermediate Std. (2nd source)		Surrogate BFB/TFT (100 mg/L) Aliquot in µL	Final Conc. (µg/L) Analyte / Surrogate
	Concentration (mg/L)	Aliquot (µL)		
Gasoline	5000	1	2.5	1000/50
Stoddard	2500	2	2.5	1000/50
JP4	2500	2	2.5	1000/50

9.3.3. Continuing Calibration Standard

Using intermediate standards, prepare daily calibration standards in a syringe with 5 ml of organic-free water as suggested below:

DCC Standard	Intermediate Std. (1st source)		Surrogate BFB/TFT (100 mg/L) Aliquot in µl	Final Conc. (µg/L) Analyte / Surrogate
	Concentration (mg/L)	Aliquot (µl)		
Gasoline	2500	2	2.5	1000/50
Stoddard	2500	2	2.5	1000/50
JP4	2500	2	2.5	1000/50

9.4. **LCS/Matrix Spike Standard**

9.4.1. Use second-source intermediate standards for laboratory control standard (LCS) and matrix spike (MS) standards. Refer to Section 9.2.3 for Stoddard and JP4. Use Gasoline standard from AccuStandard (5,000 mg/L) for gasoline spike.

9.5. **Surrogate Standards**

9.5.1. Purchase stock standards or equivalent as certified solutions traceable to NIST standards.

Name	Source	Solvent	Conc. (µg/mL)
Bromofluorobenzene / TFT	Ultra Scientific	Methanol	2000

9.5.2. Prepare intermediate surrogate standard as follows:

Stock Standard			Solvent	Intermediate Standard	
Standard Name	Conc. (µg/ml)	Aliquot (µl)		Final Vol. (ml)	Final Conc. (mg/L)
Bromofluorobenzene / TFT	2000	100	Methanol	2	100

9.6. **Retention Time Window Standard**

9.6.1. Purchase the following standards or equivalent.

Name	Source	Conc. (mg/L)
GRO	AccuStandard	2000
2-Methylpentane	Supelco	2000
1,2,4-Trimethylbenzene	Ultra Scientific	5000 *
DRO	AccuStandard	2000

* Dilute to 2000 mg/L before use for retention time analysis.

9.7. Other concentration levels may be used as appropriate.

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Prepare aqueous samples in accordance with EMAX-5030. Add surrogates to all samples to yield 40µg/L prior to purging unless otherwise specified by the project.

10.1.2. Prepare soil samples in accordance with EMAX-5035. Add surrogates to all samples to yield 40µg/Kg prior to purging unless otherwise specified by the project.

10.1.3. Prepare LCS/LCD by spiking a reagent blank to yield 500 µg/L spike standard unless otherwise specified by the project. Add surrogate at the same level as the samples.

10.1.4. Prepare MS/MSD sample using the assigned matrix QC sample similarly as the LCS sample.

10.2. **Instrument Parameters**

10.2.1. Initially, set the instrument parameters as suggested in the table below.

10.2.2. Gas Chromatographic Condition

Carrier gas flow (column) helium	9-10 ml/min
Make up gas (He)	20-21 ml/min

Air	60 psi
Helium	80 psi
Hydrogen	50 psi

Temperature Settings:					
Inst. GC 39			**Inst. GC 55**		
Injector Temperature: OFF Detector Temperature: 235 °C			Injector Temperature: 150 °C Detector Temperature: 235 °C		
Temp °C	Hold Time,min	Rate, °C/min	Temp °C	Hold Time,min	Rate, °C/min
35	6	0	35	6	0
70	0	8	70	0	8
120	3	5	120	3	5
245	4	30	245	3	30

10.2.3. Purge and Trap Condition

	Inst. GC 39	**Inst. GC 55**
Purge	10 mins at 40 °C	15 mins at 30 °C
Dry Purge / Desorb Preheat	3 mins at 170 °C	1 min at 180 °C
Desorb	2 mins at 180 °C	2 mins at 180 °C
Bake	14 mins at 185 °C	10 mins at 220 °C

10.2.4. Optimize the instrument for its intended use.

10.2.5. Record the instrument operating condition on the instrument maintenance log and post the latest instrument parameter setup in front of the instrument for ease when performing the instrument routine check.

10.2.6. When instrument parameter setup requires change due to instrument optimization document the change as described in Section 10.2.5.

10.3. **Calibration**

10.3.1. Initial Calibration (ICAL)

10.3.1.1. Analyze initial calibration standards (Refer to Section 9.3.1 for standard preparation) as described in Section 10.4.

10.3.1.2. Refer to Section 10.5 for calculation.

10.3.1.3. Acceptance criteria are specified in Appendix 1.

10.3.2. Initial Calibration Verification (ICV)

10.3.2.1. After establishing ICAL, analyze the ICV standard (Refer to Section 9.3.2 for standard preparation) to verify the validity of the ICAL. Refer Appendix 1 for acceptance criteria. If non-compliant refer to Section 12 for corrective action.

10.3.3. Retention Time Window Check (RTW)

10.3.3.1. Spike 5 ml water reagent with 0.5 µL of 2000 mg/L RTW standard.

10.3.3.2. Analyze the RTW standard after every ICAL to set carbon cut-off ranges (e.g, C_6 - C_{10}, C_5 - C_{12}). Refer to Section 10.4.3.

10.3.4. Continuing Calibration (DCC)

10.3.4.1. Analyze the DCC standard (Refer to Section 9.3.3 for standard preparation) to verify the validity of the ICAL. Refer to Appendix 1 for acceptance criteria. If non-compliant refer to Section 12 for corrective action.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. Analyze instrument blank to ensure that the instrument is free from contamination.

10.4.1.2. Analyze DCC to check ICAL validity.

10.4.1.3. Analyze Method Blank to check for preparation batch contamination.

10.4.1.4. Analyze Lab Control Sample to check accuracy.

10.4.1.5. Analyze Lab Control Sample Duplicate (if required by the project).

10.4.1.6. Analyze samples to a maximum number of 12-hour runs or as specified by the project.

10.4.1.7. Analyze matrix spikes (MS/MSD) per project requirement.

10.4.1.8. Record the analytical sequence in the Analytical Run Log.

10.4.1.9. Print instrument sequence before and after the analysis run and attach to the analytical run. Document any changes that occurred during the process.

10.4.2. Identification and Quantitation

10.4.2.1. Identification is based on pattern recognition. Hence, compare sample chromatograms to reference hydrocarbons standard chromatograms for their response hydrocarbon range and peak distribution to determine the most probable petroleum product.

10.4.2.2. All peaks eluting within the established RT window identifies the GRO, JP4 and Stoddard.

10.4.2.3. When the elution profile of a sample does not match that of gasoline standard, JP4 or Stoddard, but falls within the retention time window, quantitate results as gasoline range organics (GRO) and denote the observed deviation in case narrative.

10.4.2.4. Quantitation is achieved by the summation of all peaks in the chromatogram minus the solvent peak, and the sample result is calculated using Equation 10.5.3.

10.4.2.5. Integrate the total peak area response and quantitate the total area by using the ACF of Gasoline, JP4 or Stoddard (see section 10.5.1).

10.4.2.6. When manual integration is necessary follow the procedures described in EMAX-DM01 Section 4.4.3.

10.4.3 Retention Time Windows (RTW)

10.4.3.1 Establishing RTW

10.4.3.1.1 Run RTW standard over a period of 72 hours.

10.4.3.1.2 Calculate the Standard Deviation (SD) of absolute retention time obtained for each analyte (Use Equation 10.5.1.2).

10.4.3.1.3 The width of RTW is defined by ±3XSD obtained from Section 10.4.3.1.2.

10.4.3.2 Evaluating RTW

10.4.3.2.1 If the SD is equal to 0.00, default to the previous study until historical data is obtained to define the RTW for the current instrument.

10.4.3.2.2 For new instruments, use the established retention time from another instrument having the same instrument parameters (e.g. detector, temperature program and column.) If there are no instruments with the same instrument parameter, use 0.03 minutes as the default RTW until historical data is obtained to define the RTW for the current instrument parameters condition.

10.4.3.3 Application of RTW

10.4.3.3.1 Establish the center of absolute retention time for each analyte to include the surrogate(s) from the daily calibration check at the beginning of the analytical shift then apply the established RTW.

10.4.3.3.2 Whenever the observed retention time is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing analyses.

10.4.3.4 Updating RTW

10.4.3.4.1 Re-establish the RTW as described in Section 10.4.3.1 when any of the following conditions occur.

- Yearly RTW update
- Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of the RTW)
- Major instrument maintenance (e.g. replacements of detector or column, temperature program change, etc.)

10.4.3.4.2 If the calculated new RTW is significantly narrower than the previously established RTW, default to the previously established RTW.

10.4.4 Sample Result Evaluation

10.4.4.1 Check surrogate recoveries against project specific requirement (PSR). In the absence of PSR, default to in-house QC procedures described in Appendix 1.

10.4.4.2 Dilute and re-analyze samples having concentrations greater than the highest calibration range.

10.4.4.3 Dilute and re-analyze samples having saturated peak(s) within $C_6 - C_{10}$. See Figure 1 for typical saturated peak.

10.4.4.4 Re-analyze samples suspected of carry-over from a proceeding sample that has high concentration.

10.4.4.5 Report discrete peak(s) observed as required by the project. Column bleed subtraction is not generally required in GRO analysis.

10.5 **Calculations**

10.5.1 Initial Calibration

10.5.1.1 **Calculate the Calibration Factor (CF)**

$$CF = \frac{R_a}{C_k} \qquad \text{Eq.-10.5.1.1}$$

where:

CF *- is the calibration factor*

R_a *- is the analyte response measured in peak area*

C_k *- is the known concentration of the analyte in µg/L (water); µg/Kg (soil)*

10.5.1.1 **Calculate the Standard Deviation**

$$SD = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \overline{x})^2}{N-1}} \qquad \text{Eq.-10.5.1.2}$$

where:

SD *- is the standard deviation*

x_i *- is the result at the i^{th} measurement*

$\overline{x}$ *- is the mean*

N *- is the number of measurements*

10.5.1.1 **Calculate the Percent Relative Standard Deviation (%RSD)**

$$\%RSD = \left[\frac{SD}{ACF}\right] 100 \qquad \text{Eq.-10.5.1.3}$$

where:

%RSD *- is the percent relative standard deviation*

SD *- is the standard deviation*

ACF *- is the average calibration factor*

10.5.1.1 **Calculate the Average Calibration Factor (ACF)**

$$ACF = \frac{\sum CF}{N}$$ Eq.-10.5.1.4

where:

ACF - is the average calibration factor

∑CF - is the summation of the calibration factors

N -is the number of calibration points

10.5.2 Calculate the Percent Difference for DCC from ACF

$$\%D = \frac{|Ck - Cf|}{Ck} * 100$$ Eq.-10.5.2

where:

%D - is the percent difference DCC from the ACF

Ck - is the known concentration of analyte, in µg/L

Cf - is the concentration found, in µg/L

10.5.3 Calculate Sample Results

10.5.3.1 **Water Samples**

$$C = \left[\frac{R_a}{ACF}\right]\left[\frac{V_e}{S_a}\right]DF$$ Eq.-10.5.3.1

10.5.3.1 **Soil Samples**

$$C = \left[\frac{R_a}{ACF}\right]\left[\frac{V_e}{\left(S_a\right)\left(\%S\right)}\right]DF$$ Eq.-10.5.3.2

where:

C - is the concentration of analyte in µg/L (water), µg/Kg (soil)

R_a - is the analyte response measured in peak area

ACF - is the average calibration factor from initial standard calibration

V_e - is the purgeable volume in ml

S_a - is the sample amount in ml (water); g (soil)

DF - is the dilution factor

%S - is the percent solid of the sample

10.5.4 Accuracy and Precision

10.5.4.1 **Percent Recovery**

$$\%\ R = \left[\frac{(C_f - C_s)}{C_o}\right] 100 \qquad \text{Eq.-10.5.4.1}$$

where:

% - *is the percent recovery*

C_f - *is the concentration found, in µg/L*

C_s - *is the concentration of the sample in µg/L (water); in µg/Kg (soil). For LCS, C_s=0*

C_o - *is the known concentration of spiked solution*

10.5.4.1 **Relative Percent Difference**

$$\%\ RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \qquad \text{Eq.-10.5.4.2}$$

where:

RPD - *is the relative percent difference*

C_1 - *is the measured concentration of the first sample aliquot*

C_2 - *is the measured concentration of the second sample aliquot*

10.6 **Data Reduction**

10.6.1 Make a copy of the analytical run log and sample preparation log.

10.6.2 Print a copy of the raw data and the QC report.

10.6.3 Highlight the data to be reported.

10.6.4 Collate the reportable raw data separating the QC results from the sample results.

10.6.5 Keep all other data generated with the analytical folder marked with “For record only”.

10.7 **Report Generation**

10.7.1 Generate the method.txt file using WDBX[2].exe.

10.7.2 Generate the sample results using F1NVX[2].exe

10.7.3 Generate the QC summary using QCVX[2].exe

10.7.4 Generate Lab Chronicle using LABCHRNX[2].exe

10.7.5 Generate the Case Narrative using CN1.exe

10.8 **Data Review**

[2] X – version number of the program.

10.8.1 Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analyses.

- Case Narrative
- Lab Chronicle
- Sample Results
- LCS/LCSD Summary
- MS/MSD Summary
- ICAL Summary
- ICV Summary
- DCC Summary
- Analytical Log
- Sample Preparation Log
- Non-Conformance Report (if any)

10.8.2 Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- ✓ Check that qualitative identification is done properly.
- ✓ Check that surrogate recoveries against Project Specific Requirement (PSR). In the absence of PSR, default to in-house QC limits.
- ✓ Check that sample results are integrated properly and results over calibration range are diluted and re-analyzed within the calibration range.
- ✓ Where manual integration was performed, check that it was done properly and documentation was retained in accordance to EMAX-DM01 Section 4.4.3.
- ✓ Check that presence of saturated peak(s) are diluted, and quantitated properly.
- ✓ Check that suspected carry-overs are re-analyzed and results are reported accordingly.
- ✓ Check that discrete peaks [other than column bleeds] are reported according to project requirement.
- ✓ If any of the above checkpoints indicate a problem, re-analysis is required.

10.8.3 Review the attached logs that they are properly filled.

10.8.4 Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.8.5 Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.6 Submit the analytical folder for secondary review.

10.9 **Preventive Maintenance**

10.9.1 On a daily basis, perform the checks listed in the instrument maintenance log prior to sample analysis. Refer to Form 8015GFM – Instrument Maintenance Log.

10.9.2 Conduct routine preventive instrument maintenance and document the activity in the instrument-specific maintenance log. Routine maintenance ensures that all equipment are operating under optimum conditions, thus reducing the possibility of instrument malfunction, and consequently affecting sample results. Routine maintenance activities are suggested below:

Maintenance Activity	Description	Frequency
Autosampler	Inspect autosampler needle and check response.	Daily prior to analysis.
Verification	Check gas pressure Check instrument parameters to ensure normal operating conditions. Check instrument performance (e.g., Daily calibration check, instrument blank)	Daily prior to analysis.
Documentation	Record all instrument maintenance performed in the instrument maintenance log.	Daily prior to analysis
System Cleaning	Remove dust from fans and vent covers. Lubricate mechanical parts.	Every 6 months or as necessary
Check Flow Path Components	Change the carrier gas trap(s) and purifier	Once a year or as necessary
Complete Inspection	Perform general inspection of the complete system. Inspect autosampler cabling and configuration setting. Replace column if necessary. Replace worn out parts.	Once a year

10.9.3 Maintain an inventory of instrument parts and supplies for routine maintenance.

11.0 QUALITY CONTROL

11.1. **Sample Preparation**

11.1.1. A preparation batch shall consist of a MB, LCS, MS/MSD and ≤ 20 field samples.

11.1.2. Decontaminate volumetric flasks used for standard preparation with methanol.

11.1.3. All solvents and reagents shall undergo quality control check in the stationary laboratory prior to its use in accordance to EMAX-QC01.

11.2. **Analytical Batch**

11.2.1. Initial Calibration must be established and verified by daily continuing calibration as described in Appendix 1.

11.2.2. Analytical batch shall consist of a valid ICAL, QC samples and field samples bracketed with DCC every 12-hour analytical sequence, unless other frequency is prescribed by the project.

11.2.3. A record must be established that the analytical instrument is free from contamination prior to any analysis. This can be achieved by analyzing a solvent blank and identifying its result as instrument blank.

11.2.4. Organic-free water shall be used for method blank and LCS for water matrix.

11.2.5. Organic-free sand shall be used for method blank and LCS for soil matrix.

11.3. **Method QC**

11.3.1. LOD and LOQ must be established before the analytical procedure can be used and verified according to EMAX-QA04.

11.3.2. Retention Time Window must be established and updated as prescribed.

11.3.3. Demonstration of capability must be established before the analytical procedure can be used.

11.3.4. All analysts conducting this analysis must have established demonstration of capability.

12.0 CORRECTIVE ACTION

12.1. Corrective actions associated with this analytical procedure are described in the Summary of Quality Control Procedures (Refer to Appendix 1).

12.2. **Calibration**

12.2.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:

12.2.1.1. If RSD > 20%, check each calibration point. If an outlier exists, re-analyze that calibration point.

12.2.1.2. If ICV not within the expected recovery range, review the chromatogram.

- Bias low results are indicative of poor purging or standard degradation.
- Bias high is indicative of inaccurate standard injection of instrument or contamination.
- Consider preparing a fresh ICV standard and re-analyze the ICV.

12.2.1.3. If problem persists, inform the Supervisor prior to re-calibration

12.2.2. If the continuing calibration is non-compliant, consider the suggestions described in correcting ICV.

12.2.3. If instrument blank/reagent blank is non-compliant, consider the following suggestions to correct the problem:

12.2.3.1. Check the reagent water source, e.g. check if same source was used by a similar analysis on a different instrument to rule out reagent contamination.

12.2.3.2. Bake the sample concentrator and/or GC column for at least 15 min.

12.2.3.3. Re-calculate the data and/or re-analyze the extract, if any of the above checks reveal a problem.

12.2.3.4. If problem persists, inform the Supervisor prior to re-analysis.

12.3. **Surrogates**

12.3.1. If surrogates are non-compliant, but is not due to matrix effects, consider the following suggestions to correct the problem:

12.3.1.1. Check that the surrogate peak is properly integrated.

12.3.1.2. Check for calculation errors and that the concentrations of the surrogate solutions are correct.

- High recoveries may be due to co-eluting matrix interference, examine the sample chromatogram.
- Low recoveries may be due to a poor purge, check the purge tube with a blank before re-analyzing the sample.

12.3.1.3. Check instrument performance to determine if it is within acceptable guidelines.

12.4. **Sample Preparation QCs**

12.4.1. If method blank is non-compliant, consider the following suggestions to correct the problem:

- Check the sample results. If sample results are non-detect, you may report the result upon concurring with the PM.
- If sample results are not reportable, determine the source of contamination and correct the problem. Reanalyze method blank and all samples processed with the contaminated blank.

12.4.2. If LCS is non-compliant, consider the following suggestions to correct the problem:

- Check for standard degradation. Prepare a new LCS standard.
- Check instrument performance to determine if it is within acceptable guidelines.
- Reanalyze the extract if any of the above checks reveal a problem.
- Otherwise, re-extract all samples associated with the non-compliant LCS with a new set of QC samples.

12.5. A Non-Conformance Report (NCR) is required when any of the following circumstances occur:

- Anomalies, other than specified in Appendix 1, are observed.
- Sample is out of technical holding time.

12.5.1. Refer to EMAX-QA08 for NCR details.

13.0 POLLUTION PREVENTION

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2. Prepare all standards in fume hoods.

14.0 WASTE MANAGEMENT

14.1. No samples may be dumped on the laboratory sink.

14.2. Separate and properly identify all unused expired analytical standards for proper disposal.

14.3. Place all wastes generated during analytical process in properly labeled satellite waste containers for proper collection.

14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. **Definition of Terms**

15.1.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents. Preparation batch is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate. Analytical batch is compose of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.2. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.3. Duplicate Sample – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.4. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.1.5. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.6. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyze to monitor the accuracy of the analytical system.

15.1.7. Matrix – is a component or form of a sample.

15.1.8. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyze to monitor matrix effect on a method's recovery efficiency.

15.1.9. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

15.1.10. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.

15.1.11. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

15.1.12. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.2. **Application of EMAX QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing gasoline range organics unless otherwise other directive is specified by the project requirements.

15.3. **Department of Defense (DoD) and Department of Energy (DOE) Projects**

15.3.1. Samples from DoD and DOE sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, shall be applied

16.0 REFERENCES

16.1. US EPA SW846 Method 8015C, Revision 3, February 2007.

16.2. US EPA SW846 Method 8015D, Revision 4, June 2003.

16.3. US EPA SW846 Method 8015B, Revision 2, December 1996.

16.4. US EPA SW846 Method 8000B, Revision 2, December 1996.

16.5. EMAX Quality Systems Manual, as updated.

17.0 APPENDICES

17.1. **Figures**

17.1.1.	Figure 1	Typical Peak Evaluation
17.1.2.	Figure 2	Typical GRO Chromatogram
17.1.3.	Figure 3	Typical Hydrocarbon Marker Chromatograms
17.1.4.	Figure 4	Typical ICAL Summary
17.1.5.	Figure 5	Typical Continuing Calibration Summary
17.1.6.	Figure 6	Typical Raw Data
17.1.7.	Figure 7	Typical Sample Result Summary
17.1.8.	Figure 8	Typical LCS/LCSD Summary
17.1.9.	Figure 9	Typical MS/MSD Summary
17.1.10.	Figure 10	Typical Case Narrative

17.2. **Appendices**

17.2.1.	Appendix 1	Summary of Quality Control Procedures

17.2.2. Appendix 2 Demonstration of Capability

17.3 Forms

17.3.1 8015GFS Sample Preparation Log

17.3.1 8015GFA Analytical Run Log

17.3.2 8015GFM Instrument Maintenance Log

Figure 1: TYPICAL PEAK EVALUATION

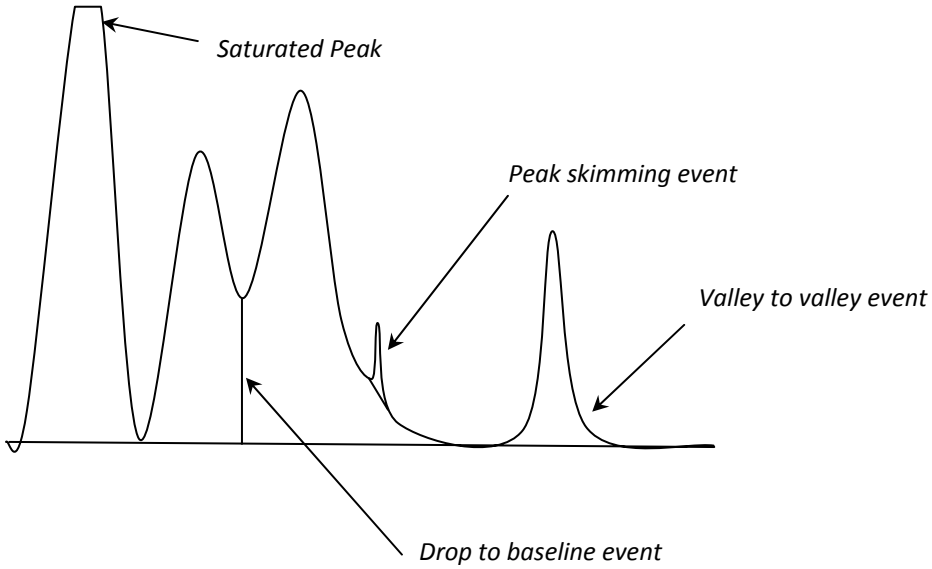


Figure 2: **TYPICAL CHROMATOGRAM**

EPA METHOD 8015 by FID
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea03\ea03.010
Method : c:\ezchrom\methods\vg39a03.met
Sample ID : IVG39A0302 500/40
Acquired : Jan 03, 2014 21:37:01
Printed : Jan 06, 2014 12:19:37
User : SERGIO

Channel A Results

#	Peak Name	Ret.Time(Min)	Area	Ave. CF	ESTD Conc.(PPB)
15	1,1,1-TFT	3.125	984381.0	22722.0	43.32
41	Bromofluorobenzene	10.675	788502.0	17848.0	44.18
G1	GASOLINE(TOTAL)		14063293.0	27233.2	516.40
G2	GRO(C6-C10)		9336308.0	20606.5	453.08
G3	GRO(2MP-124TMB)		9284983.0	20571.2	451.36
G4	GRO(C5-C12)		13063346.0	26615.6	490.81
G5	GRO(C6-C12)		13013487.0	26585.6	489.49
G6	GRO(C5-C10)		9386167.0	20636.5	454.83

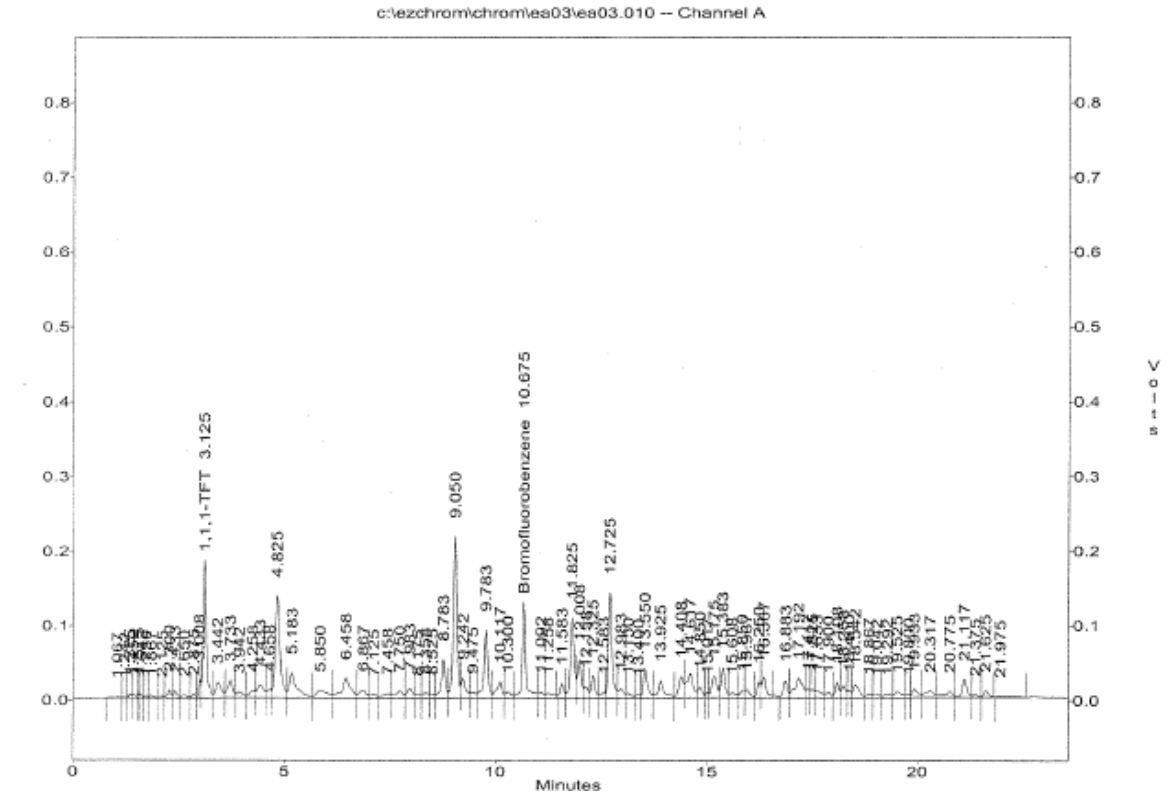


Figure 3: TYPICAL HYDROCARBON MARKER CHROMATOGRAMS

EPA METHOD 8015 by FID
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea03\ea03.013
Method : c:\ezchrom\methods\vg39a03.met
Sample ID : GRO 1UL
Acquired : Jan 03, 2014 23:32:32
Printed : Jan 06, 2014 12:22:28
User : SERGIO

Channel A Results

#	Peak Name	Ret.Time(Min)	Area	Ave. CF	ESTD Conc.(PPB)
--	1,1,1-TFT	3.150	0.0	0.0	0.00
--	Bromofluorobenzene	10.700	0.0	0.0	0.00
G1	GASOLINE(TOTAL)		29925396.0	27233.2	1098.86
G2	GRO(C6-C10)		21311152.0	20606.5	1034.20
G3	GRO(2MP-124TMB)		21311152.0	20571.2	1035.97
G4	GRO(C5-C12)		29925396.0	26615.6	1124.35
G5	GRO(C6-C12)		29925396.0	26585.6	1125.62
G6	GRO(C5-C10)		21311152.0	20636.5	1032.69

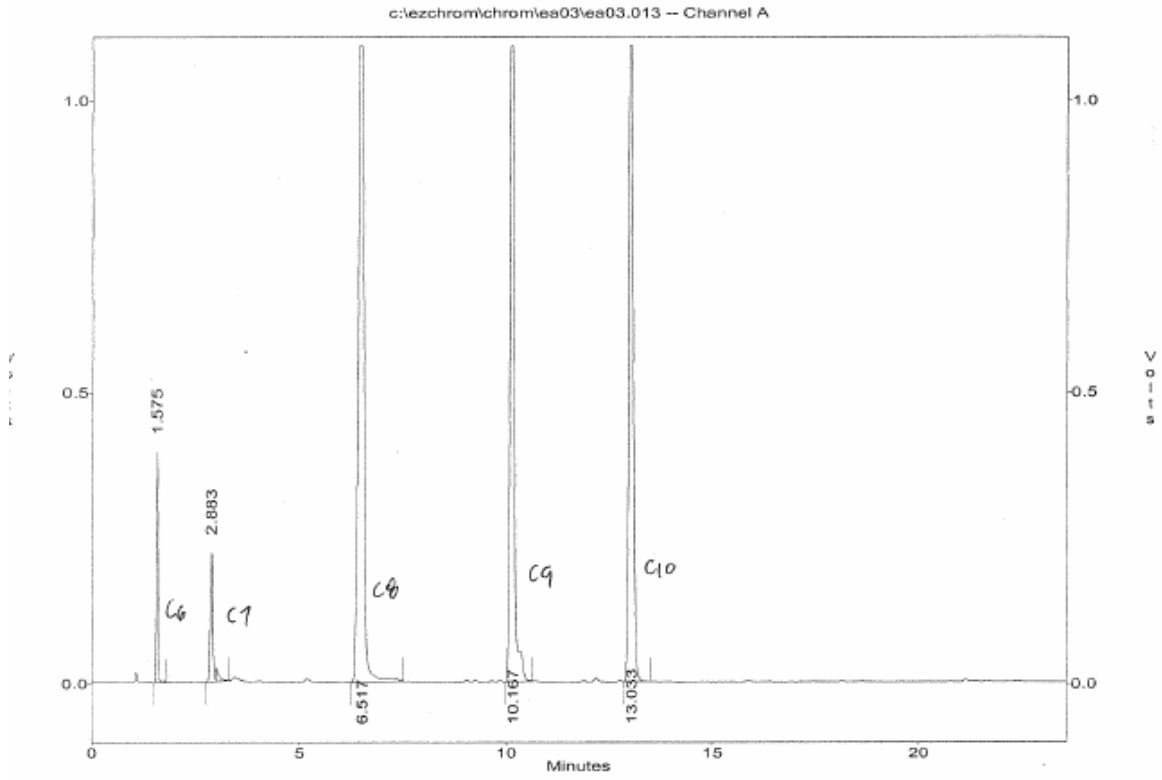


Figure 3: TYPICAL HYDROCARBON MARKER CHROMATOGRAMS (continuation)

EPA METHOD 8015 by FID
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea03\ea03.014
Method : c:\ezchrom\methods\vg39a03.met
Sample ID : 2MP/1,2,4-TMB
Acquired : Jan 04, 2014 00:11:01
Printed : Jan 06, 2014 12:22:38
User : SERGIO

Channel A Results

#	Peak Name	Ret.Time(Min)	Area	Ave. CF	ESTD Conc.(PPB)
--	1,1,1-TFT	3.150	0.0	0.0	0.00
--	Bromofluorobenzene	10.700	0.0	0.0	0.00
G1	GASOLINE(TOTAL)		14307337.0	27233.2	525.36
G2	GRO(C6-C10)		9312826.0	20606.5	451.94
G3	GRO(2MP-124TMB)		0.0	20571.2	0.00
G4	GRO(C5-C12)		14304282.0	26615.6	537.44
G5	GRO(C6-C12)		9312826.0	26585.6	350.30
G6	GRO(C5-C10)		14304282.0	20636.5	693.15

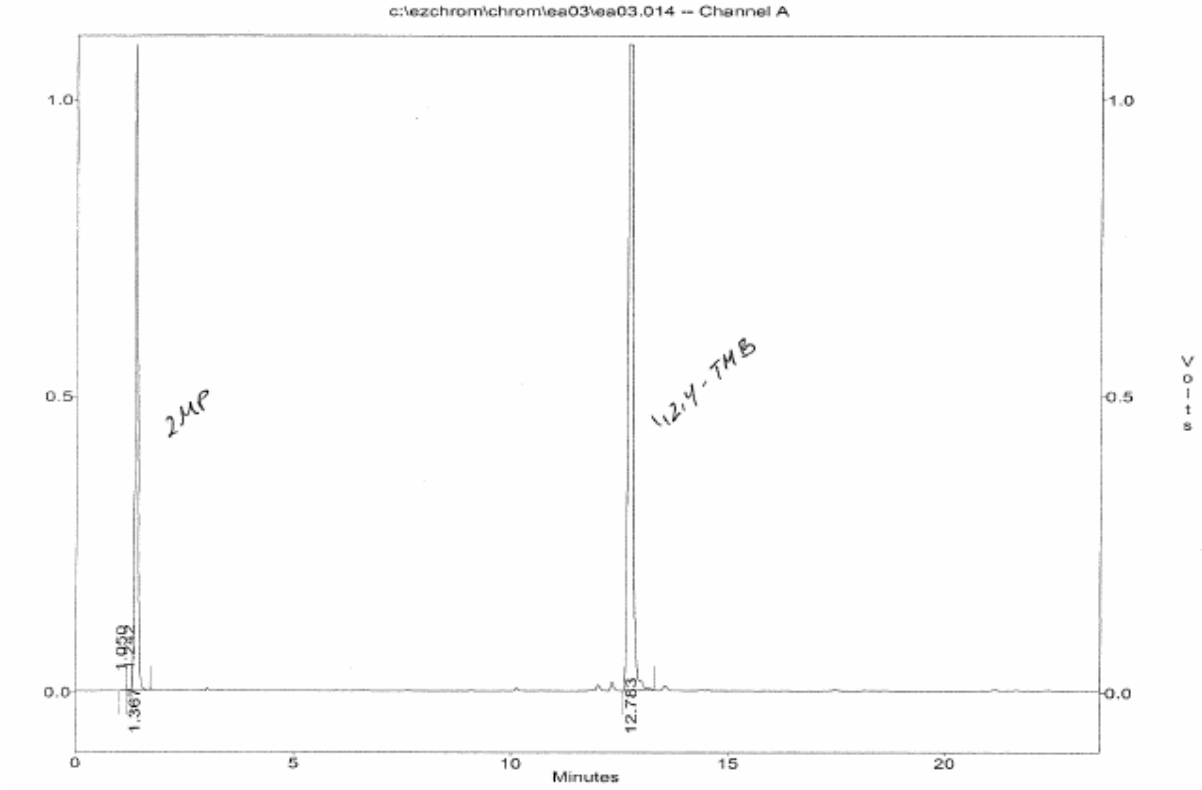


Figure 3: TYPICAL HYDROCARBON MARKER CHROMATOGRAMS (continuation)

EPA METHOD 8015 by FID
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea03\ea03.015
Method : c:\ezchrom\methods\vg39a03.met
Sample ID : PENTANE/NAPHTHALENE
Acquired : Jan 04, 2014 00:49:28
Printed : Jan 06, 2014 12:22:47
User : SERGIO

Channel A Results

#	Peak Name	Ret.Time(Min)	Area	Ave. CF	ESTD Conc.(PPB)
--	1,1,1-TFT	3.150	0.0	0.0	0.00
--	Bromofluorobenzene	10.700	0.0	0.0	0.00
G1	GASOLINE(TOTAL)		17371524.0	27233.2	637.88
G2	GRO(C6-C10)		0.0	20606.5	0.00
G3	GRO(2MP-124TMB)		0.0	20571.2	0.00
G4	GRO(C5-C12)		17347616.0	26615.6	651.78
G5	GRO(C6-C12)		7450149.0	26585.6	280.23
G6	GRO(C5-C10)		9897466.0	20636.5	479.61

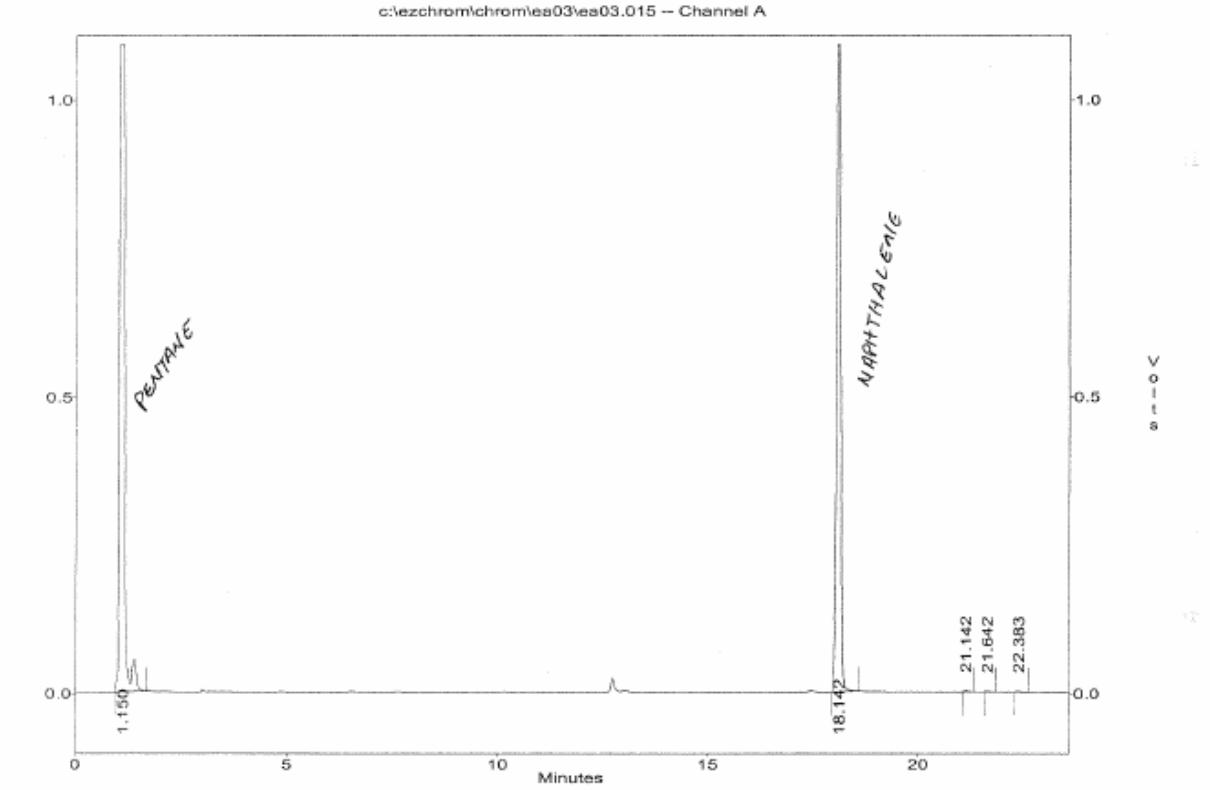


Figure 3: TYPICAL HYDROCARBON MARKER CHROMATOGRAMS (continuation)

EPA METHOD 8015 by FID
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea03\ea03.016
Method : c:\ezchrom\methods\vg39a03.met
Sample ID : UNDECANE/DODECANE
Acquired : Jan 04, 2014 01:27:55
Printed : Jan 06, 2014 12:23:00
User : SERGIO

Channel A Results

#	Peak Name	Ret.Time(Min)	Area	Ave. CF	ESTD Conc.(PPB)
--	1,1,1-TFT	3.150	0.0	0.0	0.00
--	Bromofluorobenzene	10.700	0.0	0.0	0.00
G1	GASOLINE(TOTAL)		7098382.0	27233.2	260.65
G2	GRO(C6-C10)		0.0	20606.5	0.00
G3	GRO(2MP-124TMB)		0.0	20571.2	0.00
G4	GRO(C5-C12)		6837192.0	26615.6	256.89
G5	GRO(C6-C12)		6837192.0	26585.6	257.18
G6	GRO(C5-C10)		0.0	20636.5	0.00

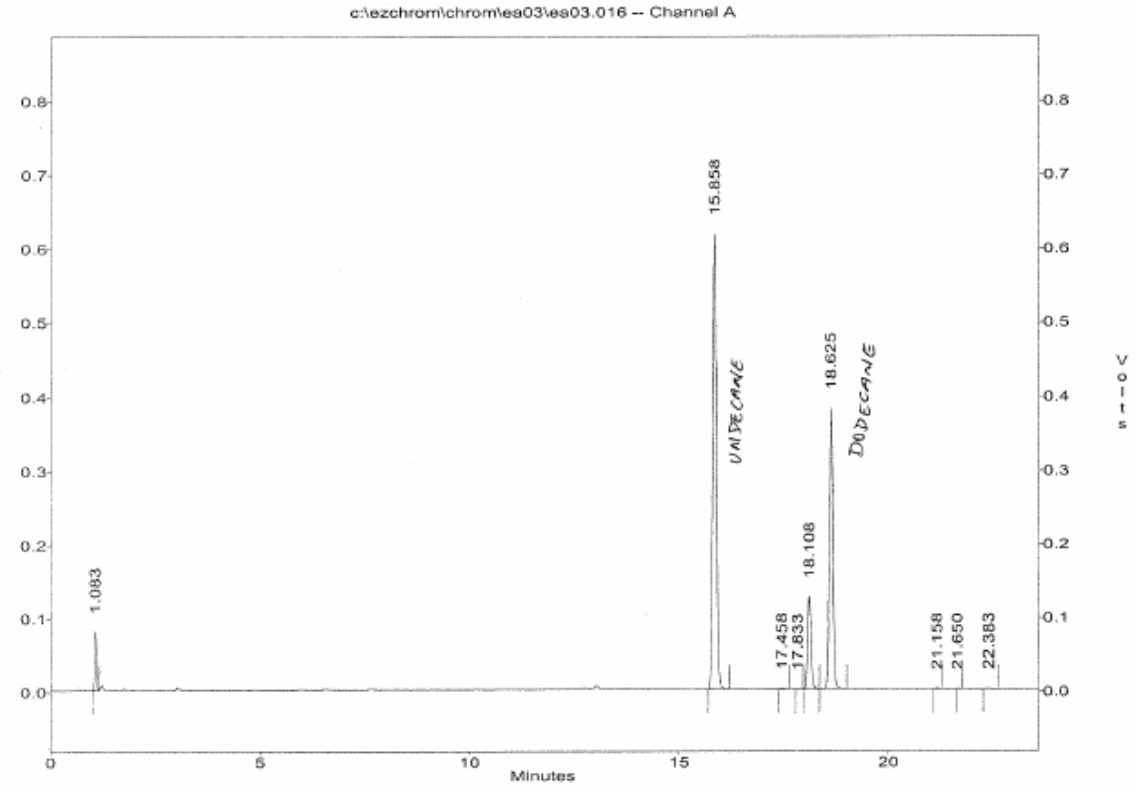


Figure 4: TYPICAL ICAL SUMMARY

INITIAL CALIBRATION
5030B/M8015

Lab Name : EMAX Inc
Instrument ID : GCT39
GC Column : DB-5
Column size ID : 30MX.53MM
LFID & Datetime: EA03003A 01/03/14 17:08
LFID & Datetime: EA03004A 01/03/14 17:46
LFID & Datetime: EA03005A 01/03/14 18:25
LFID & Datetime: EA03006A 01/03/14 19:03
LFID & Datetime: EA03007A 01/03/14 19:41
LFID & Datetime: EA03008A 01/03/14 20:20
CONC UNIT: ppb

	CONC	CALIBRATION FACTORS (AREA)/UNIT						
COMPOUND	X	1.00X	2.50X	5.00X	25.00X	50.00X	75.00X	MEAN	%RSD
Gasoline(TOTAL)	20.00	24652	27578	27219	29767	26888	27295	27233.2	6.0
GRO(C6-C10)	20.00	18461	21683	20770	21960	20286	20480	20606.5	6.0
GRO(2MP-124TMB)	20.00	18461	21683	20770	21865	20227	20422	20571.2	6.0
GRO(C5-C12)	20.00	23119	27187	26894	29198	26465	26832	26615.6	7.4
GRO(C6-C12)	20.00	23022	27147	26869	29193	26457	26826	26585.6	7.5
GRO(C5-C10)	20.00	18558	21723	20794	21964	20294	20486	20636.5	5.9
SURROGATE	X	1.00X	2.00X	3.00X	4.00X	5.00X	6.00X	MEAN	%RSD
Bromofluorobenzene	10.00	15758	16495	16129	20469	18793	19444	17848.0	11.1
1,1,1-Trifluorotoluene	10.00	22945	22299	21809	22278	23050	23951	22722.0	3.3

VG39A03.MET

INITIAL CALIBRATION
5030B/M8015

Lab Name : EMAX Inc
Instrument ID : GCT39
GC Column : DB-5
Column size ID : 30MX.53MM
LFID & Datetime: EA03003A 01/03/14 17:08
LFID & Datetime: EA03004A 01/03/14 17:46
LFID & Datetime: EA03005A 01/03/14 18:25
LFID & Datetime: EA03006A 01/03/14 19:03
LFID & Datetime: EA03007A 01/03/14 19:41
LFID & Datetime: EA03008A 01/03/14 20:20

	RT OF STANDARDS (MIN)						MEAN	RT WINDOW		RTWINDOW
COMPOUND	1.0X	2.5X	5.0X	25.0X	50.0X	75.0X	RT	FROM	TO	WIDTH
Gasoline(TOTAL)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
GRO(C6-C10)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
GRO(2MP-124TMB)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
GRO(C5-C12)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
GRO(C6-C12)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
GRO(C5-C10)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
SURROGATE	1.0X	2.0X	3.0X	4.0X	5.0X	6.0X	RT	FROM	TO	WIDTH
Bromofluorobenzene	10.700	10.683	10.683	10.675	10.675	10.675	10.682	10.639	10.725	0.043
1,1,1-Trifluorotoluene	3.192	3.158	3.167	3.117	3.125	3.125	3.147	3.023	3.271	0.124

VG39A03.MET

Figure 5: TYPICAL CONTINUING CALIBRATION SUMMARY

CONTINUE CALIBRATION
5030B/M8015

Lab Name : EMAX Inc
Instrument ID : GCT39
GC Column : DB-5
Column size ID : 30MX.53MM
Mid Conc Init LFID & Datetime: EA03006A 01/03/2014 19:03
Conc Cont LFID & Datetime: EA09003A 01/09/2014 13:25
CONC UNIT : ppb

COMPOUND	RT MINUTES	RT WINDOW FROM	RT WINDOW TO	TRUE CONC	AVERAGE CF	RESULT AREA	RESULT CONC	%D	QL	%D LIMITS
Gasoline(TOTAL)	NA	NA	NA	500.0	27233.2	13446054	493.74	-1		20
GRO(C6-C10)	NA	NA	NA	500.0	20606.5	10381184	503.78	1		20
GRO(2MP-124TMB)	NA	NA	NA	500.0	20571.2	10231844	497.39	-1		20
GRO(C5-C12)	NA	NA	NA	500.0	26615.6	13343700	501.35	0		20
GRO(C6-C12)	NA	NA	NA	500.0	26585.6	13342070	501.85	0		20
GRO(C5-C10)	NA	NA	NA	500.0	20636.5	10382814	503.13	1		20
SURROGATE	**MINUTES**	**FROM**	**TO**	**TRUECON**	**CF**	**AREA**	**CONC**	**%D**	**QL**	**LIMITS**
Bromofluorobenzene	10.700	10.657	10.743	40.0	17848.0	731282	40.97	2		20
1,1,1-Trifluorotoluene	3.192	3.068	3.316	40.0	22722.0	921311	40.55	1		20

Figure 6: TYPICAL RAW DATA

EPA METHOD 8015 by FID
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea09\ea09.009
Method : c:\ezchrom\methods\vg39a03.met
Sample ID : 14A018-03 5.0ML W
Acquired : Jan 09, 2014 17:21:15
Printed : Jan 24, 2014 11:58:54
User : SERGIO

Channel A Results

#	Peak Name	Ret.Time(Min)	Area	Ave. CF	ESTD Conc.(PPB)
5	1,1,1-TFT	3.192	908699.0	22722.0	39.99
7	Bromofluorobenzene	10.700	638363.0	17848.0	35.77
G1	GASOLINE(TOTAL)		221859.0	27233.2	8.15
G2	GRO(C6-C10)		137610.0	20606.5	6.68
G3	GRO(2MP-124TMB)		137610.0	20571.2	6.69
G4	GRO(C5-C12)		212863.0	26615.6	8.00
G5	GRO(C6-C12)		209157.0	26585.6	7.87
G6	GRO(C5-C10)		141316.0	20636.5	6.85

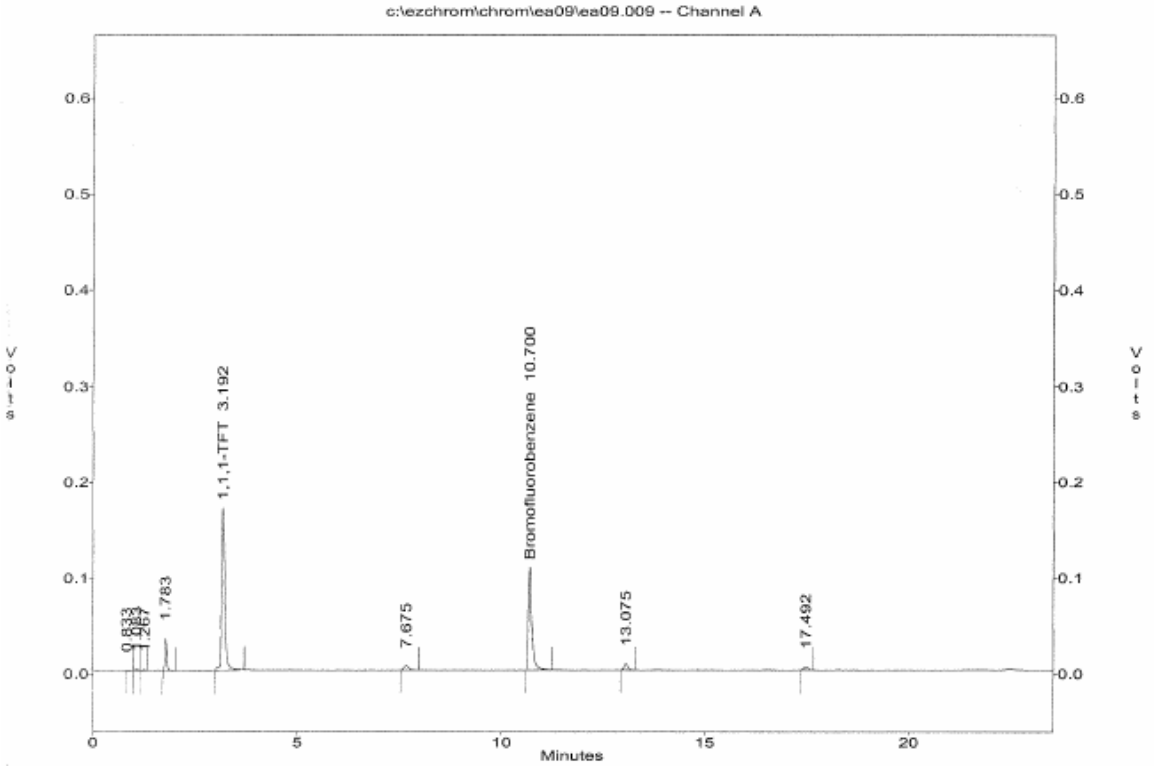


Figure 7: TYPICAL SAMPLE RESULT SUMMARY

METHOD 5030B/8015C
TOTAL PETROLEUM HYDROCARBONS BY PURGE AND TRAP

Client : XYZ, INC.	Date Collected: 01/07/14
Project : CLEAN WATER	Date Received: 01/08/14
Batch No. : 14A018	Date Extracted: 01/09/14 17:21
Sample ID: A5-MW15-010714	Date Analyzed: 01/09/14 17:21
Lab Samp ID: A018-03	Dilution Factor: 1
Lab File ID: EA09009A	Matrix : WATER
Ext Btch ID: VG39A06	% Moisture : NA
Calib. Ref.: EA09003A	Instrument ID : GCT039

PARAMETERS	RESULTS (mg/L)	LOQ (mg/L)	DL (mg/L)	LOD (mg/L)
GASOLINE	0.0067J	0.020	0.0050	0.010

SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
BROMOFLUOROBENZENE	0.0358	0.04000	89.4	60-140

Parameter	H-C Range
Gasoline	C6-C10

Figure 8: **TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER
BATCH NO.: 14A018
METHOD: METHOD 5030B/8015C

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	MBLK1W				
LAB SAMP ID:	VG39A06B	VG39A06L	VG39A06C		
LAB FILE ID:	EA09006A	EA09004A	EA09005A		
DATE EXTRACTED:	01/09/1415:23	01/09/1414:03	01/09/1414:42	DATE COLLECTED:	NA
DATE ANALYZED:	01/09/1415:23	01/09/1414:03	01/09/1414:42	DATE RECEIVED:	01/09/14
PREP. BATCH:	VG39A06	VG39A06	VG39A06		
CALIB. REF:	EA09003A	EA09003A	EA09003A		

ACCESSION:

PARAMETER	BLNK RSLT (mg/L)	SPIKE AMT (mg/L)	BS RSLT (mg/L)	BS % REC	SPIKE AMT (mg/L)	BSD RSLT (mg/L)	BSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Gasoline	ND	0.500	0.477	95	0.500	0.459	92	4	60-130	30

SURROGATE PARAMETER	SPIKE AMT (mg/L)	BS RSLT (mg/L)	BS % REC	SPIKE AMT (mg/L)	BSD RSLT (mg/L)	BSD % REC	QC LIMIT (%)
Bromofluorobenzene	0.0400	0.0395	99	0.0400	0.0390	97	70-130

Figure 9: **TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER
BATCH NO.: 14A018
METHOD: METHOD 5030B/8015C

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	A5-MW15-010714				
LAB SAMP ID:	A018-03	A018-03M	A018-03S		
LAB FILE ID:	EA09009A	EA09010A	EA09011A		
DATE EXTRACTED:	01/09/1417:21	01/09/1417:59	01/09/1418:38	DATE COLLECTED:	01/07/14
DATE ANALYZED:	01/09/1417:21	01/09/1417:59	01/09/1418:38	DATE RECEIVED:	01/08/14
PREP. BATCH:	VG39A06	VG39A06	VG39A06		
CALIB. REF:	EA09003A	EA09003A	EA09003A		

ACCESSION:

PARAMETER	SMPL RSLT (mg/L)	SPIKE AMT (mg/L)	MS RSLT (mg/L)	MS % REC	SPIKE AMT (mg/L)	MSD RSLT (mg/L)	MSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Gasoline	0.00668J	0.500	0.445	88	0.500	0.465	92	4	50-130	30

SURROGATE PARAMETER	SPIKE AMT (mg/L)	MS RSLT (mg/L)	MS % REC	SPIKE AMT (mg/L)	MSD RSLT (mg/L)	MSD % REC	QC LIMIT (%)
Bromofluorobenzene	0.0400	0.0390	98	0.0400	0.0392	98	60-140

Figure 10: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN WATER

SDG : 14A018

METHOD 5030B/8015C
TOTAL PETROLEUM HYDROCARBONS BY PURGE AND TRAP

A total of ten (10) water samples were received on 01/08/14 for TPH Gasoline analysis, Method 5030B/8015C in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

Holding Time
Samples were analyzed within the prescribed holding time.

Calibration
Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source (ICV). Continuing calibration (CCV) verifications were carried on a frequency specified by the project. All calibration requirements were within acceptance criteria. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank
Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample
A set of LCS/LCD was analyzed with the samples in this SDG.
Percent recoveries for VG39A06L/C were all within QC limits.

Matrix QC Sample
A set of MS/MSD was analyzed with the samples in this SDG.
Percent recoveries for A018-03M/S were within project QC limits.

Surrogate
Surrogate was added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis
Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC Procedure	Frequency	Acceptance Criteria	Corrective Action	1st Rvw	2nd Rvw
Five-point initial calibration for all analytes	Initially; as needed	ACF RSD: ≤ 20%	Correct the problem then repeat initial calibration		
Initial calibration verification (ICV)	After initial calibration	Within ± 20% of expected value	Correct the problem then repeat initial calibration		
Calibration verification (DCC)	Every 12 hours of analysis time and at the end of analysis sequence	Within ± 20% of expected value	Correct the problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification		
Method blank	One per preparation batch	No analytes detected ≥ ½ LOQ	If sample results are ND or contamination is < 10X of sample result, consult with the PM if results are reportable. Otherwise, determine the source of contamination and correct the problem. Reanalyze method blank and all samples processed with the contaminated blank. If re-analysis is not possible, apply B to specific analyte(s) on all associated.		
LCS	One LCS per preparation batch	Within project QC Limits	Re-prep and reanalyze the LCS and all associated samples		
Surrogate spike	Every sample, spiked sample, standard, and method blank	Within project QC Limits	If no apparent matrix interference is observed, reanalyze the sample. Otherwise inform the PM for further instruction.		
MS/MSD	One MS/MSD per every 20 project samples per matrix	Refer to project QC Limits	Ensure that spike concentration, spike addition was accurate and calculation is correct. If chromatogram exhibits matrix interference narrate observation in the case narrative.		
Chromatogram	All sample results	Within calibration range No saturated peak(s)	Dilute and re-analyze all samples over the calibration range Diluted and re-analyzed all samples demonstrating saturated peak(s) even if the total integrated peaks do not exceed the calibration range.		
Notes: Discrete peaks are included for GRO Discrete peaks are subtracted for Gasoline Refer to PSR for flagging criteria. Report results between LOD and LOQ.				Reviewed By Date	

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
GASOLINE RANGE ORGANICS
METHOD: SW 8015C

SOP: EMAX-8015G
Conc Unit: µg/L
Sample Amount(ml): 5

Instrument ID: 39
Analysis date: 1/6/2014
Analyzed by: Cervantes, Sergio

PARAMETER	EA06004A	EA06005A	EA06006A	EA06007A	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	VG39A01L	VG39A01C	VG39A02L	VG39A02C							
Gasoline(TOTAL)	458	485	447	426	500	454	91	24.5	5	70 - 130	PASSED
GRO(C6-C10)	503	540	501	477	500	505	101	25.8	5	70 - 130	PASSED
GRO(2MP-124TMB)	504	542	503	480	500	507	101	25.4	5	70 - 130	PASSED
GRO(C5-C12)	461	490	453	432	500	459	92	24.2	5	70 - 130	PASSED
Bromofluorobenzene	42.3	42.8	41.7	39.4	40	41.5	104	1.50	4	70 - 130	PASSED
1,1,1-Trifluorotoluene	46.4	46.3	45.6	45.6	40	46.0	115	0.43	1	70 - 130	PASSED

SOP: EMAX-8015G
Conc Unit: µg/Kg
Sample Amount(g): 5

Instrument ID: 39
Analysis date: 1/6/2012
Analyzed by: Cervantes, Sergio

PARAMETER	EA06013A	EA06014A	EA06015A	EA06016A	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	GMA001SL	GMA001SC	GMA001SX	GMA001SY							
Gasoline(TOTAL)	20480	19683	20590	20124	25000	20200	81	409	2	60 - 130	PASSED
GRO(C6-C10)	22739	21724	22671	22202	25000	22300	89	472	2	60 - 130	PASSED
GRO(2MP-124TMB)	22676	21739	22618	22216	25000	22300	89	433	2	60 - 130	PASSED
GRO(C5-C12)	20651	19816	20681	20251	25000	20300	81	406	2	60 - 130	PASSED
Bromofluorobenzene	2228	2109	2176	2166	2000	2170	109	49	2	70 - 130	PASSED
1,1,1-Trifluorotoluene	2337	2246	2335	2338	2000	2310	116	45	2	70 - 130	PASSED

8015GFS: SAMPLE PREPARATION LOG

EMAX LABORATORIES, INC.

EXTRACTION LOG
for
PURGEABLE TPH

Page 1

Note: For samples, relevant QCs/Standards extracted, refer to attached extraction sequence.

Comments:

Book #.: E39-036

Preparation Batch:

Matrix

SOP	Rev. #
☐ EMAX-5035	3
☐ EMAX-AK101	2
☐ EMAX-	

Standards	ID	Amount Added (ml)
Surrogate		
LCS/MS		
Methanol		
Methanol w/ Surrogate		

Reagent	Lot # / ID
Silica Sand	

Prepared By:

Standard Added By: Checked By:

Extract Location:

Disposal By: Disposal Date:

8015GFA: **ANALYTICAL RUN LOG**

EMAX LABORATORIES, INC.

ANALYTICAL RUN LOG
for
PURGEABLE TPH

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Comments:

Purge Volume = 5 ml

Book #: A39-047

Instrument No.: 39

Analytical Sequence:

Method File: ☐ ☐ ☐

Analytical Batch:

SOP #	Rev. #
☐ EMAX-5030B	3
☐ EMAX-BTEXM	1
☐ EMAX-8015G	5
☐ EMAX-AK101	2
☐ EMAX-	

STANDARDS ID	Amt Added (µL)	Conc. (mg/L)
ICAL		
ICAL		
ICV		
ICV		
DCC GAS		
DCC BTEX		
DCC		
BFB/TFT (Sample)		
BFB/TFT (DCC)		
LCS/LCSD		
MS/MSD		
GRO (HC-Chain)		

Solvent	ID/Lot #
Methanol	
Reagent Water	
	Lot #
pH strip	

ELECTRONIC DATA ARCHIVAL	
Location	Date
☐ EZC-3-BTEX	

Analyzed By:

Date:

8015GFM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: 39 Month: Year: 201_ Book # M39-010

DATE							
Computer Date/Time							
Printer Paper							
GC Keyboard Check							
Check Temperature Program							
Column Head Pressure (5-10 psi)							
Carrier Source Pressure (He @ 50psi)							
H_2 Generator: 40-50 psi							
Air Pressure (45 psi)							
Analyst's initial							
Trouble Shooting							
Problem(s) Encountered							
Return-to-Control (Analyst Initial)							

Code	Problems Encountered	Code	Corrrective Action / Maintenance

STANDARD OPERATING PROCEDURES

ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY

SOP No.:	EMAX-8081	Revision No.	8	Effective Date:	16-Jun-14
Prepared By:	Tu Nisamaneepong			Date:	06-16-14
Approved By:	Kenette Pimentel QA Manager			Date:	06.16.14
Approved By:	Caspar Pang Laboratory Director			Date:	06-16-14

Control Number: 8081-08-

1.0 SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentration of various Organochlorine Pesticides in soil, sediment, sludge, and wastewater samples by gas chromatography method. This SOP is an adaptation of EPA 8081B. Since EPA 8081B is an update and enhancement of EPA 8081A, this SOP is also applicable to EPA 8081A.

2.0 SUMMARY OF METHOD

2.1. This method provides gas chromatographic conditions for the identification and quantitation of Organochlorine Pesticides with dual Electron Capture Detectors (ECD). The samples are extracted in methylene chloride and exchanged to hexane before GC analysis.

2.2. **Interferences**

2.2.1. Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. Avoiding contact with any plastic materials and checking all solvents for phthalate contamination can minimize interferences. Glassware must be scrupulously cleaned.

2.2.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination is most likely present in sediment samples. The TBA procedure, GPC or other cleanup technique can be used for sulfur removal.

2.2.3. Other interferences such as aliphatic compounds, aromatics and nitrogen-containing compounds may be eliminated by using Florisil cleanup.

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.

3.1.2. Table 10 lists the DL, LOD and LOQ values of the target analytes of this method.

4.0 DYNAMIC RANGE

4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point. All samples analyzed above this concentration are considered "over-range" and shall require dilution for proper quantitation.

4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required for proper quantitation.

4.3. The linear dynamic range for this method as determined in this SOP is listed on the table below.

Analytes	Water (μg/L)	Soil (μg/Kg)
alpha-BHC, Endosulfan I, gamma-BHC, Heptachlor, Aldrin, alpha-Chlordane, beta-BHC, delta-BHC, gamma-Chlordane, Heptachlor Epoxide	0.1 – 0.8	2 – 26
4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Dieldrin, Endrin, Endosulfan II, Endosulfan Sulfate, Endrin Aldehyde, Endrin Ketone	0.1 – 1.6	2 – 52
Methoxychlor	1 – 8	10– 260
Toxaphene	2 – 20	50 – 660
Technical Chlordane	1 – 15	50 – 500
2,4'-DDD, 2.4'-DDE, 2,4'-DDT, Oxychlordane, cis-Nonachlor, trans-Nonachlor, Mirex	0.1 – 0.8	2 – 26

5.0 SAMPLE HOLDING TIME AND PRESERVATION

5.1. **Holding Time**

5.1.1. Extract water and soil samples within 7 and 14 days from date of collection, respectively.

5.1.2. Analyze extracts within 40 days after extraction completion date.

5.2. **Preservation**

5.2.1. Store samples and extract at ≤6^{o}C.

6.0 ASSOCIATED SOPs

6.1. EMAX-DM01 Data Flow and Review

6.2. EMAX-QA04 Detection Limit Study

6.3. EMAX-QA08 Corrective Action

6.4. EMAX-QC01 Quality Control for Chemicals

6.5. EMAX-QC02 Analytical Standard Preparation

6.6. EMAX-QC07 Glassware Cleaning

6.7. EMAX-SM03 Waste Disposal

6.8. EMAX-SM04 Analytical and QC Sample Labeling

6.9. EMAX-3520 Extraction, Continuous Liquid/Liquid

6.10. EMAX-3550 Extraction, Pulse Sonication

6.11. EMAX-3540 Extraction, Soxhlet

6.12. EMAX-3620 Cleanup, Florisil

6.13. EMAX-3660 Cleanup, Sulfur

7.0 SAFETY

7.1. Read all SDS of chemicals listed in this SOP.

7.2. ECD contains minute quantity of Radioactive Ni (63). Conduct a wipe test (experienced personnel or manufacturer only) semi-annually or sooner if potential problem is suspected.

7.3. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves at all times when performing this procedure.

7.4. If, for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amounts of tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND REAGENTS

8.1. **Instruments and Supplies**

Gas Chromatograph	PE Clarus 680
Detector	Dual Electron Capture Detectors
Column	RTX CLPEST I (30 m x 0.32 mm x 0.32 μm) RTX CLPEST II (30 m x 0.32 mm x 0.25 μm) (Alternate columns may be used after verification of performance)
Data System	EZ Chrom Elite
Auto Sampler	PE Clarus 680 or equivalent
Gas	ultra-high purity nitrogen Peak Scientific Hydrogen Generator PH-600 PE Hydrogen Generator PGX Series 3
Microsyringes	10, 25, 100and 500 ul with a 0.006 mm ID needle (Hamilton 702N or equivalent) for dilution purposes
Transfer Pipette	Pasteur

8.2. **Chemicals and Reagents**

Solvent [GC-grade]	Methylene Chloride, Hexane, Acetone

9.0 STANDARDS

9.1. **Standard Preparation**

9.1.1. Follow procedures for all standard preparations and labeling as described in EMAX-QC02 and EMAX-SM04, respectively.

9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

9.2. **Stock Standard**

9.2.1. Purchase Primary Calibration stock standards as certified solutions in one mixture. After opening, transfer the stock standard to an inert amber vial and store with a minimum of headspace.

9.2.2. Purchase a Secondary set of stock standards from a different source to verify the concentration of the first set of standard. Treat the secondary standard similarly as the primary standard.

9.2.3. Purchase LCS/MS, surrogate and performance evaluation standards as certified solutions from various suppliers.

9.2.4. All standards shall be stored at ≤ 6°C.

9.3. **Intermediate Standard**

9.3.1. Prepare intermediate standards as suggested in Table 3 (primary source) and Table 4 (secondary source).

9.3.2. Store all prepared standards in an inert vial with minimum headspace at ≤ 6°C.

9.4. **Initial Calibration Standard (ICAL)**

9.4.1. Prepare five or more calibration standards as suggested in Table 2 from primary intermediate standard (refer to Table 3).

9.5. **Initial Calibration Verification (ICV)**

9.5.1. Prepare ICV at concentration levels suggested in Table 5 using intermediate standard from second source stock standard (refer to Table 4).

9.6. **Daily Calibration Check Standard (DCC)**

9.6.1. Prepare DCC from the same source as the ICAL standard as suggested in Table 5.

9.7. **Surrogate Standard**

9.7.1. Prepare surrogate standard as suggested in Table 6.

9.8. **LCS/MS Spike Standard**

9.8.1. Prepare LCS solution as suggested in Table 4.

9.8.2. Prepare MS spike standard as suggested in Table 7.

9.9. **Performance Evaluation Mixture (PEM)**

9.9.1. Prepare PEM as suggested in Table 8.

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Prepare aqueous samples as described in EMAX-3520 or EMAX-3510.

10.1.2. Prepare solid samples as described in EMAX-3550, EMAX-3540 or EMAX-3545.

10.1.3. Perform extract clean up (if necessary) as described in EMAX-3620, EMAX-3640 or EMAX-3660, whichever is appropriate.

10.2. **Instrument Parameters**

10.2.1. Method 8081A requires an analytical system complete with a temperature programmable gas chromatograph equipped with an autosampler suitable for on column injection of 1 to 5 µl.

10.2.2. Gas Pressure

N_2 gas line : 60 – 90 psi

H_2 gas line : 60 – 90 psi

10.2.3. Detector Temperature : 305°C (Inst. E8 and F9)

10.2.4. Injector Temperature : 220°C (Inst. E8 & F9)

10.2.5. Make-up Gas Flow : N_2 at 40 mL/min

10.2.6. Carrier Gas Flow

For Temperature Program (Inst. F9) : 3.8 mL/min

For Temperature Program (Inst. E8) : See flow program below

Carrier Gas Ramp	Rate (mL/min)	Setpoint (mL/min)	Hold (min)
Initial	0	2.7	5.5
1	2.0	4.0	20.0

10.2.7. Oven Temperature Program (Instrument E8)

Oven Ramp	Rate (°C/min)	Temp (°C)	Hold (min)
Initial	0	140	0.50
1	25	230	2.00
2	5	260	0.20
3	25	290	2.50

10.2.8. Oven Temperature Program (Instrument F9)

Oven Ramp	Rate (°C/min)	Temp (°C)	Hold (min)
Initial	0	130	0.50
1	25	230	2.00
2	5	260	0.20
3	25	290	3.00

10.3. **Calibration**

10.3.1. **Performance Evaluation Check**

10.3.1.1. Analyze instrument blank and a PEM containing DDT and Endrin to monitor the system performance at 12-hour interval prior to performing any calibration.

10.3.1.2. Calculate the breakdown by using Equations 10.6.6 ($\%B_T$) for DDT and 10.6.7 ($\%B_E$) for Endrin.

10.3.1.3. Check Appendix 1 for acceptance criteria before proceeding with sample analysis.

10.3.1.4. If system failed to meet the acceptance criteria, refer to Section 12 for corrective action.

10.3.2. **Initial Calibration (ICAL)**

10.3.2.1. Perform ICAL if instrument is new, ICV or DCC failed to meet acceptance criteria or after a major instrument repair.

10.3.2.2. A minimum of five calibration standards, or as suggested in Table 1, over the concentration range of interest are sequentially injected into the GC. Refer to Table 1 for ICAL concentrations. Peak areas are obtained from each analyte.

10.3.2.3. Application of ICAL Curve for Quantitation

10.3.2.3.1. Generate a summary of calibration factors for each analyte at each concentration using Eq-10.6.1. Calculate the Average Calibration Factor (ACF), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to, Eq. 10.6.2, Eq. 10.6.3 and Eq. 10.6.4, respectively.

- If RSD is ≤ 20% ACF may be applied.
- Apply Inverse Weighting Factor (1/y or 1/y2; y being the instrument response) if it is determined to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of ≤ 20% and correlation coefficient of ≥ 0.995.
- Apply linear least squares regression if past experience or priori knowledge of instrument response is known to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of ≤ 20% and correlation coefficient of ≥ 0.995.
- It may be appropriate to force the regression through zero for specific analytes[1]. When exercising this option [as included in the data acquisition software], make sure that the origin (0,0) is not included as a calibration point but rather the intercept is set to zero. This option shall only be applied if the curve favors better accuracy of quantitation.

10.3.2.4. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

10.3.2.5. Refer to Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.

10.3.3. **Initial Calibration Verification (ICV)**

10.3.3.1. Analyze ICV prepared from another source as described in Section 9.5 to verify the concentrations of the ICAL.

[1] SW846 Method 8000B, Section 7.5.3; SW846 Method 8000C, Section 11.5.2.1

10.3.3.2. Calculate the CF and the percent difference (%D) according to Eq-10.6.1 and 10.6.5 respectively. Refer to Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.

10.3.4. **Multicomponent Target Analyses**

10.3.4.1. For multicomponent target analytes (Toxaphene), a five-point calibration standard shall be included in initial calibration for pattern recognition and quantitation.

10.3.4.2. Integrate the total response of the chromatogram to obtain the total area. Calculate the calibration factor (CF) by using Equation 10.6.1.

10.3.5. **Daily Calibration Check (DCC)**

10.3.5.1. Analyze DCC at the start of the 12-hour shift prior to sample analysis and close the analytical run with an ending DCC.

10.3.5.2. Calculate the %D by using Equation 10.6.5. Check Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.

10.4. **Analysis**

10.4.1. **Analytical Sequence**

10.4.1.1. Following the instrument data acquisition software, prepare the analytical sequence file as suggested below:

- Pesticide Prime – 20/200 ppb pesticide injected at the beginning of analytical sequence if the GC has not been used for a day or more
- IB – instrument blank
- PEM – performance evaluation mixture
- ICAL – initial calibration standards
- ICV or DCC1 – initial calibration verification or continuing calibration standard
- MB – method blank
- LCS – lab control sample
- Samples – up to 12 hours
- DCC2 – continuing calibration standard or ending DCC

10.4.2. **Sample Analysis**

10.4.2.1. Transfer a minimum of 0.5 ml of extract to a 2-ml auto sampler vial (or equivalent) using a Pasteur pipette. Seal the vial with a polypropylene screw cap with PTFE/red silicone rubber septa. Similarly, prepare the analytical standards and QC samples.

10.4.2.2. Introduce sample extract into the GC using direct injection technique (1 to 5 μl) after all system quality control criteria have been met.

10.4.2.3. If the response exceeds the linear range of the system, dilute the sample and re-analyze.

10.4.3. **Sample Result Evaluation**

10.4.3.1. Check QC parameters as soon as the data is available.

- ✓ Check LCS recoveries against Appendix 1.
- ✓ Check MB that it is project compliant.
- ✓ Check retention time.
- ✓ Check surrogate recoveries against Appendix 1.
- ✓ Check concentration of target analytes. If the response exceeds the calibration range, dilute and re-analyze the sample until the response falls within the calibration range.
- ✓ If any of the above checkpoints indicate a problem, re-analysis is required. If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate extraction problem, fill-up an NCR and order re-extraction for the affected sample(s).

10.4.3.2. Positive identification is made when a peak falls within the retention time window of a target analyte on both columns established by the standard reference compound.

10.4.3.3. If one column meets the retention time criteria and a retention time shift is suspected on the other column, use the following guideline in reporting the data:

- ✓ Check that the expanded window does not exceed the RTW of the column in control or the established RTW or the CLP RTW (refer to table 7) whichever is greater.
- ✓ If the above condition is met, report the data and include a description of the observation in the case narrative.

10.4.4. **Retention Time Windows**

10.4.4.1. Establishing RTW

10.4.4.1.1. Collect at least three Daily Calibration Standards analyzed over a period of 72 hours.

10.4.4.1.2. Calculate the Standard Deviation (SD) of absolute retention time obtained for each analyte.

10.4.4.1.3. The width of RTW is defined by $\pm$3X SD obtained from 10.4.4.1.2.

10.4.4.2. Evaluating RTW

10.4.4.2.1. If the SD is equal to 0.00, default to the previous study until historical data is obtained or use the CLP[1] retention time window (refer to Table 9) which ever is narrower.

10.4.4.2.2. For new instruments, in the interim use the CLP retention time window (refer to Table 9) until RTW is obtained for the new instrument parameters condition.

[1] CLP-OLM4.2 Table 1 D-79/PEST

10.4.4.3. Application of RTW

10.4.4.3.1. Establish the center of absolute retention time for each analyte to include the surrogate(s) from the daily calibration check at the beginning of the analytical shift then apply the established RTW.

10.4.4.3.2. Whenever the observed retention time is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing the analyses.

10.4.4.4. Updating RTW

10.4.4.4.1. Re-establish the RTW as described in Section 10.4.4.1 when any of the following conditions occur:

- Yearly RTW update
- Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of RTW)
- Major instrument maintenance (e.g. replacement of detector or column; temperature program change, etc.)

10.4.5. **Manual Integration**

10.4.5.1. Refer to EMAX-DM01 for details of manual integration.

10.4.6. **Dealing with Carryover**

- ✓ Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.
- ✓ If there was no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.
- ✓ If there was a target analyte detected as found in the sample following the sample that exceeded the calibration range (for pesticides concentration ≥ 500/1000/5000 ppb; for Toxaphene concentration ≥ 25 ppm; for chlordane concentration ≥ 10 ppm and for PCBs concentration ≥ 100 ppm) re-analyze the sample to rule-out carry over. If carry over is confirmed, proceed with data reduction and report the data from re-analysis.

10.5. **Data Reduction**

10.5.1. Check the chromatogram of positively identified peaks.

- ✓ Peaks fall within the established retention time window on both columns.
- ✓ Peaks are sharp and not saturated.
- ✓ Peaks are properly integrated (refer to Figure 1 for Peak Evaluation Techniques).
- ✓ Target analyte peak is present in both columns to confirm positive identification.

10.5.2. Positive identification is confirmed when the identified analyte is present in both columns. The agreement between the quantitative results should be evaluated after the identification is made. Calculate the relative percent difference (RPD) between the two results according to Equation 10.6.10.2.

10.5.2.1. If the RPD is less than 40% and the peaks do not indicate any anomalies, report the higher result.

10.5.2.2. If the RPD is less than 40% and one of the peaks indicate an anomaly, report the result from the better peak.

10.5.2.3. If the RPD is greater than 40%, use professional judgment to select the most appropriate result. If no evidence of any chromatographic interference, report the higher result.

10.6. **Calculations**

10.6.1. Calculate for Calibration Factor (CF)

$$CF = \frac{R_a}{C_k} \qquad \text{Eq.-10.6.1}$$

where:

CF - is the calibration factor

R_a - is the analyte response measured in peak area

C_k - is the known concentration of the analyte in μg/L

10.6.2. *Calculate for Standard Deviation*

$$SD = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \overline{x})^2}{N-1}} \qquad \text{Eq.-10.6.2}$$

where:

SD - is the standard deviation

x_i - is the result at the ith measurement

$\overline{x}$ - is the mean

N - is the number of measurements

10.6.3. Calculate for Percent Relative Standard Deviation (%RSD)

$$\%RSD = \left[\frac{SD}{ACF}\right] 100 \qquad \text{Eq.-10.6.3}$$

where:

%RSD - is the percent relative standard deviation

SD - is the standard deviation

ACF - is the average calibration factor

10.6.4. Calculate for Average Calibration Factor (ACF)

$$ACF = \frac{\sum CF}{N} \qquad \text{Eq.-10.6.4}$$

where:

ACF - is the average calibration factor

$\sum CF$ - is the summation of the calibration factors

N - is the number of calibration points

10.6.5. **Calculate for Least Square Linear Regression**

$$y = ax + b$$ Eq.-10.5.1.3

where:

y = Response factor

x = Concentration

a = x1 = slope of the line

$$a = \frac{\sum \left(x - \bar{x}\right)\left(y - \bar{y}\right)}{\left(x - \bar{x}\right)^2}$$

where:

$\bar{x}$ = Average of amount ratios

$\bar{y}$ = Average of response ratios

b=x0 = intercept of the line

$$b = \bar{y} - a * \bar{x}$$

10.6.6. **Calculate for Inverse Weighting Factor**

$$y = ax + b$$ Eq.-10.5.1.4

where:

y = Response Factor

x = Concentration

a = x1 = slope of the line

$$a = \frac{\sum \left[\left(x - x_a\right)\left(y - y_a\right)\right]}{\sum \left(x - x_a\right)^2}$$

where:

$$x_a = \sum \left[x\left(1/x\right)/\sum\left(1/x\right)\right]$$

$$y_a = \sum \left[y\left(1/x\right)/\sum\left(1/x\right)\right] \text{ or}$$

$$x_a = \sum \left[x\left(1/x^2\right)/\sum\left(1/x^2\right)\right]$$

$$y_a = \sum\left[y\left(1/x^2\right)/\sum\left(1/x^2\right)\right]$$

b = x0 = intercept of the line

$$b = y_a - a * x_a$$

10.6.7. Calculate for Percent Difference for DCC from ACF

$$\%D = \left[\frac{CF - ACF}{ACF}\right] 100 \qquad \text{Eq.-10.6.5}$$

where:

%D - is the % Difference

ACF - is the Average Calibration Factor

CF - is the Calibration Factor of the DCC

10.6.8. Calculate for % Breakdown for DDT (%B_T).

$$\%B_T = \frac{A_D + A_E}{A_T + A_D + A_E} \qquad \text{Eq.-10.6.6}$$

where:

$\%B_T$ - % DDT Breakdown

A_D - Total area of DDD

A_E - Total area of DDE

A_T - Total area of DDT

10.6.9. Calculate for % Breakdown for Endrin (%B_E)

$$\%B_E = \frac{A_A + A_K}{A_E + A_A + A_K} \qquad \text{Eq.-10.6.7}$$

where:

$\%B_E$ - % Endrin Breakdown

A_A - Total area of Endrin Aldehyde

A_K - Total area of Endrin Ketone

A_E - Total area of Endrin

10.6.10. Sample Results

10.6.10.1. **Water Samples**

$$C = \left(\frac{R_a}{ACF}\right)\left(\frac{V_e}{S_a}\right)DF \qquad \text{Eq.-10.6.8.1}$$

where:

C - Concentration of sample measured in μg/L

Ra - Total response of analyte in peak area

ACF - Average response factor measure in ICAL

V_e - Volume of extract in ml

S_a - Sample amount in ml

DF - Dilution factor of sample extract

10.6.10.2. **Soil Samples**

$$C = \left(\frac{R_a}{ACF}\right)\left(\frac{V_e}{S_a\left(\%Solid\right)}\right)DF \qquad \text{Eq.-10.6.8.2}$$

where

C - Concentration of analyte to be measured (μg/kg)

R_a - Total response of analyte in peak area

ACF - Average response factor

V_e - Volume of extract in ml

S_a - Sample Amount in g

% Solid- $\frac{100 - \%\ moisture}{100}$

DF - Dilution factor of the sample extract

10.6.11. Multi-peak Compound in Sample (Toxaphene)

10.6.11.1. Total area is integrated and concentration is determined by equation 10.6.8.1 or 10.6.8.2.

10.6.12. Accuracy and Precision

10.6.12.1. **Percent Recovery**

$$\%R = \frac{C_f - C}{C_s} * 100 \qquad \text{Eq.-10.6.10.1}$$

where:

%R - percent recovery

C_f - concentration found in spiked sample

C - concentration of unspiked sample (For LCS, C=0)

Cs - theoretical concentration of surrogate spike

10.6.12.2. **Relative Percent Difference (RPD)**

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \qquad \text{Eq.-10.6.10.2}$$

where:

%RPD - Relative Percent Difference

C1 - Measured concentration of the first sample aliquot

C2 - Measured concentration of the second sample aliquot

10.7. **Report Generation**

10.7.1. Generate the method.txt file using WDB1C.exe.

10.7.2. Generate Lab Chronicle using LABCHRN1.exe.

10.7.3. Generate sample results using F1NV3C.exe.

10.7.4. Generate the QC Summary file using QCV3CN.exe.

10.7.5. Generate the case narrative using CN1.exe.

10.7.6. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analyses.

- Sample Results
- LCS Summary
- MS/MSD Summary
- DCC Summary
- ICAL Summary
- ICV Summary
- Copy of Analysis Log
- Copy of Preparation Log

10.8. **Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the Project Specific Requirements (PSR).

- ✓ Check that all samples required for analysis are performed.
- ✓ Check that samples are extracted and analyzed within Holding Time.
- ✓ Check that all calibration requirements are fulfilled.
- ✓ Check the chromatogram of all positively identified peak(s).
- ✓ Check surrogate recoveries against required limits.
- ✓ Check that concentration of target analytes are within calibration range.

If any of the above checkpoints indicate a problem, re-analysis is required.

10.8.2. Review the case narrative and edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.3. Submit the analysis package for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Refer to Form 8081FM for daily routine maintenance check points.

10.9.2. Record instrument maintenance performed in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back in control.

10.9.3. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality. The table below is a list of preventive maintenance activities that are essential to consider in performing this SOP.

Maintenance Activity	Description	Frequency
Autosampler Check	Inspect and clean syringe. Check autosampler response.	Daily prior to analysis
Verification	Check instrument parameters to ensure normal operating conditions. Check liner as necessary. Check instrument performance (e.g., daily calibration check, instrument blank, DDT/Endrin breakdown).	Daily prior to analysis
Documentation	Record maintenance in instrument service logs.	Daily prior to analysis
Leak Test	Perform inlet pressure decay test.	Every 6 months or as necessary
System Cleaning	Remove dust from fans and vent covers, inspect and clean inlet and detector where applicable.	Every 6 months or as necessary
Check Flow Path Components	Check and replace the following as necessary: tubing assembly, union, sample probe, and loop.	Once a year or as necessary
Complete Inspection	Perform general inspection of the complete system. Inspect autosampler cabling and configuration setting.	Once a year

11.0 QUALITY CONTROL

11.1. **Preparative Batch**

11.1.1. A preparative batch shall consist of a method blank, LCS, MS/MSD (when required by the project) and a maximum of 20 field samples of similar matrix.

11.1.2. In the absence of MS/MSD, prepare LCS/LCD to check for precision.

11.1.3. Surrogate standard shall be added to all samples, including method blank LCS/LCD and MS/MSD. Check PSR for QC Control Limits.

11.1.4. Perform QC check prior to utilizing the surrogate and LCS/MS spike standards by analyzing the prepared standard at the spiking level. Results should be within ± 20% of the expected value.

11.2. **Analytical Batch QC**

11.2.1. Instrument Performance Evaluation Check must be analyzed daily. Acceptance criteria and corrective action are discussed in Section 10.3.1.4 and Appendix 1.

11.2.2. A continuing calibration shall be performed before any other analysis is done. The continuing calibration procedure and the acceptance criteria are discussed in Section 10.3.5 and Appendix 1.

11.3. **Method QC**

11.3.1. Analyst demonstration of proficiency is a must prior to performing this analysis.

11.3.2. A valid LOD and LOQ must exist prior to sample analysis.

11.3.3. A valid ICAL must exist prior to sample analysis.

11.3.4. Instrument performance must be checked prior to sample analysis. Check Appendix 1 for acceptance criteria.

11.3.5. Prepare and analyze QC samples, to include, method blank, LCS (LCD), and MS/MSD. QC Control Limits shall follow the Project Specific Requirement (PSR) in each analytical folder.

12.0 CORRECTIVE ACTION

12.1. Corrective actions associated with this analytical procedure are described in the Summary of In-House Quality Control Procedures in Appendix 1. Document out-of-control event/s and corrective action in the analytical logbook. If the problem persists, consult the supervisor.

12.2. If PEM failed to meet the DDT and Endrin breakdown acceptance criteria, consider the following suggestions to correct the problem:

12.2.1. Deactivate or replace the injection liner.

12.2.2. Check that the injector nut is leak-free.

12.2.3. If problem persists, inform the Supervisor.

12.3. If Initial calibration is non-compliant, consider the following suggestions to correct the problem:

12.3.1. If %RSD is out of acceptance criteria, review result and identify presence of an outlier.

12.3.2. If one of the standard returns a bias-low or bias-high on all of the analytes, then that point is considered an outlier, prepare a standard at that ICAL point and reanalyze.

12.3.3. If the highest ICAL point appears to be saturated, drop the highest point.

12.3.4. If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.

Note: The lowest calibration point identifies the reporting limit (RL). Therefore, check that the RL is in conformance to the current projects where the ICAL will be used.

12.3.5. If instrumentation problem is suspected, consider the following suggestions to correct the problem:

12.3.5.1. Check the connections and make sure they are air-tight and perform maintenance as needed.

12.3.5.2. Check the gas flow.

12.3.5.3. Prepare a fresh standard and repeat calibration.

12.3.6. If the problem persists, inform the supervisor.

12.4. If the ICV is non-compliant, consider the following suggestions to correct the problem:

12.4.1. Re-analyze ICV (to rule out poor injection).

12.4.2. If ICV is still out of acceptance criteria, prepare a fresh standard and re-analyze to rule out any preparation error

12.4.3. If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration.

12.4.4. If the problem persists, inform the supervisor.

12.5. If the instrument blank is non-compliant, consider the following suggestions to correct problem:

12.5.1. Rule out instrument contamination by performing the instrument daily maintenance, such as changing septum, cleaning liner, cleaning or using new auto sampler syringe.

12.5.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.

12.5.3. Rule out preparation contamination by preparing a new instrument blank.

12.5.4. If the problem persists, inform the supervisor.

12.6. If Continuing Calibration is non-compliant, consider the following suggestions to correct the problem:

12.6.1. Change the liner.

12.6.2. Clean injection port.

12.6.3. Prepare new standard.

12.6.4. Cut or replace column.

12.6.5. Clean the detector.

12.6.6. Rule out leaks by checking all connections.

12.6.7. If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL.

12.7. If Method Blank is non-compliant, consider the following suggestions to correct the problem:

12.7.1. Rule out instrument contamination by checking instrument blank.

12.7.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01.

12.7.3. Rule out glassware contamination used for extraction as described in EMAX-QC07.

12.7.4. Re-extract MB and the associated samples with reagents free of contamination or with newly opened reagents.

12.7.5. If the problem persists, inform the supervisor.

12.8. If LCS is non-compliant, perform the following suggestions to correct the problem:

12.8.1. If result is bias-high or bias-low, check the LCS Standard by analyzing at the spike level.

12.8.2. If LCS check is within 80-120% of expected value, check calibration of the micropipette or syringe used for spiking. Re-extract and re-analyze the LCS and the associated samples.

12.8.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS Standard, re-extract and re-analyze LCS and the associated samples.

12.9. Execute a Non-Conformance Report (NCR) when the following circumstances occur:

12.9.1. If corrective action needs the function of other department; e.g., if the sample needs to be re-extracted, refer to EMAX-QA08 for details of completing an NCR.

12.9.2. If corrective action needs the assistance of the project manager; e.g. If the sample is non-compliant to the technical holding time requirement, insufficient amount of sample, or other non-conforming issues.

12.10. For other problems encountered, inform the supervisor immediately for further instructions.

13.0 POLLUTION PREVENTION

13.1. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.

13.2. All unused expired analytical standards shall be separated and properly identified prior to endorsing them to WDU for proper disposal.

14.0 WASTE MANAGEMENT

14.1. All unused samples, expired analytical standards and other waste generated during the analytical process, endorsed to WDU shall be disposed in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. **Definition of Terms**

15.1.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.

15.1.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

15.1.2.1. **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.

15.1.2.2. **Analytical Batch** – is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include

samples originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.3. Detection Limit (DL) – is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

15.1.4. Limit of Detection (LOD) – is defined as the smallest amount or concentration of substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative result rate (Type II error) is 1%.

15.1.5. Limit of Quantitation (LOQ) – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

15.1.6. Safety Data Sheet (SDS) – is written information concerning a chemical physical properties, toxicity, health hazards, fire hazard and reactivity data including storage, spill and handling precautions.

15.1.7. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.8. Calibration Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.9. Carry-over – are contaminants retained in the instrument/apparatus from a highly contaminated sample that is passed into the succeeding sample(s).

15.1.10. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.1.11. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical process to monitor contamination.

15.1.12. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.

15.1.13. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.

15.1.14. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

15.1.15. Sample Duplicate – is a replicate of a sub-sample taken one sample, prepared and analyzed within the same preparation batch.

15.1.16. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.17. Matrix – A component or form of a sample.

15.1.18. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.

15.1.19. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

15.1.20. Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard; used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.

15.1.21. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.

15.1.22. Reagent Soil – organic-free Ottawa sand or equivalent.

15.2. **Application of QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing organochlorine pesticides analysis by GC. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

15.3. **Department of Defense (DoD) Projects and Department of Energy (DoE) Projects**

15.3.1. Samples from DoD and DoE sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, shall be applied.

16.0 REFERENCES

16.1. "Test Methods for Evaluating Solid Waste, Physical / Chemical Methods", EPA Publication SW-846 Methods 8000B and 8081B, as updated

16.2. EMAX Quality Systems Manual, as updated

17.0 APPENDICES

17.1. **Figures**

17.1.1.	Figure 1	Peak Evaluation Technique
17.1.2.	Figure 2	Typical Chromatogram
17.1.3.	Figure 3	Typical Initial Calibration Summary
17.1.4.	Figure 4	Typical Retention Time Window Summary
17.1.5.	Figure 5	Typical PEM PEST Breakdown Calculation Summary
17.1.6.	Figure 6	Typical Sample Result Summary
17.1.7.	Figure 7	Typical LCS Report Summary
17.1.8.	Figure 8	Typical MS/MSD Report Summary
17.1.9.	Figure 9	Typical Case Narrative

17.2. **Tables**

17.2.1.	Table 1	ICAL Concentration of Individual Analytes
17.2.2.	Table 2	ICAL Standard Preparation
17.2.3.	Table 3	Intermediate Primary Standard Preparation
17.2.4.	Table 4	Intermediate Secondary Standard Preparation

17.2.5. Table 5 Check Standard Preparation
17.2.6. Table 6 Surrogate Standard Preparation
17.2.7. Table 7 Spike Standard Preparation
17.2.8. Table 8 Performance Evaluation Mixture Preparation
17.2.9. Table 9 CLP Retention Time Window for Pesticides
17.2.10. Table 10 Established Limit of Detection (LOD) & Limit of Quantitation (LOQ)

17.3. **Appendices**

17.3.1. Appendix 1 Summary of In-House Quality Control Procedures
17.3.2. Appendix 2 Demonstration of Capability

17.4. **Forms**

17.4.1. 8081FS Sample Preparation Log
17.4.2. 8081FA Analytical Run Log
17.4.3. 8081FM Instrument Maintenance Log

Figure 1: PEAK EVALUATION TECHNIQUE

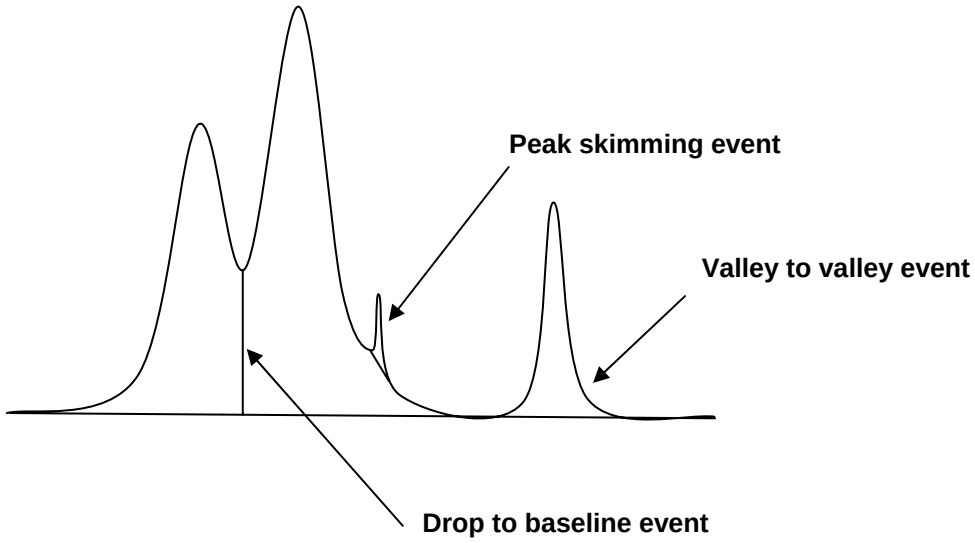
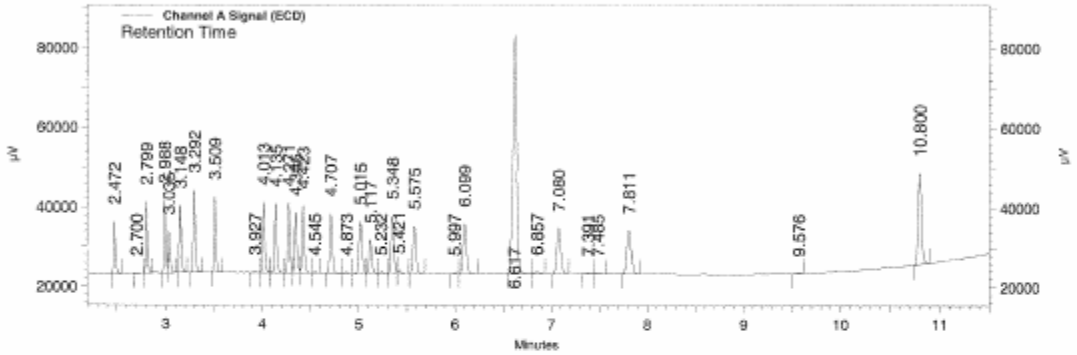


Figure 2: TYPICAL CHROMATOGRAM

Organochlorine Pesticides by GC/ECD
EMAX Laboratories, Inc.

Sample ID: ICPE8A2601 (20/200ppb PEST ICV)
Instrument ID: E8
Method Name: C:\EZChrom Elite\Methods\CPE8A26.met
Data: C:\EZChrom Elite\Data\MA26\MA26034.dat
User: Enrico
Acquired: 01/27/14 06:35:24
Printed: 01/27/14 11:45:07



Channel A Signal (ECD) Results

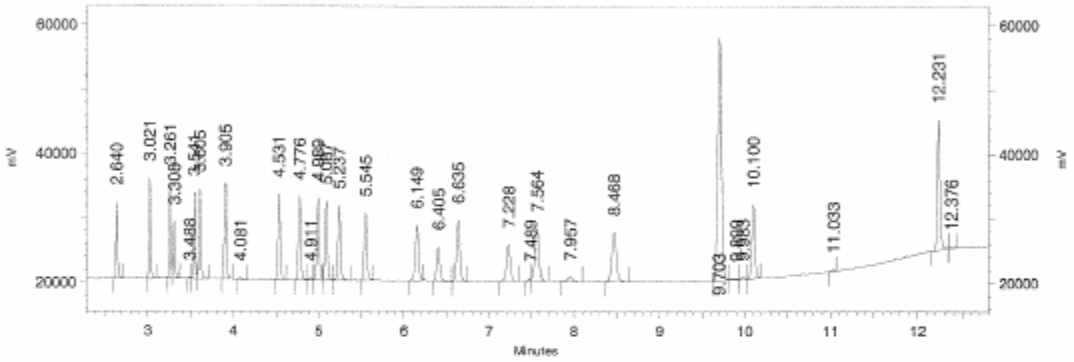
Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration (ppb)	Integration Codes
TCX	2.476	2.472	955775	47593.1	20.082	BB
alpha-BHC	2.804	2.799	1157551	58657.7	19.734	BB
gamma-BHC	2.993	2.988	1194590	60273.4	19.820	BV
beta-BHC	3.040	3.035	755069	37664.5	20.047	VI
delta-BHC	3.153	3.148	1136840	57926.2	19.626	BB
Heptachlor	3.297	3.292	1538033	76393.2	20.133	BB
Aldrin	3.515	3.509	1420481	70593.4	20.122	BB
Heptachlor Epoxide	4.020	4.013	1522180	75468.6	20.170	VV
gamma-Chlordane	4.141	4.135	1539037	76628.8	20.084	VV
alpha-Chlordane	4.277	4.271	1556812	76507.4	20.349	VV
DDE	4.352	4.345	1385456	71405.8	19.403	VV
Endosulfan I	4.429	4.423	1586424	81710.0	19.415	VV
Dieldrin	4.713	4.707	1462708	72989.9	20.040	BV
Endrin	5.021	5.015	1372902	69755.1	19.682	BV
DDD	5.124	5.117	888487	47187.6	18.829	VV
Endosulfan II	5.356	5.348	1607919	77469.6	20.755	BV
DDT	5.581	5.575	1409455	61818.5	22.800	BB
Endrin aldehyde	6.107	6.099	1682493	81300.8	20.695	VB
Methoxychlor	6.625	6.617	8917026	42300.6	210.801	BV
Endosulfan Sulfate	7.087	7.080	1781217	86351.4	20.628	BB
Endrin Ketone	7.820	7.811	1892134	97359.1	19.435	BB
DCB	10.807	10.800	2835411	137403.8	20.636	BB

Figure 2: TYPICAL CHROMATOGRAM

Organochlorine Pesticides by GC/ECD
EMAX Laboratories, Inc.

Page 2 of 2

Sample ID: ICPE8A2601 (20/200ppb PEST ICV)
Instrument ID: E8
Method Name: C:\EZChrom Elite\Methods\CPE8A26.met
Data: C:\EZChrom Elite\Data\MA26\MA26034.dat
User: Enrico
Acquired: 01/27/14 06:35:24
Printed: 01/27/14 11:45:07



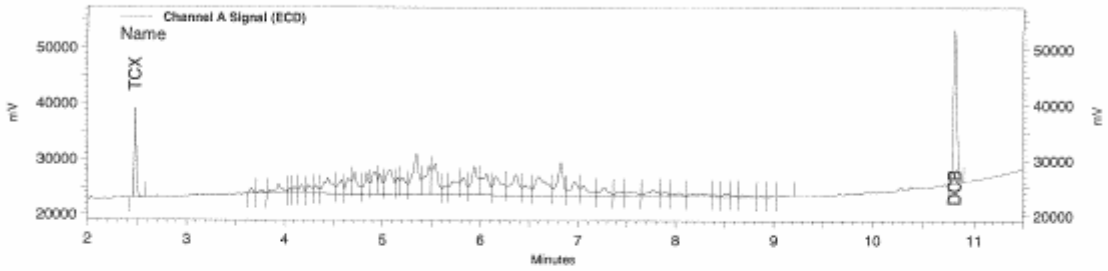
Channel B Signal
(ECD) Results

Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration (ppb)	Integration Codes
TCX	2.644	2.640	883518	43071.9	20.513	BB
alpha-BHC	3.027	3.021	1073354	54398.4	19.731	BB
gamma-BHC	3.267	3.261	1035320	52652.3	19.663	BV
beta-BHC	3.313	3.308	674615	33191.9	20.325	VB
delta-BHC	3.547	3.541	977515	51049.0	19.149	VV
Heptachlor	3.612	3.605	1160948	59362.2	19.557	VB
Aldrin	3.911	3.905	1240610	62034.4	19.999	BB
Heptachlor Epoxide	4.537	4.531	1305228	65794.6	19.838	BB
gamma-Chlordane	4.783	4.776	1339776	67877.6	19.738	BV
alpha-Chlordane	4.995	4.989	1366492	68399.9	19.978	VV
Endosulfan I	5.092	5.087	1332936	67777.1	19.666	VV
DDE	5.245	5.237	1314457	67645.1	19.432	VB
Dieldrin	5.552	5.545	1244362	63414.0	19.623	BB
Endrin	6.156	6.149	1182887	59723.1	19.806	BB
DDD	6.412	6.405	734238	38299.0	19.171	BB
Endosulfan II	6.641	6.635	1371009	66625.0	20.578	BB
DDT	7.235	7.228	959460	44911.8	21.363	BB
Endrin Aldehyde	7.571	7.564	1433036	69406.3	20.647	VB
Endosulfan Sulfate	8.476	8.468	1515182	72700.4	20.841	BB
Methoxychlor	9.708	9.703	5835344	26720.6	218.384	BV
Endrin Ketone	10.107	10.100	1577755	79509.5	19.844	BB
DCB	12.236	12.231	2256977	108283.0	20.843	BV

Figure 2: TYPICAL CHROMATOGRAM

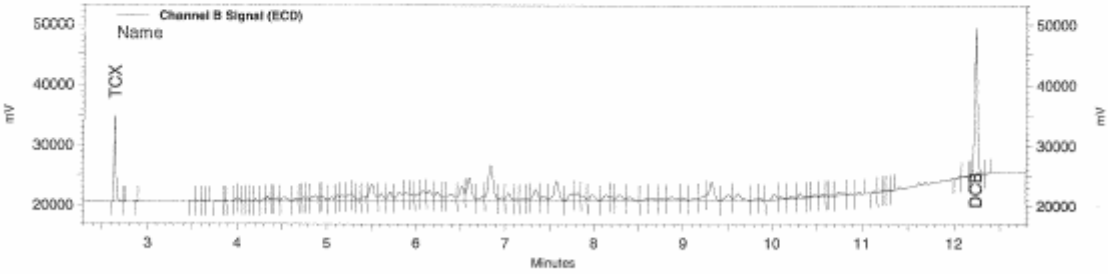
EPA 8081 by GC/ECD
EMAX Analytical Laboratories, Inc.

Sample ID: ITOE8A2601 (500/25ppb TOXA ICV)
Instrument ID: E8
Method Name: C:\EZChrom Elite\Methods\TOE8A26.met
Data: C:\EZChrom Elite\Data\MA26\MA26044.dat
User: Enrico
Acquired: 01/27/14 09:57:27
Printed: 01/27/14 14:58:32



Channel A Signal (ECD) Results

Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration	Integration Codes
TCX	2.480	2.473	1169230	47804.9	24.46	BB
DCB	10.813	10.804	3396622	139579.7	24.33	IB
Toxaphene			26342693	57718.4	456.40	



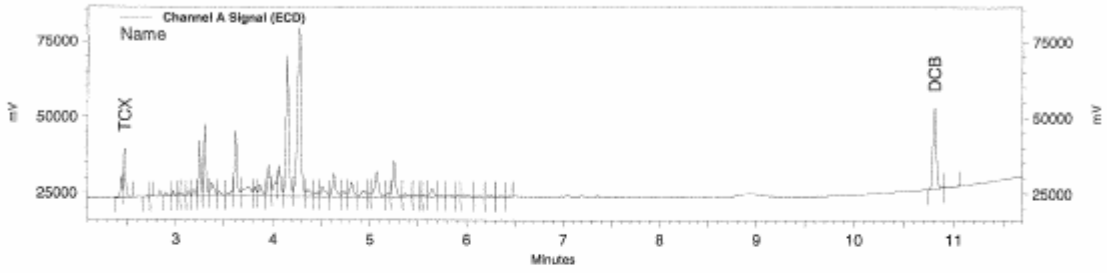
Channel B Signal (ECD) Results

Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration	Integration Codes
TCX	2.648	2.641	1055995	42950.1	24.59	BV
DCB	12.243	12.235	2709359	112370.7	24.11	VV
Toxaphene			16053757	37656.5	426.32	

Figure 2: TYPICAL CHROMATOGRAM

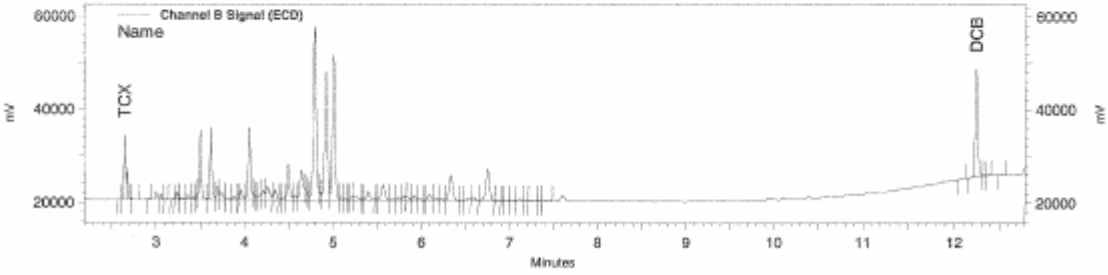
EPA 8081 by GC/ECD
EMAX Analytical Laboratories, Inc.

Sample ID: ICRE8A2601 (500/25ppb CHLO ICAL)
Instrument ID: E8
Method Name: C:\EZChrom Elite\Methods\CRE8A26.met
Data: C:\EZChrom Elite\Data\MA26\MA26017.dat
User: Enrico
Acquired: 01/27/14 00:47:35
Printed: 01/27/14 15:39:10



Channel A Signal (ECD) Results

Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration	Integration Codes
TCX	2.475	2.475	1186519	56032.1	25.24	VB
DCB	10.805	10.808	3266020	136656.8	23.90	BV
Chlordane			31698308	70804.8	447.69	



Channel B Signal (ECD) Results

Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration	Integration Codes
TCX	2.643	2.643	955172	47673.5	24.89	SV
DCB	12.233	12.236	2585430	111115.9	23.27	SS
Chlordane			25015410	55675.2	449.31	

Figure 3: TYPICAL INITIAL CALIBRATION SUMMARY

INITIAL CALIBRATION
METHOD 8081

Lab Name : EMAX Inc
Instrument ID : E8 (PE CLARUS680 GC)
GC Column : STX-CLPESTICIDES
Column size ID : 30MX0.32MMIDX0.32UM
LFID & Datetime: MA26018A 01/27/14 01:07 MA26026A 01/27/14 03:51
LFID & Datetime: MA26019A 01/27/14 01:28 MA26027A 01/27/14 04:11
LFID & Datetime: MA26020A 01/27/14 01:48 MA26028A 01/27/14 04:32
LFID & Datetime: MA26021A 01/27/14 02:08 MA26029A 01/27/14 04:52
LFID & Datetime: MA26022A 01/27/14 02:29 MA26030A 01/27/14 05:13
LFID & Datetime: MA26023A 01/27/14 02:49 MA26031A 01/27/14 05:33
LFID & Datetime: MA26024A 01/27/14 03:10 MA26032A 01/27/14 05:54
LFID & Datetime: MA26025A 01/27/14 03:30 MA26033A 01/27/14 06:14
CONC UNIT: ppb

	CONC	CALIBRATION FACTORS (AREA)/UNIT									
COMPOUND	X	1.00X	2.00X	4.00X	8.00X	16.00X	32.00X	48.00X	64.00X	MEAN	%RSD
alpha-BHC	1.25	50461	49985	49796	51003	58714	65922	69878	73502	58657.7	16.8
Hexachlorobenzene	1.25	151680	148744	145587	138470	133198	123809	116985	114133	134075.9	10.8
gamma-BHC	1.25	55418	53472	53056	53869	60438	65819	68546	71570	60273.4	12.4
beta-BHC	1.25	39043	38406	38222	36583	37752	37503	36734	37073	37664.5	2.3
delta-BHC	1.25	51082	49967	49089	50261	57320	64522	68615	72553	57926.2	16.2
Heptachlor	1.25	75430	75028	75331	73383	76309	78158	78010	79497	76393.2	2.6
Aldrin	1.25	67579	66786	65338	65515	70864	74279	76074	78312	70593.4	7.2
Heptachlor Epoxide	1.25	78332	76078	74193	72576	75070	75350	75340	76810	75468.6	2.3
gamma-Chlordane	1.25	81370	76728	75232	72774	75604	76192	76714	78415	76628.8	3.3
alpha-Chlordane	1.25	76556	75072	74055	72745	76650	78084	78783	80113	76507.4	3.2
DDE	2.50	71592	67348	66695	65767	71126	74599	76133	77986	71405.8	6.4
Endosulfan I	1.25	84110	81668	80093	78406	81687	82227	82191	83299	81710.0	2.2
Dieldrin	2.50	69740	68141	67673	67755	74240	77494	78779	80097	72989.9	7.2
Endrin	2.50	71365	68714	66974	65349	69459	71328	71420	73432	69755.1	3.8
DDD	2.50	41701	41991	41886	42268	47206	51556	54297	56596	47187.6	13.1
Endosulfan II	2.50	78419	77252	75515	74694	78285	78634	78291	78667	77469.6	2.0
DDT	2.50	60030	58843	58134	57951	61977	64277	65890	67446	61818.5	6.0
Endrin Aldehyde	2.50	90040	86147	83229	80437	80903	78039	76352	75260	81300.8	6.2
Methoxychlor	12.50	46278	44926	43915	42720	43071	40728	38661	38106	42300.6	6.9
Endosulfan Sulfate	2.50	91863	88645	86689	85372	86467	84607	83791	83376	86351.4	3.3
Endrin Ketone	2.50	97937	95853	94346	93656	98673	99419	99172	99816	97359.1	2.5
Oxychlordane	1.25	80110	76552	74949	70885	69034	65794	63498	63506	70540.9	8.8
2,4'-DDE	1.25	61390	61447	61948	59722	59374	58300	57066	58216	59682.8	3.0
trans-Nonachlor	1.25	99849	98909	97932	94988	94271	92067	89873	91036	94865.7	4.0
2,4'-DDD	1.25	45894	46222	47460	46120	46089	45603	45404	46864	46207.1	1.4
2,4'-DDT	1.25	63857	63837	63385	61709	61841	60822	59872	61548	62108.8	2.3
cis-Nonachlor	1.25	103410	104959	101085	96481	95137	93261	91561	93833	97465.8	5.2
Mirex	1.25	124772	123651	121188	114831	109357	101324	95348	94069	110567.4	11.3
SURROGATE	X	1.00X	2.00X	4.00X	8.00X	16.00X	32.00X	48.00X	64.00X	MEAN	%RSD
TCX	1.25	50050	49210	47871	46818	47525	46713	46347	46212	47593.1	2.9
DCB	1.25	150585	148308	144355	140284	139179	130342	123677	122500	137403.8	7.8

Figure 3: TYPICAL INITIAL CALIBRATION SUMMARY

INITIAL CALIBRATION
METHOD 8081

Lab Name : EMAX Inc
Instrument ID : E8 (PE CLARUS680 GC)
GC Column : STX-CLPESTICIDES2
Column size ID : 30MX0.32MMIDX0.25UM
LFID & Datetime: MA26018B 01/27/14 01:07 MA26026B 01/27/14 03:51
LFID & Datetime: MA26019B 01/27/14 01:28 MA26027B 01/27/14 04:11
LFID & Datetime: MA26020B 01/27/14 01:48 MA26028B 01/27/14 04:32
LFID & Datetime: MA26021B 01/27/14 02:08 MA26029B 01/27/14 04:52
LFID & Datetime: MA26022B 01/27/14 02:29 MA26030B 01/27/14 05:13
LFID & Datetime: MA26023B 01/27/14 02:49 MA26031B 01/27/14 05:33
LFID & Datetime: MA26024B 01/27/14 03:10 MA26032B 01/27/14 05:54
LFID & Datetime: MA26025B 01/27/14 03:30 MA26033B 01/27/14 06:14
CONC UNIT: ppb

	CONC	CALIBRATION FACTORS (AREA)/UNIT								
COMPOUND	X	1.00X	2.00X	4.00X	8.00X	16.00X	32.00X	48.00X	64.00X	MEAN	%RSD
alpha-BHC	1.25	50646	48650	45661	45897	51758	60345	64329	67899	54398.4	15.8
Hexachlorobenzene	1.25	142667	129534	130501	124881	119255	112893	107022	104412	121395.5	10.7
gamma-BHC	1.25	45669	45113	44964	46196	52559	59047	62269	65403	52652.3	16.1
beta-BHC	1.25	31707	31626	31893	32056	34007	34380	34498	35369	33191.9	4.6
delta-BHC	1.25	46881	43645	42802	42935	48916	56753	61137	65324	51049.0	17.3
Heptachlor	1.25	64469	60382	56873	54863	57269	59256	60104	61682	59362.2	5.1
Aldrin	1.25	57630	56940	55957	56535	61716	66453	68960	72085	62034.4	10.2
Heptachlor Epoxide	1.25	67663	65619	63343	62127	65151	66233	67036	69183	65794.5	3.5
gamma-Chlordane	1.25	76197	66656	64093	63110	65815	67298	68580	71272	67877.6	6.2
alpha-Chlordane	1.25	70136	67140	65002	63977	67396	69438	70904	73206	68399.9	4.5
DDE	2.50	68214	64858	62697	62248	66686	70036	72249	74172	67645.1	6.4
Endosulfan I	1.25	66117	65987	64767	64418	67911	69585	70883	72549	67777.1	4.4
Dieldrin	2.50	58155	57964	57460	58302	63671	68435	70661	72665	63414.0	10.0
Endrin	2.50	60556	59309	57503	56433	59308	60591	60787	63298	59723.1	3.5
DDD	2.50	34514	33973	34920	34687	37768	40811	43220	46498	38299.0	12.3
Endosulfan II	2.50	65007	64584	63685	63806	67073	68650	69168	71027	66625.0	4.2
DDT	2.50	42030	43174	42810	42513	44950	46480	47650	49689	44911.9	6.2
Endrin Aldehyde	2.50	72006	71214	69592	68371	70244	68647	67376	67801	69406.3	2.4
Methoxychlor	12.50	27839	26742	26456	26189	27140	26720	26095	26583	26720.6	2.1
Endosulfan Sulfate	2.50	73417	74342	72545	71104	72952	72408	71798	73038	72700.4	1.4
Endrin Ketone	2.50	79128	76695	75503	76098	80835	82285	82088	83444	79509.5	3.9
Oxychlordane	1.25	64121	62089	64713	61264	56762	55132	54054	54658	59099.1	7.5
2,4'-DDE	1.25	70189	62449	62560	58101	56371	54969	53988	55432	59257.5	9.3
trans-Nonachlor	1.25	99838	95641	90381	84884	83673	82103	81026	82847	87549.1	8.0
2,4'-DDD	1.25	37678	36580	38190	39037	38970	39056	38826	40355	38586.6	2.9
2,4'-DDT	1.25	47592	47648	48035	46663	46259	45738	45245	46948	46766.0	2.1
cis-Nonachlor	1.25	81732	82641	83614	82015	81911	81936	81389	84595	82479.3	1.3
Mirex	1.25	103786	105295	105696	98553	93488	86478	81497	80940	94466.8	11.1
SURROGATE	X	1.00X	2.00X	4.00X	8.00X	16.00X	32.00X	48.00X	64.00X	MEAN	%RSD
TCX	1.25	43237	44796	44334	42283	43035	42766	42017	42107	43071.9	2.4
DCB	1.25	119147	116443	112721	109164	108641	103690	99108	97351	108283.0	7.2

Figure 3: TYPICAL INITIAL CALIBRATION SUMMARY

INITIAL CALIBRATION
METHOD 8081/608

Lab Name : EMAX Inc
Instrument ID : E8 (PE CLARUS680 GC)
GC Column : STX-CLPESTICIDES
Column size ID : 30MX0.32MMIDX0.32UM
LFID & Datetime: MA26036A 01/27/14 07:16
LFID & Datetime: MA26037A 01/27/14 07:36
LFID & Datetime: MA26038A 01/27/14 07:56
LFID & Datetime: MA26039A 01/27/14 08:16
LFID & Datetime: MA26040A 01/27/14 08:36
LFID & Datetime: MA26041A 01/27/14 08:57
LFID & Datetime: MA26042A 01/27/14 09:17
LFID & Datetime: MA26043A 01/27/14 09:37
CONC UNIT: ppb

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT 1.00X	2.00X	5.00X	10.00X	15.00X	20.00X	30.00X	40.00X	MEAN	%RSD
Toxaphene	50.00	58631	53413	56818	57730	58674	58520	58234	59727	57718.4	3.3

INITIAL CALIBRATION
METHOD 8081/608

Lab Name : EMAX Inc
Instrument ID : E8 (PE CLARUS680 GC)
GC Column : STX-CLPESTICIDES2
Column size ID : 30MX0.32MMIDX0.25UM
LFID & Datetime: MA26036B 01/27/14 07:16
LFID & Datetime: MA26037B 01/27/14 07:36
LFID & Datetime: MA26038B 01/27/14 07:56
LFID & Datetime: MA26039B 01/27/14 08:16
LFID & Datetime: MA26040B 01/27/14 08:36
LFID & Datetime: MA26041B 01/27/14 08:57
LFID & Datetime: MA26042B 01/27/14 09:17
LFID & Datetime: MA26043B 01/27/14 09:37
CONC UNIT: ppb

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT 1.00X	2.00X	5.00X	10.00X	15.00X	20.00X	30.00X	40.00X	MEAN	%RSD
Toxaphene	50.00	38754	36281	36249	36766	37480	37838	38209	39675	37656.5	3.2

Figure 3: TYPICAL INITIAL CALIBRATION SUMMARY

INITIAL CALIBRATION
METHOD EPA 8081

Lab Name : EMAX Inc
Instrument ID : E8 (PE CLARUS680 GC)
GC Column : STX-CLPESTICIDES
Column size ID : 30MX0.32MMIDX0.32UM
LFID & Datetime: MA26009A 01/26/14 22:07
LFID & Datetime: MA26010A 01/26/14 22:27
LFID & Datetime: MA26011A 01/26/14 22:47
LFID & Datetime: MA26012A 01/26/14 23:07
LFID & Datetime: MA26013A 01/26/14 23:27
LFID & Datetime: MA26014A 01/26/14 23:47
LFID & Datetime: MA26015A 01/27/14 00:07
LFID & Datetime: MA26016A 01/27/14 00:27
CONC UNIT: ppb

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT 1.00X	2.00X	4.00X	10.00X	20.00X	30.00X	40.00X	60.00X	MEAN	%RSD
CHLORDANE	25.00	89545	68121	80192	70426	67071	64747	63980	62357	70804.9	13.2

Lab Name : EMAX Inc
Instrument ID : E8 (PE CLARUS680 GC)
GC Column : STX-CLPESTICIDES2
Column size ID : 30MX0.32MMIDX0.25UM
LFID & Datetime: MA26009B 01/26/14 22:07
LFID & Datetime: MA26010B 01/26/14 22:27
LFID & Datetime: MA26011B 01/26/14 22:47
LFID & Datetime: MA26012B 01/26/14 23:07
LFID & Datetime: MA26013B 01/26/14 23:27
LFID & Datetime: MA26014B 01/26/14 23:47
LFID & Datetime: MA26015B 01/27/14 00:07
LFID & Datetime: MA26016B 01/27/14 00:27
CONC UNIT: ppb

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT 1.00X	2.00X	4.00X	10.00X	20.00X	30.00X	40.00X	60.00X	MEAN	%RSD
CHLORDANE	25.00	74070	56360	52924	51998	52846	52524	52477	52202	55675.2	13.6

Figure 4: TYPICAL ICAL RETENTION TIME WINDOW SUMMARY

INITIAL CALIBRATION
METHOD 8081

Lab Name : EMAX Inc
Instrument ID : E8 (PE CLARUS680 GC)
GC Columm : STX-CLPESTICIDES
Column size ID : 30MX0.32MMIDX0.32UM
LFID & Datetime: MA26018A 01/27/14 01:07 MA26026A 01/27/14 03:51
LFID & Datetime: MA26019A 01/27/14 01:28 MA26027A 01/27/14 04:11
LFID & Datetime: MA26020A 01/27/14 01:48 MA26028A 01/27/14 04:32
LFID & Datetime: MA26021A 01/27/14 02:08 MA26029A 01/27/14 04:52
LFID & Datetime: MA26022A 01/27/14 02:29 MA26030A 01/27/14 05:13
LFID & Datetime: MA26023A 01/27/14 02:49 MA26031A 01/27/14 05:33
LFID & Datetime: MA26024A 01/27/14 03:10 MA26032A 01/27/14 05:54
LFID & Datetime: MA26025A 01/27/14 03:30 MA26033A 01/27/14 06:14

	RT OF STANDARDS (MIN)								MEAN	RT WINDOW		RTWINDOW
COMPOUND	1.0X	2.0X	4.0X	8.0X	16.0X	32.0X	48.0X	64.0X	RT	FROM	TO	WIDTH
alpha-BHC	2.800	2.801	2.800	2.805	2.804	2.804	2.803	2.801	2.802	2.782	2.822	0.020
Hexachlorobenzene	2.704	2.707	2.708	2.699	2.701	2.705	2.705	2.701	2.704	2.684	2.724	0.020
gamma-BHC	2.988	2.991	2.988	2.995	2.993	2.993	2.992	2.989	2.991	2.969	3.013	0.022
beta-BHC	3.036	3.039	3.036	3.043	3.040	3.041	3.039	3.037	3.039	3.017	3.061	0.022
delta-BHC	3.149	3.151	3.148	3.155	3.153	3.155	3.152	3.151	3.152	3.131	3.173	0.021
Heptachlor	3.293	3.295	3.293	3.299	3.297	3.297	3.296	3.293	3.295	3.271	3.319	0.024
Aldrin	3.511	3.512	3.511	3.516	3.515	3.515	3.513	3.511	3.513	3.487	3.539	0.026
Heptachlor Epoxide	4.016	4.019	4.016	4.023	4.020	4.021	4.019	4.016	4.019	3.989	4.049	0.030
gamma-Chlordane	4.137	4.139	4.136	4.143	4.141	4.141	4.140	4.137	4.139	4.107	4.171	0.032
alpha-Chlordane	4.273	4.275	4.272	4.279	4.277	4.277	4.276	4.273	4.275	4.243	4.307	0.032
DDE	4.348	4.349	4.348	4.353	4.352	4.353	4.351	4.348	4.350	4.318	4.382	0.032
Endosulfan I	4.425	4.427	4.425	4.431	4.429	4.429	4.428	4.425	4.427	4.392	4.462	0.035
Dieldrin	4.711	4.712	4.709	4.716	4.713	4.715	4.713	4.709	4.712	4.680	4.744	0.032
Endrin	5.021	5.020	5.019	5.025	5.021	5.024	5.021	5.019	5.021	4.991	5.051	0.030
DDD	5.121	5.121	5.120	5.127	5.124	5.127	5.124	5.121	5.123	5.093	5.153	0.030
Endosulfan II	5.353	5.353	5.352	5.359	5.356	5.357	5.355	5.352	5.355	5.326	5.384	0.029
DDT	5.580	5.580	5.579	5.584	5.581	5.583	5.581	5.579	5.581	5.551	5.611	0.030
Endrin Aldehyde	6.104	6.104	6.103	6.108	6.107	6.107	6.104	6.101	6.105	6.073	6.137	0.032
Methoxychlor	6.624	6.623	6.624	6.629	6.625	6.628	6.624	6.620	6.625	6.587	6.663	0.038
Endosulfan Sulfate	7.085	7.085	7.085	7.091	7.087	7.089	7.085	7.083	7.086	7.050	7.122	0.036
Endrin Ketone	7.817	7.819	7.817	7.821	7.820	7.820	7.817	7.815	7.818	7.782	7.854	0.036
Oxychlordane	3.925	3.927	3.927	3.919	3.921	3.925	3.925	3.921	3.924	3.905	3.943	0.019
2,4'-DDE	3.999	4.000	4.000	3.992	3.995	4.000	4.000	3.995	3.998	3.976	4.020	0.022
trans-Nonachlor	4.257	4.259	4.259	4.251	4.253	4.259	4.259	4.253	4.256	4.233	4.279	0.023
2,4'-DDD	4.551	4.552	4.552	4.544	4.547	4.551	4.552	4.545	4.549	4.524	4.574	0.025
2,4'-DDT	4.880	4.880	4.880	4.872	4.875	4.879	4.880	4.875	4.878	4.855	4.901	0.023
cis-Nonachlor	5.072	5.072	5.072	5.064	5.067	5.071	5.072	5.067	5.070	5.048	5.092	0.022
Mirex	6.788	6.788	6.788	6.781	6.783	6.785	6.788	6.781	6.785	6.757	6.813	0.028
SURROGATE	1.0X	2.0X	4.0X	8.0X	16.0X	32.0X	48.0X	64.0X	RT	FROM	TO	WIDTH
TCX	2.472	2.475	2.472	2.477	2.476	2.476	2.475	2.473	2.474	2.454	2.494	0.020
DCB	10.804	10.804	10.803	10.807	10.807	10.805	10.801	10.801	10.804	10.754	10.854	0.050

Figure 4: TYPICAL ICAL RETENTION TIME WINDOW SUMMARY

INITIAL CALIBRATION
METHOD 8081

Lab Name : EMAX Inc
Instrument ID : E8 (PE CLARUS680 GC)
GC Column : STX-CLPESTICIDES2
Column size ID : 30MX0.32MMIDX0.25UM
LFID & Datetime: MA26018B 01/27/14 01:07 MA26026B 01/27/14 03:51
LFID & Datetime: MA26019B 01/27/14 01:28 MA26027B 01/27/14 04:11
LFID & Datetime: MA26020B 01/27/14 01:48 MA26028B 01/27/14 04:32
LFID & Datetime: MA26021B 01/27/14 02:08 MA26029B 01/27/14 04:52
LFID & Datetime: MA26022B 01/27/14 02:29 MA26030B 01/27/14 05:13
LFID & Datetime: MA26023B 01/27/14 02:49 MA26031B 01/27/14 05:33
LFID & Datetime: MA26024B 01/27/14 03:10 MA26032B 01/27/14 05:54
LFID & Datetime: MA26025B 01/27/14 03:30 MA26033B 01/27/14 06:14

	RT OF STANDARDS (MIN)								MEAN	RT WINDOW		RTWINDOW
COMPOUND	1.0X	2.0X	4.0X	8.0X	16.0X	32.0X	48.0X	64.0X	RT	FROM	TO	WIDTH
alpha-BHC	3.021	3.024	3.023	3.029	3.027	3.027	3.025	3.024	3.025	3.007	3.043	0.018
Hexachlorobenzene	2.941	2.944	2.945	2.936	2.939	2.944	2.943	2.939	2.941	2.923	2.959	0.018
gamma-BHC	3.261	3.264	3.261	3.268	3.267	3.267	3.264	3.263	3.264	3.247	3.281	0.017
beta-BHC	3.308	3.311	3.309	3.316	3.313	3.313	3.312	3.309	3.311	3.294	3.328	0.017
delta-BHC	3.541	3.545	3.543	3.549	3.547	3.548	3.545	3.544	3.545	3.528	3.562	0.017
Heptachlor	3.607	3.611	3.608	3.615	3.612	3.612	3.609	3.608	3.610	3.592	3.628	0.018
Aldrin	3.907	3.909	3.908	3.913	3.911	3.912	3.909	3.908	3.910	3.891	3.929	0.019
Heptachlor Epoxide	4.533	4.536	4.533	4.540	4.537	4.539	4.536	4.533	4.536	4.513	4.559	0.023
gamma-Chlordane	4.780	4.783	4.780	4.785	4.783	4.785	4.781	4.779	4.782	4.761	4.803	0.021
alpha-Chlordane	4.992	4.993	4.992	4.999	4.995	4.997	4.995	4.991	4.994	4.976	5.012	0.018
DDE	5.241	5.244	5.241	5.248	5.245	5.247	5.244	5.241	5.244	5.228	5.260	0.016
Endosulfan I	5.089	5.092	5.089	5.096	5.092	5.095	5.093	5.089	5.092	5.073	5.111	0.019
Dieldrin	5.548	5.551	5.549	5.556	5.552	5.555	5.551	5.548	5.551	5.534	5.568	0.017
Endrin	6.155	6.156	6.155	6.161	6.156	6.159	6.156	6.153	6.156	6.136	6.176	0.020
DDD	6.409	6.412	6.411	6.416	6.412	6.415	6.412	6.408	6.412	6.393	6.431	0.019
Endosulfan II	6.640	6.641	6.639	6.645	6.641	6.644	6.641	6.636	6.641	6.619	6.663	0.022
DDT	7.232	7.235	7.233	7.240	7.235	7.237	7.235	7.231	7.235	7.211	7.259	0.024
Endrin Aldehyde	7.568	7.571	7.569	7.575	7.571	7.573	7.569	7.567	7.570	7.548	7.592	0.022
Methoxychlor	9.704	9.707	9.705	9.711	9.708	9.709	9.705	9.703	9.707	9.679	9.734	0.028
Endosulfan Sulfate	8.473	8.476	8.475	8.479	8.476	8.479	8.475	8.471	8.476	8.451	8.500	0.024
Endrin Ketone	10.103	10.105	10.104	10.108	10.107	10.108	10.104	10.101	10.105	10.075	10.135	0.030
Oxychlordane	4.419	4.420	4.421	4.413	4.415	4.420	4.420	4.415	4.418	4.398	4.438	0.020
2,4'-DDE	4.769	4.771	4.772	4.764	4.767	4.771	4.771	4.767	4.769	4.748	4.790	0.021
trans-Nonachlor	4.908	4.908	4.909	4.901	4.904	4.908	4.909	4.904	4.906	4.888	4.924	0.018
2,4'-DDD	5.611	5.612	5.612	5.604	5.607	5.611	5.612	5.607	5.609	5.591	5.627	0.018
2,4'-DDT	6.243	6.243	6.244	6.236	6.239	6.244	6.244	6.240	6.242	6.223	6.261	0.019
cis-Nonachlor	6.328	6.328	6.329	6.321	6.324	6.329	6.329	6.325	6.327	6.309	6.345	0.018
Mirex	9.928	9.929	9.927	9.921	9.923	9.928	9.927	9.924	9.926	9.902	9.950	0.024
SURROGATE	1.0X	2.0X	4.0X	8.0X	16.0X	32.0X	48.0X	64.0X	RT	FROM	TO	WIDTH
TCX	2.640	2.643	2.640	2.647	2.644	2.644	2.643	2.641	2.643	2.620	2.666	0.023
DCB	12.231	12.233	12.232	12.236	12.236	12.235	12.231	12.229	12.233	12.198	12.268	0.035

Figure 5: TYPICAL PEM PEST BREAKDOWN CALCULATION SUMMARY

PEM PEST BREAKDOWN CALCULATION
METHOD 8081

Lab Name : EMAX
Instrument ID : GCTO16 HP-5890
GC Columm : RTX-CLPEST RTX-CLPESTII
Column size ID : .32MMX30M .32MMX30M
PEM LFID & Datetime : WF17026A WF17026B 06/18/10 00:50

Base on AREA

	AREA				% Breakdown				
LFID	DDD	DDE	DDT	TOTAL	DDD	DDE	TOTAL	QL	QCLIMIT
WF17026A	0.0	11917.0	826713.0	838630.0	0.00	1.42	1.42		15
WF17026B	21368.0	17092.0	933363.0	971823.0	2.20	1.76	3.96		15
LFID	ENDRIN	ENDRIN ALDEHYDE	ENDRIN KETONE	TOTAL	ENDRIN ALDEHYDE	ENDRIN KETONE	TOTAL	QL	QCLIMIT
WF17026A	893765.0	48233.0	35430.0	977428.0	4.93	3.62	8.56		15
WF17026B	945951.0	60214.0	48710.0	1054875.0	5.71	4.62	10.33		15

Figure 6: TYPICAL SAMPLE RESULT SUMMARY

METHOD SW3520C/8081A
PESTICIDES

Client : XYZ, INC.	Date Collected: 01/15/14
Project : CLEAN PROJECT	Date Received: 01/16/14
Batch No. : 14A051	Date Extracted: 01/21/14 14:00
Sample ID: EMW02012014	Date Analyzed: 01/28/14 18:09
Lab Samp ID: A051-03	Dilution Factor: 1
Lab File ID: MA28020A	Matrix : WATER
Ext Btch ID: CPA024W	% Moisture : NA
Calib. Ref.: MA28011A	Instrument ID : GCE8

PARAMETERS	RESULTS (ug/L)	LOQ (ug/L)	DL (ug/L)	LOD (ug/L)
ALPHA-BHC	(ND)|ND	0.10	0.0050	0.010
GAMMA-BHC	(ND)|ND	0.10	0.0050	0.010
BETA-BHC	(ND)|ND	0.10	0.0070	0.010
HEPTACHLOR	(ND)|ND	0.10	0.0050	0.010
DELTA-BHC	(ND)|ND	0.10	0.0070	0.010
ALDRIN	(ND)|ND	0.10	0.0050	0.010
HEPTACHLOR EPOXIDE	(ND)|ND	0.10	0.0050	0.010
GAMMA-CHLORDANE	(ND)|ND	0.10	0.0050	0.010
ALPHA-CHLORDANE	(ND)|ND	0.10	0.0050	0.010
ENDOSULFAN I	(ND)|ND	0.10	0.0080	0.010
4,4'-DDE	(ND)|ND	0.10	0.0050	0.010
DIELDRIN	(ND)|ND	0.10	0.0050	0.010
ENDRIN	(ND)|ND	0.10	0.0080	0.010
4,4'-DDD	(ND)|ND	0.10	0.0050	0.010
ENDOSULFAN II	(ND)|ND	0.10	0.0050	0.010
4,4'-DDT	(ND)|ND	0.10	0.0050	0.010
ENDRIN ALDEHYDE	(ND)|ND	0.10	0.0050	0.010
ENDOSULFAN SULFATE	(ND)|ND	0.10	0.0050	0.010
ENDRIN KETONE	(ND)|ND	0.10	0.0050	0.010
METHOXYCHLOR	(ND)|ND	1.0	0.050	0.10
TOXAPHENE	(ND)|ND	2.0	0.25	0.50
CHLORDANE	(ND)|ND	1.0	0.12	0.25

SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
TETRACHLORO-M-XYLENE	(0.3346)|0.3344	0.4000	(83.7)|83.6	25-140
DECACHLOROBIPHENYL	(0.4240)|0.4139	0.4000	(106)|103	30-135

RL : Reporting limit
Left of | is related to first column ; Right of | related to second column
Final result indicated by ( )

Figure 7: **TYPICAL LCS/LCSD REPORT SUMMARY**

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN PROJECT
BATCH NO.: 14A051
METHOD: SW3520C/8081A

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	MBLK1W				
LAB SAMP ID:	CPA024WB	CPA024WL	CPA024WC		
LAB FILE ID:	MA28015A	MA28016A	MA28017A		
DATE EXTRACTED:	01/21/1414:00	01/21/1414:00	01/21/1414:00	DATE COLLECTED:	NA
DATE ANALYZED:	01/28/1416:27	01/28/1416:47	01/28/1417:08	DATE RECEIVED:	01/21/14
PREP. BATCH:	CPA024W	CPA024W	CPA024W		
CALIB. REF:	MA28011A	MA28011A	MA28011A		

ACCESSION:

PARAMETER	BLNK RSLT (ug/L)	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
alpha-BHC	(ND)|ND	0.200	(0.203)|0.189	(101)|94	0.200	(0.204)|0.190	(102)|95	(0)|1	60-130	30
gamma-BHC	(ND)|ND	0.200	(0.202)|0.198	(101)|99	0.200	(0.202)|0.199	(101)|100	(0)|1	25-135	30
beta-BHC	(ND)|ND	0.200	0.206|(0.213)	103|(106)	0.200	0.204|(0.214)	102|(107)	1|(0)	65-125	30
Heptachlor	(ND)|ND	0.200	0.204|(0.208)	102|(104)	0.200	0.203|(0.206)	101|(103)	0|(1)	40-130	30
delta-BHC	(ND)|ND	0.200	(0.212)|0.204	(106)|102	0.200	(0.210)|0.203	(105)|101	(1)|0	45-135	30
Aldrin	(ND)|ND	0.200	(0.210)|0.205	(105)|102	0.200	(0.208)|0.204	(104)|102	(1)|0	25-140	30
Heptachlor Epoxide	(ND)|ND	0.200	(0.221)|0.216	(110)|108	0.200	0.212|(0.215)	106|(108)	4|(0)	60-130	30
gamma-Chlordane	(ND)|ND	0.200	0.215|(0.218)	108|(109)	0.200	0.213|(0.218)	106|(109)	1|(0)	60-125	30
alpha-Chlordane	(ND)|ND	0.200	(0.219)|0.212	(110)|106	0.200	(0.217)|0.210	(108)|105	(1)|1	65-125	30
Endosulfan I	(ND)|ND	0.200	(0.208)|0.204	(104)|102	0.200	(0.208)|0.202	(104)|101	(0)|1	50-110	30
4,4'-DDE	(ND)|ND	0.200	(0.224)|0.212	(112)|106	0.200	(0.221)|0.207	(110)|104	(1)|2	35-140	30
Dieldrin	(ND)|ND	0.200	(0.213)|0.212	(106)|106	0.200	(0.212)|0.209	(106)|104	(0)|1	60-130	30
Endrin	(ND)|ND	0.200	(0.228)|0.224	(114)|112	0.200	(0.229)|0.221	(114)|110	(0)|1	55-135	30
4,4'-DDD	(ND)|ND	0.200	0.217|(0.227)	108|(114)	0.200	0.213|(0.223)	106|(112)	2|(2)	25-150	30
Endosulfan II	(ND)|ND	0.200	(0.215)|0.209	(108)|104	0.200	(0.213)|0.207	(106)|104	(1)|1	30-130	30
4,4'-DDT	(ND)|ND	0.200	(0.255)|0.244	(127)|122	0.200	(0.253)|0.242	(126)|121	(1)|1	45-140	30
Endrin aldehyde	(ND)|ND	0.200	(0.213)|0.202	(106)|101	0.200	(0.211)|0.208	(105)|104	(1)|3	55-135	30
Endosulfan Sulfate	(ND)|ND	0.200	(0.226)|0.219	(113)|110	0.200	(0.225)|0.217	(112)|108	(0)|1	55-135	30
Endrin Ketone	(ND)|ND	0.200	0.208|(0.211)	104|(105)	0.200	0.207|(0.209)	104|(104)	0|(1)	75-125	30
Methoxychlor	(ND)|ND	2.00	2.30|(2.88)	115|(144)	2.00	2.28|(2.68)	114|(134)	1|(7)	55-150	30

SURROGATE PARAMETER	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	QC LIMIT (%)
Tetrachloro-m-xylene	0.4000	(0.3778)|0.3682	(94.4)|92.0	0.4000	(0.3608)|0.3530	(90.2)|88.3	25-140
Decachlorobiphenyl	0.4000	(0.4321)|0.4309	(108)|108	0.4000	(0.4262)|0.4226	(107)|106	30-135

Figure 8: **TYPICAL MS/MSD REPORT SUMMARY**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN PROJECT
BATCH NO.: 14A051
METHOD: SW3520C/8081A

MATRIX: WATER % MOISTURE: NA

DILUTION FACTOR:	1	1.1	1.02		
SAMPLE ID:	EMW02012014				
LAB SAMP ID:	A051-03	A051-03M	A051-03S		
LAB FILE ID:	MA28020A	MA28021A	MA28022A		
DATE EXTRACTED:	01/21/1414:00	01/21/1414:00	01/21/1414:00	DATE COLLECTED:	01/15/14
DATE ANALYZED:	01/28/1418:09	01/28/1418:29	01/28/1418:50	DATE RECEIVED:	01/16/14
PREP. BATCH:	CPA024W	CPA024W	CPA024W		
CALIB. REF:	MA28011A	MA28011A	MA28011A		

ACCESSION:

PARAMETER	SMPL RSLT (ug/L)	SPIKE AMT (ug/L)	MS RSLT (ug/L)	MS % REC	SPIKE AMT (ug/L)	MSD RSLT (ug/L)	MSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
alpha-BHC	(ND)|ND	0.220	(0.232)|0.217	(105)|99	0.204	(0.215)|0.201	(105)|99	(8)|8	60-130	30
gamma-BHC	(ND)|ND	0.220	0.227|(0.230)	103|(105)	0.204	(0.211)|0.211	(103)|103	(7)|9	25-135	30
beta-BHC	(ND)|ND	0.220	0.228|(0.253)	104|(115)	0.204	0.210|(0.231)	103|(113)	8|(9)	65-125	30
Heptachlor	(ND)|ND	0.220	0.230|(0.236)	105|(107)	0.204	0.211|(0.217)	103|(106)	9|(8)	40-130	30
delta-BHC	(ND)|ND	0.220	(0.235)|0.234	(107)|106	0.204	(0.217)|0.215	(106)|105	(8)|8	45-135	30
Aldrin	(ND)|ND	0.220	(0.236)|0.236	(107)|107	0.204	(0.218)|0.218	(107)|107	(8)|8	25-140	30
Heptachlor Epoxide	(ND)|ND	0.220	0.241|(0.243)	110|(110)	0.204	(0.233)|0.223	(114)|109	(3)|9	60-130	30
gamma-Chlordane	(ND)|ND	0.220	(0.243)|0.243	(110)|110	0.204	(0.225)|0.225	(110)|110	(8)|8	60-125	30
alpha-Chlordane	(ND)|ND	0.220	(0.249)|0.237	(113)|108	0.204	(0.229)|0.221	(112)|108	(8)|7	65-125	30
Endosulfan I	(ND)|ND	0.220	(0.235)|0.227	(107)|103	0.204	(0.217)|0.211	(106)|103	(8)|7	50-110	30
4,4'-DDE	(ND)|ND	0.220	(0.252)|0.234	(115)|106	0.204	(0.232)|0.219	(114)|107	(8)|7	35-140	30
Dieldrin	(ND)|ND	0.220	(0.245)|0.239	(111)|109	0.204	(0.227)|0.221	(111)|108	(8)|8	60-130	30
Endrin	(ND)|ND	0.220	(0.261)|0.253	(119)|115	0.204	(0.240)|0.235	(118)|115	(8)|7	55-135	30
4,4'-DDD	(ND)|ND	0.220	0.246|(0.256)	112|(116)	0.204	0.227|(0.238)	111|(117)	8|(7)	25-150	30
Endosulfan II	(ND)|ND	0.220	(0.242)|0.235	(110)|107	0.204	(0.225)|0.218	(110)|107	(7)|8	30-130	30
4,4'-DDT	(ND)|ND	0.220	(0.292)|0.278	(133)|126	0.204	(0.271)|0.257	(133)|126	(7)|8	45-140	30
Endrin aldehyde	(ND)|ND	0.220	(0.240)|0.236	(109)|107	0.204	(0.222)|0.214	(109)|105	(8)|10	55-135	30
Endosulfan Sulfate	(ND)|ND	0.220	(0.257)|0.249	(117)|113	0.204	(0.238)|0.230	(117)|113	(8)|8	55-135	30
Endrin Ketone	(ND)|ND	0.220	0.235|(0.240)	107|(109)	0.204	0.220|(0.223)	108|(109)	7|(7)	75-125	30
Methoxychlor	(ND)|ND	2.20	2.62|(3.05)	119|(139)	2.04	2.43|(2.82)	119|(138)	8|(8)	55-150	30

SURROGATE PARAMETER	SPIKE AMT (ug/L)	MS RSLT (ug/L)	MS % REC	SPIKE AMT (ug/L)	MSD RSLT (ug/L)	MSD % REC	QC LIMIT (%)
Tetrachloro-m-xylene	0.4400	(0.4345)|0.4180	(98.7)|95.0	0.4080	(0.3784)|0.3710	(92.7)|90.9	25-140
Decachlorobiphenyl	0.4400	(0.4836)|0.4728	(110)|107	0.4080	(0.4427)|0.4327	(109)|106	30-135

* : Out side of QC Limit.

Figure 9: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN PROJECT

SDG : 14A051

METHOD SW3520C/8081A
PESTICIDES

A total of three (3) water samples were received on 01/16/14 for Pesticides Organochlorine analysis, Method SW3520C/8081A in accordance with Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2 and Project QAPP 5/2012.

Holding Time
Samples were analyzed within the prescribed holding time.

Instrument Performance and Calibration
Instrument performance was checked prior to calibration. DDT and Endrin breakdown were within specification. Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using secondary source (ICV). Continuing calibration (CCV) was carried on at a frequency required by the project. All project calibration requirements were satisfied. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank
Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample
A set of LCS/LCD was analyzed with the samples in this SDG.
Percent recoveries for CPA024WL/C were all within QC limits.

Matrix QC Sample
A set of MS/MSD was analyzed with the samples in this SDG.
Percent recoveries for A051-03M/S were within project QC limits.

Surrogate
Surrogates were added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis
Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter. Positive sample results were confirmed by a second column. Relative percentage difference (RPD) between the two results was evaluated. If RPD is less than 40% and peaks are well defined the higher result is reported. Where RPD is greater than 40% the chromatogram is checked for anomalies and results are selected based on processed knowledge. If there is no evidence of any chromatographic ambiguity, the higher result is reported.

Table 1: ICAL CONCENTRATION OF INDIVIDUAL ANALYTES

PARAMETERS	ICAL STANDARD CONCENTRATION (µg/L)							
PESTICIDES	1	2	3	4	5	6	7	8
Aldrin	1.25	2.5	5	10	20	40	60	80
alpha-BHC	1.25	2.5	5	10	20	40	60	80
beta-BHC	1.25	2.5	5	10	20	40	60	80
delta-BHC	1.25	2.5	5	10	20	40	60	80
gamma-BHC	1.25	2.5	5	10	20	40	60	80
alpha-Chlordane	1.25	2.5	5	10	20	40	60	80
gamma-Chlordane	1.25	2.5	5	10	20	40	60	80
Heptachlor	1.25	2.5	5	10	20	40	60	80
Heptachlor Epoxide	1.25	2.5	5	10	20	40	60	80
Endosulfan I	1.25	2.5	5	10	20	40	60	80
4,4’ – DDD	2.5	5	10	20	40	80	120	160
4,4’ – DDE	2.5	5	10	20	40	80	120	160
4,4’ – DDT	2.5	5	10	20	40	80	120	160
Dieldrin	2.5	5	10	20	40	80	120	160
Endrin	2.5	5	10	20	40	80	120	160
Endosulfan II	2.5	5	10	20	40	80	120	160
Endosulfan Sulfate	2.5	5	10	20	40	80	120	160
Endrin Aldehyde	2.5	5	10	20	40	80	120	160
Endrin Ketone	2.5	5	10	20	40	80	120	160
Methoxychlor	12.5	25	50	100	200	400	600	800
2,4’ – DDD	1.25	2.5	5	10	20	40	60	80
2,4’ – DDE	1.25	2.5	5	10	20	40	60	80
2,4’ – DDT	1.25	2.5	5	10	20	40	60	80
Oxychlordane	1.25	2.5	5	10	20	40	60	80
cis-Nonachlor	1.25	2.5	5	10	20	40	60	80
Trans-Nonachlor	1.25	2.5	5	10	20	40	60	80
Mirex	1.25	2.5	5	10	20	40	60	80
Tetrachloro-m-xylene (surrogate)	1.25	2.5	5	10	20	40	60	80
Decachlorobiphenyl (Surrogate)	1.25	2.5	5	10	20	40	60	80
TOXAPHENE	50	100	250	500	750	1000	1500	2000
TECHNICAL CHLORDANE	25	100	250	500	750	1000	1500	2000

Table 2: ICAL STANDARD PREPARATION

Standard #	Compound Name	Intermediate Solution Conc. (µg/L)	Preparation			Final Conc. (µg/L)
			Aliquot (µL)	Solvent	Final Volume (µL)	
PESTICIDES						
1	Pest ICAL Mix1	80 / 160 / 800	12.5	Hexane	800	1.25 /2. 5 / 12.5
2	Pest ICAL Mix1	80 / 160 / 800	25	Hexane	800	2.5 / 5 / 25
3	Pest ICAL Mix1	80 / 160 / 800	50	Hexane	800	5 / 10 / 50
4	Pest ICAL Mix1	80 / 160 / 800	100	Hexane	800	10 / 20 / 100
5	Pest ICAL Mix1	80 / 160 / 800	200	Hexane	800	20 / 40 / 200
6	Pest ICAL Mix1	80 / 160 / 800	400	Hexane	800	40 / 80 / 400
7	Pest ICAL Mix1	80 / 160 / 800	600	Hexane	800	60 / 120 / 600
8	Pest ICAL Mix1	80 / 160 / 800	800	-	800	80 / 160 / 800
TOXAPHENE						
1	Toxaphene ICAL	2000	20	Hexane	800	50
2	Toxaphene ICAL	2000	40	Hexane	800	100
3	Toxaphene ICAL	2000	100	Hexane	800	250
4	Toxaphene ICAL	2000	200	Hexane	800	500
5	Toxaphene ICAL	2000	300	Hexane	800	750
6	Toxaphene ICAL	2000	400	Hexane	800	1000
7	Toxaphene ICAL	2000	600	Hexane	800	1500
8	Toxaphene ICAL	2000	800	-	800	2000
TECHNICAL CHLORDANE						
1	Tech. Chlordane ICAL	2000	10	Hexane	800	25
2	Tech. Chlordane ICAL	2000	40	Hexane	800	100
3	Tech. Chlordane ICAL	2000	100	Hexane	800	250
4	Tech. Chlordane ICAL	2000	200	Hexane	800	500
5	Tech. Chlordane ICAL	2000	300	Hexane	800	750
6	Tech. Chlordane ICAL	2000	400	Hexane	800	1000
7	Tech. Chlordane ICAL	2000	600	Hexane	800	1500
8	Tech. Chlordane ICAL	2000	800	-	800	2000
PESTICIDES MIX 3						
1	Pest ICAL Mix 3	80	12.5	Hexane	800	1.25
2	Pest ICAL Mix 3	80	25	Hexane	800	2.5
3	Pest ICAL Mix 3	80	50	Hexane	800	5
4	Pest ICAL Mix 3	80	100	Hexane	800	10
5	Pest ICAL Mix 3	80	200	Hexane	800	20
6	Pest ICAL Mix 3	80	400	Hexane	800	40
7	Pest ICAL Mix 3	80	600	Hexane	800	60
8	Pest ICAL Mix 3	80	800	-	800	80

Table 3: INTERMEDIATE PRIMARY STANDARD PREPARATION

PESTICIDES MIX 1*: RESTEK*	Stock Std. Conc. (µg/mL)	Final Conc. (µg/L)	Preparation
Aldrin	8	80	Using a gas-tight syringe, measure 1000 µL of (Restek) Pesticides Mix 1 and 40 µL of (Supelco) Pest Surrogate and dilute with hexane to 100 mL.
alpha-BHC	8	80	
beta-BHC	8	80	
delta-BHC	8	80	
gamma-BHC	8	80	
alpha-Chlordane	8	80	
gamma-Chlordane	8	80	
Heptachlor	8	80	
Heptachlor Epoxide	8	80	
Endosulfan I	8	80	
4,4'-DDD	16	160	
4,4'-DDE	16	160	
4,4'-DDT	16	160	
Dieldrin	16	160	
Endrin	16	160	
Endosulfan II	16	160	
Endosulfan Sulfate	16	160	
Endrin Aldehyde	16	160	
Endrin Ketone	16	160	
Methoxychlor	80	800	
Surrogate: *SUPELCO*			
Tetrachloro-m-xylene	200	80	
Decachlorobiphenyl	200	80	
TOXAPHENE: *AccuStandard*			
Toxaphene	1000	2000	Using a gas-tight syringe, measure 200 µL of (AccuStandard) Toxaphene standard and 50 µL of (Supelco) Pest Surrogate and dilute with hexane to 100 mL.
Surrogate: *SUPELCO*			
Tetrachloro-m-xylene	200	100	
Decachlorobiphenyl	200	100	
TECHNICAL CHLORDANE: *AccuStandard*			
Technical Chlordane	1000	2000	Using a gas-tight syringe, measure 200 µL of (AccuStandard) Tech. Chlordane standard and 50 µL of (AccuStandard) Pest Surrogate and dilute with hexane to 100 mL.
Surrogate: *SUPELCO*			
Tetrachloro-m-xylene	200	100	
Decachlorobiphenyl	200	100	
PESTICIDES MIX 3*: SPEXCERTIPREP*			
2,4'-DDD	1000	80	Using a gas-tight syringe, measure 8 µL of (SpexCertiPrep) Pesticides Mix3 and dilute with hexane to 100 mL.
2,4'-DDE	1000	80	
2,4'-DDT	1000	80	
Oxychlordane	1000	80	
cis-Nanochlordane	1000	80	
trans-Nanochlordane	1000	80	
Mirex	1000	80	

Table 4: INTERMEDIATE SECONDARY STANDARD PREPARATION

PESTICIDES MIX 1*: SUPELCO*	Stock Std. Conc. (µg/mL)	Final Conc. (µg/L)	Preparation
Aldrin	2000	200	Using a gas-tight syringe, measure 10 µL of (Supelco) Pesticides Mix and 180 µL of (SpexCertiPrep) Methoxychlor and dilute with hexane to 100 mL.
alpha-BHC	2000	200	
beta-BHC	2000	200	
delta-BHC	2000	200	
gamma-BHC	2000	200	
alpha-Chlordane	2000	200	
gamma-Chlordane	2000	200	
Heptachlor	2000	200	
Heptachlor Epoxide	2000	200	
Endosulfan I	2000	200	
4,4'-DDD	2000	200	
4,4'-DDE	2000	200	
4,4'-DDT	2000	200	
Dieldrin	2000	200	
Endrin	2000	200	
Endosulfan II	2000	2002	
Endosulfan Sulfate	2000	200	
Endrin Aldehyde	2000	200	
Endrin Ketone	2000	200	
Methoxychlor	2000	2000	
Methoxychlor ***(SpexCertiPrep)***	1000		
Tetrachloro-m-xylene	200	200	
Decachlorobiphenyl	200	200	
TOXAPHENE: *Ultra Scientific*			
Toxaphene	2500	2000	Using a gas-tight syringe, measure 20 µL of (Ultra Scientific) Toxaphene standard and 12.5 µL of (Supelco) Pest Surrogate and dilute with hexane to 25 mL.
Tetrachloro-m-xylene	200	100	
Decachlorobiphenyl	200	100	
TECHNICAL CHLORDANE: *Ultra Scientific*			
Technical Chlordane	100	2000	Using a gas-tight syringe, measure 500 µL of (Ultra Scientific) Tech. Chlordane standard and 12.5 µL of (Supelco) Pest Surrogate and dilute with hexane to 100 mL.
Tetrachloro-m-xylene	200	100	
Decachlorobiphenyl	200	100	
PESTICIDES MIX 3*: CPI*			
2,4'-DDD	1000	80	Using a gas-tight syringe, measure 4 µL of (CPI) Pesticides Mix3 and 40 µL of (AccuStandard) Mirex and dilute with hexane to 100 mL.
2,4'-DDE	1000	80	
2,4'-DDT	1000	80	
Oxychlordane	1000	80	
cis-Nanochlordane	100	80	
trans-Nanochlordane	100	80	
Mirex ***(AccuStandard)***	100	80	

Note: Table 2 and Table 2A may be interchanged. However, the source of LCS shall follow the source of the secondary standard or from a third vendor.

Table 5: CHECK STANDARD PREPARATION (DCC/ICV)

Standard	Compound Name	Intermediate Soln. Conc. (µg/L)	Source	Preparation			Final Conc. (µg/L)
				Aliquot (µL)	Dil.Soln./ Modifier	Final Vol. (µL)	
PESTICIDES							
DCC	Pest ICAL Mix	80/160/800	Restek	200	Hexane	800	20/40/200
	Surrogate Mix	80	AccuStandard				20
ICV	Pest ICV Mix	200/2000	Supelco / SpexCertiPrep	100	Hexane	1000	20/200
	Surrogate Mix	200	Supelco				20
TOXAPHENE							
DCC	Toxaphene ICAL	2000	AccuStandard	200	Hexane	800	500
	Surrogate Mix	100	AccuStandard				25
ICV	Toxaphene ICV	200	Ultra Scientific	200	Hexane	800	500
	Surrogate Mix	100	Supelco				25
TECHNICAL CHLORDANE							
DCC	Toxaphene ICAL	2000	AccuStandard	200	Hexane	800	500
	Surrogate Mix	100	AccuStandard				25
ICV	Toxaphene ICV	200	Ultra Scientific	200	Hexane	800	500
	Surrogate Mix	100	Supelco				25

Table 6: SURROGATE STANDARD PREPARATION

Compound Name	Stock Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/L)
			Aliquot (mL)	Dil.Soln./ Modifier	Final Vol. (mL)	
Surrogate Mix (Tetrachloro-m-xylene & Decachlorobiphenyl)	200	Supelco	2.0	Hexane	1,000	400

Table 7: SPIKE STANDARD PREPARATION

Compound Name	Stock/Intermediate Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/L)
			Aliquot (µL)	Dil.Soln./ Modifier	Dil. Vol. (mL)	
Pesticide Matrix Spike Mix	2,000	Supelco	20	Hexane	100	400/4000
Methoxychlor	1,000	SpexCertiPrep	360			

Table 8: PERFORMANCE EVALUATION MIXTURE PREPARATION

Compound Name	Stock/Intermediate Soln. Conc. (µg/mL)	Source	Preparation			Final Conc (µg/L)
			Aliquot (µL)	Dil.Soln./ Modifier	Dil. Vol. (mL)	
PEM (DDT + Endrin Mix)	500	Supelco	20	Hexane	100	100/100

Table 9: CLP RETENTION TIME WINDOWS (CLP OLM4.2 D-79/PEST)

Compound	Retention Time Window (minutes)	Compound	Retention Time Window (minutes)
alpha-BHC	± 0.05	Endrin Ketone	± 0.07
beta-BHC	± 0.05	4,4'-DDD	± 0.07
gamma-BHC(Lindane)	± 0.05	4,4'-DDE	± 0.07
delta-BHC	± 0.05	4,4'-DDT	± 0.07
Heptachlor	± 0.05	Endosulfan II	± 0.07
Aldrin	± 0.05	Endosulfan Sulfate	± 0.07
alpha-Chlordane	± 0.07	Methoxychlor	± 0.07
gamma-Chlordane	± 0.07	Aroclors	± 0.07
Heptachlor Epoxide	± 0.07	Toxaphene	± 0.07
Dieldrin	± 0.07		
Endrin	± 0.07	Tetrachloro-m-xylene	± 0.05
Endrin Aldehyde	± 0.07	Decachlorobiphenyl	± 0.10

Table 10: ESTABLISHED LIMIT OF DETECTION (LOD) & LIMIT OF QUANTITATION (LOQ)

MATRIX: AQUEOUS	DL	LOD	LOQ	Unit
Aldrin	0.005	0.01	0.1	µg/L
Alpha-BHC	0.005	0.01	0.1	µg/L
Beta-BHC	0.007	0.01	0.1	µg/L
Delta-BHC	0.007	0.01	0.1	µg/L
Gamma-BHC (Lindane)	0.005	0.01	0.1	µg/L
DDD (4,4)	0.005	0.01	0.1	µg/L
DDE (4,4)	0.005	0.01	0.1	µg/L
DDT (4,4)	0.005	0.01	0.1	µg/L
Dieldrin	0.005	0.01	0.1	µg/L
Endosulfan I	0.008	0.01	0.1	µg/L
Endosulfan II	0.005	0.01	0.1	µg/L
Endosulfan Sulfate	0.005	0.01	0.1	µg/L
Endrin	0.008	0.01	0.1	µg/L
Endrin Aldehyde	0.005	0.01	0.1	µg/L
Heptachlor	0.007	0.01	0.1	µg/L
Heptachlor epoxide	0.005	0.01	0.1	µg/L
Methoxychlor	0.05	0.1	1	µg/L
Alpha-Chlordane	0.005	0.01	0.1	µg/L
Gamma-Chlordane	0.005	0.01	0.1	µg/L
Endrin Ketone	0.005	0.01	0.1	µg/L
Toxaphene	0.25	0.5	2	µg/L
Technical Chlordane	0.005	0.25	1	µg/L
SURROGATES				
Tetrachloro-m-xylene	0.005	0.01	0.02	µg/L
Decachlorobiphenyl	0.005	0.01	0.02	µg/L

Reference: Detection Limit Study dated April 20,2010.

PARAMETER (MATRIX: SOIL)	DL	LOD	LOQ	Unit
Aldrin	0.02	0.4	2	µg/Kg
Alpha-BHC	0.02	0.4	2	µg/Kg
Beta-BHC	0.02	0.4	2	µg/Kg
Delta-BHC	0.27	0.4	2	µg/Kg
Gamma-BHC (Lindane)	0.2	0.4	2	µg/Kg
DDD (4,4)	0.02	0.4	2	µg/Kg
DDE (4,4)	0.02	0.4	2	µg/Kg
DDT (4,4)	0.02	0.4	2	µg/Kg
Dieldrin	0.02	0.4	2	µg/Kg
Endosulfan I	0.2	0.4	2	µg/Kg
Endosulfan II	0.2	0.4	2	µg/Kg
Endosulfan Sulfate	0.2	0.4	2	µg/Kg
Endrin	0.2	0.4	2	µg/Kg
Endrin Aldehyde	0.35	0.4	2	µg/Kg
Heptachlor	0.2	0.4	2	µg/Kg
Heptachlor epoxide	0.2	0.4	2	µg/Kg
Methoxychlor	2	4	10	µg/Kg
Alpha-Chlordane	0.2	0.4	2	µg/Kg
Gamma-Chlordane	0.2	0.4	2	µg/Kg
Endrin Ketone	0.2	0.4	2	µg/Kg
Toxaphene	5	10	50	µg/Kg
Technical Chlordane	10	20	50	µg/Kg
SURROGATES				
Tetrachloro-m-xylene	0.167	0.33	0.7	µg/Kg
Decachlorobiphenyl	0.167	0.33	0.7	µg/Kg

Reference: Detection Limit Study dated April 5,2010.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURE

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
5 point Initial Calibration for analytes	Initially, as needed	RSD for all analytes ≤ 20% or Linear – least squares or inverse weighting factor: r ≥ 0.995.	Correct then problem then repeat initial calibration.		
Second –source Calibration Verification	Once per 5-point initial calibration	All analytes within ± 20of expected value from the ICAL.	Repeat injection of ICV. If the problem persists, perform troubleshooting and repeat the ICAL.		
Initial Calibration Verification Check	Daily, before sample analysis	All analytes within ± 20%	Correct the problem. If problem persists, repeat initial calibration		
Calibration Verification	Every 12 hours of analysis time and at the end of the analysis sequence	All analytes within ± 20%	Correct the problem then repeat initial calibration verification and re-analyze all samples since last successful calibration verification		
Breakdown check (Endrin and DDT)	Every 12-hours	Degradation ≤ 15% of each analyte.	Repeat breakdown check		
Method Blank	One per preparation batch	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Re-prep and re-analyze method blank and all samples processed with the contaminated blank		
LCS	One LCS per preparation batch	Within project QC limits	Re-prep and re-analyze the LCS and all associated samples		
Surrogate spike	Every sample, spiked sample, standard and method blank	Within project QC limits	Correct the problem then re-extract and re-analyze sample		
MS/MSD	One MS/MSD per every 20 project samples per matrix	Within project QC limits	If chromatogram is indicative of matrix interference, discuss in case narrative. Otherwise, check for probable source of error and perform corrective action as necessary		
Confirmation	100% for all positive results	Same as primary column	If quantitation criteria are not met, use confirmation for qualitative identification only.		
Comments: 1. For flagging criteria refer to PSR. Otherwise, if MB is non-compliant, apply B to specific analyte(s) on all associated samples; apply J to all values between LOD and LOQ.				Reviewed By:	
				Date	

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
METHOD: EPA 3520C / EPA 8081B

Sample Prep SOP: EMAX-3520 Rev. 5
Analytical SOP: EMAX-8081
Conc Unit: µg/L
Sample Amt(ml): 1000
Extract Volume (mL): 10

Instrument ID: E8
Extraction date: 9/12 & 9/16/13
Extracted by: Iragasa / Jmuertigue
Analysis date: 9/18 & 9/19/13
Analyzed by: E. Santos

PARAMETER	CPI018WL	CPI018WC	CPI023WL	CPI023WC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	RI18020A/B	RI18021A/B	RI18060A/B	RI18061A/B							
Aldrin	0.213	0.222	0.233	0.234	0.2	0.2	113	0.010	4.5	70 - 130	PASSED
alpha-BHC	0.219	0.231	0.250	0.245	0.2	0.2	118	0.014	5.9	70 - 140	PASSED
beta-BHC	0.241	0.275	0.299	0.269	0.2	0.3	135	0.024	8.7	70 - 140	PASSED
delta-BHC	0.235	0.248	0.263	0.261	0.2	0.3	126	0.013	5.3	70 - 140	PASSED
gamma-BHC (Lindane)	0.205	0.213	0.228	0.227	0.2	0.2	109	0.011	5.2	60 - 140	PASSED
DDD (4,4)	0.217	0.223	0.244	0.237	0.2	0.2	115	0.012	5.4	70 - 140	PASSED
DDE (4,4)	0.215	0.224	0.238	0.234	0.2	0.2	114	0.010	4.4	70 - 130	PASSED
DDT (4,4)	0.240	0.251	0.269	0.256	0.2	0.3	127	0.012	4.8	70 - 150	PASSED
Dieldrin	0.218	0.224	0.248	0.236	0.2	0.2	116	0.013	5.8	70 - 130	PASSED
Endosulfan I	0.206	0.213	0.228	0.224	0.2	0.2	109	0.010	4.7	70 - 130	PASSED
Endosulfan II	0.222	0.229	0.250	0.241	0.2	0.2	118	0.013	5.3	70 - 130	PASSED
Endosulfan sulfate	0.224	0.231	0.250	0.244	0.2	0.2	119	0.012	5.0	70 - 140	PASSED
Endrin	0.217	0.225	0.238	0.228	0.2	0.2	113	0.009	3.8	70 - 140	PASSED
Endrin Aldehyde	0.211	0.211	0.247	0.242	0.2	0.2	114	0.019	8.5	70 - 140	PASSED
Heptachlor	0.208	0.218	0.226	0.225	0.2	0.2	110	0.008	3.9	60 - 130	PASSED
Heptachlor epoxide	0.215	0.220	0.236	0.231	0.2	0.2	113	0.010	4.3	70 - 130	PASSED
Methoxychlor	2.160	2.253	2.462	2.477	2	2	117	0.157	6.7	70 - 140	PASSED
alpha-Chlordane	0.211	0.219	0.232	0.228	0.2	0.2	111	0.009	4.2	70 - 130	PASSED
gamma-Chlordane	0.224	0.232	0.248	0.244	0.2	0.2	119	0.011	4.6	70 - 140	PASSED
Endrin Ketone	0.223	0.221	0.240	0.236	0.2	0.2	115	0.009	4.1	70 - 130	PASSED
Tcx	0.365	0.396	0.398	0.406	0.4	0.4	98	0.018	4.6	30 - 130	PASSED
DCB	0.356	0.368	0.368	0.367	0.4	0.4	91	0.006	1.6	60 - 130	PASSED

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
METHOD: EPA 3550C / EPA 8081B

Sample Prep SOP: EMAX-3550 Rev. 3
Analytical SOP: EMAX-8081 Rev. 7
Conc Unit: µg/Kg
Sample Amt(g): 30
Extract Volume (mL): 10

Instrument ID: E8
Extraction date: 1/16 & 1/21/14
Extracted by: J. Villena
Analysis date: 1/29 & 1/31/14
Analyzed by: E. Santos

PARAMETER	CPA019SL	CPA019SC	CPA033SL	CPA022SC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	MA29007A	MA29008A	MA31013A	MA31014A							
Aldrin	6.30	6.29	7.15	7.07	6.67	6.7	101	0.474	7.1	60 - 130	PASSED
alpha-BHC	6.21	6.20	7.26	7.29	6.67	6.7	101	0.616	9.1	50 - 140	PASSED
beta-BHC	6.28	6.31	7.47	7.44	6.67	6.9	103	0.670	9.7	60 - 140	PASSED
delta-BHC	6.59	6.63	7.59	7.53	6.67	7.1	106	0.546	7.7	60 - 140	PASSED
gamma-BHC (Lindane)	6.29	6.27	7.08	6.96	6.67	6.6	100	0.431	6.5	60 - 130	PASSED
DDD (4,4)	7.53	7.63	7.47	7.42	6.67	7.5	113	0.090	1.2	70 - 140	PASSED
DDE (4,4)	6.67	6.75	7.47	7.39	6.67	7.1	106	0.419	5.9	60 - 140	PASSED
DDT (4,4)	8.07	8.16	8.82	8.89	6.67	8.5	127	0.431	5.1	70 - 150	PASSED
Dieldrin	6.56	6.58	7.45	7.40	6.67	7.0	105	0.493	7.0	60 - 140	PASSED
Endosulfan I	6.14	6.12	7.23	7.11	6.67	6.6	100	0.605	9.1	50 - 130	PASSED
Endosulfan II	6.74	7.00	7.58	7.53	6.67	7.2	108	0.410	5.7	70 - 130	PASSED
Endosulfan sulfate	7.02	7.31	7.79	7.74	6.67	7.5	112	0.365	4.9	60 - 140	PASSED
Endrin	7.16	7.29	7.43	7.34	6.67	7.3	110	0.112	1.5	70 - 140	PASSED
Endrin Aldehyde	6.53	6.68	7.37	7.17	6.67	6.9	104	0.396	5.7	60 - 140	PASSED
Heptachlor	6.19	6.28	7.08	7.14	6.67	6.7	100	0.506	7.6	60 - 130	PASSED
Heptachlor epoxide	6.32	6.59	7.29	7.31	6.67	6.9	103	0.502	7.3	60 - 130	PASSED
Methoxychlor	81.5	84.7	92.7	92.8	66.7	88	132	5.686	6.5	60 - 140	PASSED
alpha-Chlordane	6.59	6.60	7.33	7.28	6.67	6.9	104	0.410	5.9	60 - 130	PASSED
gamma-Chlordane	6.52	6.64	7.41	7.32	6.67	7.0	105	0.458	6.6	60 - 130	PASSED
Endrin Ketone	6.61	6.83	7.52	7.52	6.67	7.1	107	0.471	6.6	70 - 130	PASSED
Tcx	10.71	10.24	11.91	11.83	13.3	11.2	84	0.827	7.4	50 - 140	PASSED
DCB	13.1	12.7	13.9	13.7	13.3	13.4	100	0.545	4.1	50 - 140	PASSED

8081FS: SAMPLE PREPARATION LOG

Prep BatchID	**Lab SampleID**	**Aliquot**	**Unit**	**DateTime**	**Ve (ml)**	**EA**	**EVe (ml)**	**PF**	FlorisilCleanup EMAX-3620	SulfurCleanup EMAX-3660	AcidCleanup EMAX-3665	Comments

☐ ExtractionStarted @ **Ve**=ExtractVolume **EA**= ExpectedAliquot **EVe**=ExpectedExtractVolume **PF**=(EA/Aliquot)(Ve/EVe)

☐ Extraction Ended @ Prepared By:

Comment: [1]Acid cleanup is only for PCB anlaysis. Checked By:

Date

8081FA: **ANALYTICAL RUN LOG**

EMAX LABORATORIES, INC.

ANALYSIS RUN LOG
for
PESTICIDES/PCBs

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Comments:

Book #:	AE8-007
Instrument No.:	E8
Analytical Sequence:	
Method File:	
Analytical Batch:	

SOP #	Rev. #
☐ EMAX-8081	
☐ EMAX-8082	
☐ EMAX-608	
☐ EMAX-	

ICAL REFERENCE

ICAL ID	BATCH	DATE

STANDARDS ID	Conc (µg/L)
Hexane	

Column	☐ A. Elite-CLP I B. Elite-CLP II
	☐ A. RTX/STX CLPEST I B. RTX/STX CLPEST II
	☐ A. ZB-MULTI RES I B. ZB-MULTI RES II

ELECTRONIC DATA ARCHIVAL

Location	Date
☐ EZChrom Elite_PEST E8	

Analyzed By: Date:

Date Disposed: Disposed By:

This page is checked during data review.

8081FM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: E8 Month: Year: 201_ Book #: ME8-004

Date							
Column Head Pressure-Initial (psi)							
Column Head Pressure-Final (psi)							
Carrier Gas Flowrate (ml/min)							
Channel A Signal (mV)							
Channel B Signal (mV)							
Autosampler							
Update Cal. Table							
Computer Date/Time							
Septa Change							
Liner Change/Clean-up							
Analyst Initial							
TroubleShooting							
Problems Encountered							
Return to Control (Analyst Initial)							

Code	Problems Encountered	Code	Corrective Action

STANDARD OPERATING PROCEDURES

POLYCHLORINATED BIPHENYLS (PCB) AND POLYCHLORINATED TERPHENYLS (PCT) BY GAS CHROMATOGRAPHY

SOP No.:	EMAX-8082	Revision No.	5	Effective Date:	02-Jul-14
Prepared By:	Tu Nisamaneepong			Date:	07-02-14
Approved By:	Kenette Pimentel QA Manager			Date:	07.02.14
Approved By:	Caspar Pang Laboratory Director			Date:	07-02-14

Control Number: 8082-05-

1.0 SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentration of Polychlorinated Biphenyls (PCBs) and Polychlorinated Terphenyls (PCTs) as Aroclors in soil, sediment, sludge, and wastewater samples by gas chromatography method.

1.2. This SOP is an adaptation of EPA 8082A. Since EPA 8082A is an update and enhancement of 8082, this SOP is also applicable to EPA 8082.

2.0 SUMMARY OF METHOD

2.1. This method provides gas chromatographic conditions for the detection of PCB and PCT compounds with dual Electron Capture Detector (ECD). The samples are extracted in methylene chloride, exchanged to hexane and cleaned up by appropriate method before GC analysis.

2.2. **Interferences**

2.2.1. Interferences by phthalate esters co-extracted from the sample or introduced during sample preparation can pose a major problem in PCB and PCT determinations. Interferences can be minimized by avoiding contact with any plastic materials and checking all solvents for phthalate contamination. Glassware must be scrupulously cleaned. Sulfuric acid/permanganate cleanup technique can be used for Phthalate esters removal from the extract.

2.2.2. The presence of elemental sulfur will result in broad peaks that may cause chromatographic interfere with the determination of PCBs and PCTs. Sulfur contamination is most likely present in sediment samples. The TBA procedure, GPC or other cleanup technique can be used for sulfur removal from the extract.

2.2.3. Other interferences such as aliphatic compounds, aromatics and nitrogen-containing compounds may be eliminated by using Florisil cleanup.

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.

3.1.2. Refer to Table 6 for established DL, LOD and LOQ values.

4.0 DYNAMIC RANGE

4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point (see Sec. 9.4). All samples analyzed above this concentration are considered "over-range" and shall require dilution to properly quantitate.

4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required to properly quantitate.

4.3. Typical dynamic ranges are:

	PCBs	PCTs
Water (µg/L)	0.5 - 20.0	2.0 - 10.0
Soil (µg/Kg)	16.67 – 666.67	40 - 500

5.0 SAMPLE HOLDING TIME & PRESERVATION

5.1. **Holding Time**

5.1.1. PCBs and PCTs are very stable in a variety of matrices and holding times may be as long as a year.

5.1.2. Analysis should be within 40 days after extraction completion date.

5.2. **Preservation**

5.2.1. Samples and extract should be kept at ≤ 6°C.

6.0 ASSOCIATED SOPs

6.1. EMAX-DM01 Data Flow and Review

6.2. EMAX-QA014 Detection Limit

6.3. EMAX-QA08 Corrective Action

6.4. EMAX-QC02 Analytical Standard Preparation

6.5. EMAX-SM04 Analytical and QC Labeling

6.6. EMAX-3520 Extraction, Continuous Liquid/Liquid

6.7. EMAX-3546 Extraction, Microwave

6.8. EMAX-3550 Extraction, Pulse Sonication

6.9. EMAX-3540 Extraction, Soxhlet

6.10. EMAX-3580 Waste Dilution

6.11. EMAX-3620 Cleanup, Florisil

6.12. EMAX-3660 Cleanup, Sulfur

6.13. EMAX-3665 Cleanup, Acid/Permanganate

7.0 SAFETY

7.1. Read all SDS of chemicals listed in this SOP.

7.2. Treat all reagents, standards, and samples as potential hazards. ECD contains minute quantity of Radioactive Ni (63), a wipe test performed by experienced personnel or manufacturer should be conducted semiannually or sooner if potential problem is suspected. Observe standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.

7.3. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of the body, rinse the affected body part thoroughly with copious amounts of water. If irritations or any other discomfort related to the incident persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS & REAGENTS

8.1. **Instruments and Supplies**

Gas Chromatography	HP 5890 Series II
Detector	Dual Electron Capture Detectors
Column	RTX CLPESTI (30 m x 0.32 mm x 0.5 μm) RTX CLPESTII (30 m x 0.32 mm x 0.25 μm) (Alternate columns may be used after verification of performance)
Data System	EZ Chrom
Auto Sampler	HP Model 7673B or equivalent
Gas	Ultra-high purity hydrogen Ultra-high purity nitrogen
Microsyringes	10, 25, 100 and 500 μL with a 0.006 mm ID needle
Volumetric Flasks	0,50, and 100 ml with ground glass stopper
Transfer Pipette	Pasteur

8.2. **Chemicals and Reagents**

Solvent (GC-grade)	Pesticide-free grade hexane, methylene chloride, isopropanol

9.0 STANDARDS

9.1. **Preparation**

9.1.1. Prepare and label analytical standards according to EMAX-QC02 and EMAX-SM04.

9.2. **Stock Standard**

9.2.1. Purchase Primary Calibration stock standards as certified solutions at 1000 mg/L. After opening, transfer the stock standard to an inert vial and store with a minimum headspace.

9.2.2. Purchase a Secondary set of stock standards from a different source to verify the concentration of the first set of standards. Treat the secondary standards similarly as the primary standards.

9.2.3. Purchase LCS/MS and surrogate standards as certified solutions at 1000 mg/L and 200 mg/L, respectively.

9.2.4. Store all standards at ≤ 6°C, unless otherwise specified.

9.3. **Intermediate Standard**

9.3.1. Prepare intermediate standards (for both primary and secondary) at 4000 µg/L as suggested in Table 1.

9.4. **Initial Calibration Standard (ICAL)**

9.4.1. Prepare a minimum of five calibration standards using primary intermediate standard according to Table 2.

9.5. **Initial Calibration Verification (ICV)**

9.5.1. Prepare ICV using the secondary intermediate standard as described in Table 3.

9.6. **Daily Calibration Check Standard (DCC)**

9.6.1. Prepare DCC using the primary intermediate standard as described in Table 3.

9.7. **Surrogate Standard**

9.7.1. Prepare surrogate standard as described in Table 4.

9.8. **LCS/MS Spike Standard**

9.8.1. Prepare LCS/MS spike standard as described in Table 4.

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Aqueous samples shall be prepared as described in EMAX-3520.

10.1.2. Solid samples shall be prepared as described in EMAX-3550 or EMAX-3546. If necessary, clean up with sulfuric acid as described in EMAX-3665 is performed.

10.2. **Instrument Parameters**

10.2.1. EPA 8082A requires an analytical system complete with a temperature programmable gas chromatograph equipped with an autosampler suitable for on column injection of 1 to 5 µL.

10.2.2. Gas Pressure

Nitrogen Pressure : 40 psi

Hydrogen Pressure : 50 psi

10.2.3. Temperature Program

	RESTEK **RTX CLPest 1 & 2**
Initial Temp	130°C, hold for 0.5 minute
Rate 1	25°C/min
Temp 1	230°C, hold for 2 minutes
Rate 2	5°C/min
Temp 2	260°C, hold for 0.2 minute
Rate 3	25°C/min
Final Temp	290°C, hold for 1 minute (PCB) 290°C, hold for 17 mins. (PCB+PCT)
Injector	230°C
Detector	300°C
Injection Volume	1 µL

10.3. **Calibration**

10.3.1. Initial Calibration (ICAL)

10.3.1.1. A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclor 1016 and 1260 at a minimum of five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibration for each of the seven Aroclors.

10.3.1.2. A separate set of calibration standards containing Aroclor 5460 is also prepared.

10.3.1.3. Initial calibration standards (or as suggested in Table 2) of Aroclors 1016, 1260, and 5460 are analyzed by direct injection.

10.3.1.4. Three to five characteristic peaks from each Aroclor are chosen for quantitation. Peaks should be at least 25% of the height of the largest Aroclor peak. Tabulate each peak area response against concentration of injected standard. Calculate each calibration factor according to Eq. 10.6.1.1. A minimum of five sets of calibration factors will be generated for each Aroclor.

10.3.1.5. Application of ICAL Curve for Quantitation

10.3.1.5.1. Generate a summary of calibration factors for each analyte at each concentration using Eq-10.6.1. Calculate the Average Calibration

Factor (ACF), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to, Eq. 10.6.2, Eq. 10.6.3 and Eq. 10.6.4, respectively.

- If RSD is ≤ 20%, ACF may be applied.
- Apply Inverse Weighting Factor (1/y or 1/y2; y being the instrument response) if it is determined to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of ≤ 20% and correlation coefficient of ≥ 0.995.
- Apply linear least squares regression if past experience or priori knowledge of instrument response is known to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of ≤ 20% and correlation coefficient of ≥ 0.995.
- It may be appropriate to force the regression through zero for specific analytes[1]. When exercising this option [as included in the data acquisition software], make sure that the origin (0,0) is not included as a calibration point but rather the intercept is set to zero. This option shall only be applied if the curve favors better accuracy of quantitation.

10.3.1.6. In situations where a particular Aroclor is of interest for a specific project, a minimum five-point calibration curve of that Aroclor may be employed.

10.3.1.7. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

10.3.1.8. Refer to Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.

10.3.2. Initial Calibration Verification (ICV)

10.3.2.1. Prepare a mid-level calibration standard of Aroclors 1260 + 1016 and Aroclor 5460 from a second source. Analyze these after the initial calibration to verify the ICAL.

10.3.2.2. Calculate the %Difference (%D) using Eq. 10.6.2.1. This should be equal to or less than 20%.

10.3.2.3. The standards of the other Aroclors are used to determine a single-point calibration factor for each Aroclor and pattern recognition. It can be analyzed before or after the Aroclor 1016/1260 and 5460 standards.

10.3.3. Daily Calibration Check (DCC)

[1] SW846 Method 8000B, Section 7.5.3; SW846 Method 8000C, Section 11.5.2.1

10.3.3.1. For daily run, analyze DCC (mid-level Aroclor 1016 + 1260 and 5460 standards) at the start of the 12-hour shift prior to sample analysis, and close the analytical run with an ending DCC. A 12-hour shift interval must not exceed 20 samples.

10.3.3.2. The calibration check process does not require analysis of the other Aroclor standards used for pattern recognition.

10.3.3.3. Calculate the %D by using Eq. 10.6.2.1. Refer to Appendix 1 for acceptance criteria.

10.3.3.4. For projects wherein specific PCB is expected to be found, analyze DCC of that particular PCB at 500 μg/L.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. Following the instrument data acquisition software, prepare the analytical sequence file as suggested below:

- Instrument Blank
- Minimum Five Calibration Standards of Aroclors 1016/1260
- Minimum Five Calibration Standards of Aroclor 5460
- Calibration Standards of Aroclor 1254, 1248, 1242, 1232, 1221, 1262, 5432 and 5442 (as necessary) *
- Initial Calibration Verification of Aroclor 1016/1260 and Aroclor 5460
- Method Blank
- Lab Control Sample
- Samples (up to 12 hours but not more than 20 samples)
- Continuing Calibration Standard of of Aroclor 1016/1260 and Aroclor 5460 or other calibrated Aroclors

10.4.2. Sample Analysis

10.4.2.1. Transfer a minimum of 0.5 mL of extract to a 2-mL autosampler vial (or equivalent) using a Pasteur pipette. Cap the vial with a teflon septum and seal with an aluminum rim.

10.4.2.2. Introduce extract into the gas chromatograph using direct injection technique (1 to 5 μl) after all quality control criteria have been met.

10.4.2.3. If the response exceeds the linear range of the system, dilute the sample and re-analyze.

10.4.3. Sample Result Evaluation

* Include Calibration standards for Aroclor 1254, 1248, 1242, 1232, 1221, 1262, 1268, 5432 and 5442 if pattern is found in the sample.

10.4.3.1. Check QC parameters as soon as the data is available.

- ✓ Check LCS recovery that it is within QC limits (Appendix 1).
- ✓ Check MB that is project compliant.
- ✓ Check retention time.
- ✓ Check surrogate recoveries against Appendix 1.
- ✓ Check concentration of target analytes. If the response exceeds the calibration range, dilute and re-analyze the sample until the response falls within the calibration range.
- ✓ If any of the above checkpoints indicate a problem, re-analysis is required. If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate extraction problem, order re-extraction for the affected sample(s).

10.4.3.2. Identification of a multicomponent analyte in the sample is based on pattern recognition in conjunction with the elution of three to five peaks on both GC columns.

10.4.3.3. Positive identification is made when a target peak falls within the retention time window on both columns established by the standard reference compound.

10.4.3.3.1. If one column meets the retention time criteria and a retention time shift is suspected on the other column, use the following guideline in reporting the data:

- ✓ Check that the expanded window does not exceed the RTW of the column in control or the established RTW or the CLP RTW[2] whichever is greater.
- ✓ If the above condition is met, report the data and include a description of the observation in the case narrative.

10.4.3.4. The agreement between the quantitative results should be evaluated after the identification is made. Calculate the relative percent difference (RPD) between the two results according to Eq. - 10.6.4.2.

- If the RPD is less than 40% and the pattern peaks do not indicate any anomalies, report the higher result.
- If the RPD is less than 40% and the pattern peaks indicate an anomaly, report the result from the better pattern peaks.
- If the RPD is greater than 40%, use professional judgment. If no anomaly is found, report the higher result.

10.4.4. Retention Time Window

10.4.4.1. **Establishing RTW**

[2] CLP-OLM4.2 Table 1 D-79/PEST

10.4.4.1.1. Collect at least three Daily Calibration Standards analyzed over a period of 72 hours.

10.4.4.1.2. Calculate the Standard Deviation (SD) of absolute retention time obtained for each of the major peaks used for calibration.

10.4.4.1.3. Determine the width of RTW by ±3X SD.

10.4.4.2. **Evaluating RTW**

10.4.4.2.1. If the SD is equal to 0.00, default to the previous study until historical data is obtained.

10.4.4.2.2. For new instruments, in the interim use the CLP retention time window for Aroclors (±0.07 min) until RTW is obtained for the new instrument parameters condition.

10.4.4.3. **Application of RTW**

10.4.4.3.1. Establish the center of absolute retention time for each of the characteristic peak to include the surrogate(s) from the daily calibration check at the beginning of the analytical shift then apply the established RTW.

10.4.4.3.2. Whenever the observe retention time is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing analyses.

10.4.4.4. **Updating RTW**

10.4.4.4.1. Re-establish the RTW as described in Section 10.4.4.1 when any of the following conditions occur:

- Yearly RTW update
- Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of RTW)
- Major instrument maintenance (e.g. replacements of detector or column; temperature program change, etc.)

10.4.5. Manual Integration

10.4.5.1. Refer to EMAX-DM01 for details of manual integration.

10.4.6. Dealing with Carryover

10.4.6.1. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.

10.4.6.2. If there was no target analyte detected (as found in the sample that exceeded calibration range), proceed with data reduction.

10.4.6.3. If there was a target analyte detected (as found in the sample that exceeded the calibration range), re-analyze the sample to rule-out carry-over. If carry-over is confirmed, proceed with data reduction and report the data from the re-analyzis.

10.5. **Data Reduction**

10.5.1. Make a copy of the analytical run log and highlight the data to be reported.

10.5.2. Collate the reportable raw data. Segregate raw data for samples, QC data and calibration data using section dividers.

10.5.3. Keep all other unused data generated during the analysis in the analytical folder and marke with “For record only”.

10.5.4. Proceed to report generation.

10.6. **Calculations**

10.6.1. Initial Calibration

10.6.1.1. **Calculate for Calibration Factor (CF)**

$$CF = \frac{R_a}{C_a}$$ Eq.-10.6.1.1

where:

R_a - response for analyte measured in peak area

C_a - Concentration of analyte (µgL) x 1/N

N - number of identification peak in each Aroclor

10.6.1.2. **Calculate for Average Calibration Factor (ACF)**

$$ACF = \frac{\sum CF_a}{n}$$ Eq.-10.6.1.2

where:

ACF - average calibration factor

ΣCF_a - sum of calibration factors

n - number of calibration points

10.6.1.3. **Calculate for Standard Deviation**

$$SD = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \bar{x})^2}{N-1}}$$ Eq.-10.6.1.3

where:

SD - standard deviation

x_i - result at I^{th} measurement

$\bar{x}$ - mean of all x measurements

N - number of measurements

10.6.1.4. **Calculate for % Relative Standard Deviation (%RSD)**

$$\%RSD = \frac{SD}{ACF} * 100\% \qquad \text{Eq.-10.6.1.4}$$

where:

RSD - relative standard deviation

SD - standard deviation

ACF - average calibration factor

10.6.1.5. **Calculate for Least Square Line Regression**

$$y = ax + b \qquad \text{Eq.-10.6.1.5}$$

where:

y – Response Factor

x – Concentration

a = x1 = slope of the line

$$a = \frac{\sum \left(x - \bar{x}\right)\left(y - \bar{y}\right)}{\left(x - \bar{x}\right)^2}$$

$\bar{x}$ - Average of amount ratios

$\bar{y}$ - Average of response ratios

b = x0 = intercept of the line

$$b = \bar{y} - a * \bar{x}$$

10.6.1.6. **Calculate for Inverse Weighting Factor**

$$y = ax + b \qquad \text{Eq.-10.6.1.6}$$

where:

y – Response Factor

x – Concentration

a – x1 = slope of the line

$$a = \frac{\sum \left[\left(x - x_a\right)\left(y - y_a\right)\right]}{\sum \left(x - x_a\right)^2}$$

where:

$$x_a = \sum[x(1/x)/\sum(1/x)]$$

$$y_a = \sum[y(1/x)/\sum(1/x)]$$

$$x_a = \sum[x(1/x^2)/\sum(1/x^2)]$$

$$y_a = \sum[y(1/x^2)/\sum(1/x^2)]$$

b = x0 = intercept of the line

$$b = y_a - a * x_a$$

10.6.2. Calibration Check/Continuing Calibration

10.6.2.1. **Calculate Percent Difference**

$$\%D = \frac{ACF - CF}{ACF} * 100\% \qquad \text{Eq.-10.6.2.1}$$

where:

ACF - average calibration factor

CF - calibration factor at calibration check standard

10.6.3. Sample Results

10.6.3.1. Identify 3 to 5 major peaks as quantitative peaks for each Aroclor. Establish the ACF of each peak as outlined in Section 10.6.1.1. Calculate the concentration of each peak in Aroclor according to Eq. - 10.6.3.2 or Eq. - 10.6.3.3. The concentration of Aroclor equals the sum of the concentrations of these major peaks according to Eq, - 10.6.3.4.

10.6.3.2. **Water Samples**

$$C_n = \left(\frac{R_a}{ACF}\right)\left(\frac{V_e}{S_a}\right)DF \qquad \text{Eq-10.6.3.2}$$

where:

C_n - Concentration of characteristic peak n measured (μg/L)

R_a - Total response of analyte in peak area

ACF - Average calibration factor

V_e - Volume of extract in ml

S_a - Sample amount in ml

DF - Dilution factor of sample extract

10.6.3.3. **Soil Samples**

$$C_n = \left(\frac{R_a}{ACF}\right)\left(\frac{V_e}{S_a(\%Solid)}\right)DF \qquad \text{Eq.10.6.3.3}$$

where

C_n - Concentration of characteristic peak n measured (μg/kg)

R_a - Total response of analyte in peak area

ACF - Average calibration factor

V_e - Volume of extract in ml

S_a - Sample Amount in g

% Solid - $\frac{100 - \%\ moisture}{100}$

DF - Dilution factor of the sample extract

10.6.3.4. **Final Aroclor Concentration**

$$C = C_1 + C_2 + \ldots + C_n \qquad \text{Eq.-10.6.3.4}$$

where:

C - Concentration of Aroclor in sample (μg/L or μg/kg)

C_1 - Concentration of characteristic peak 1

C_2 - Concentration of characteristic peak 2

n - Characteristic peak number in each Aroclor

10.6.4. Accuracy and Precision

10.6.4.1. **Percent Recovery**

$$\%R = \frac{C_f - C}{C_s} * 100 \qquad \text{Eq.-10.6.4.1}$$

where:

%R - percent recovery

C_f - concentration of spiked sample

C_s - concentration of spike

C - concentration of unspiked sample (For LCS recovery, C=0)

10.6.4.2. **Relative Percent Difference**

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \qquad \text{Eq.-10.6.4.2}$$

where:

%RPD - Relative Percent Difference

C_1 - Measured concentration of the first sample aliquot

C_2 - Measured concentration of the second sample aliquot

10.7. **Report Generation**

10.7.1. Generate the method.txt file using WDB1C .exe

10.7.2. Generate Lab Chronicle using LABCHRN1.exe

10.7.3. Generate sample results using F1NV3C .exe

10.7.4. Generate the QC Summary file using QCV3CN .exe

10.7.5. Generate the case narrative using CN1.exe.

10.7.6. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analysis.

10.7.6.1. Case Narrative

10.7.6.2. Lab Chronicle

10.7.6.3. Sample Results

10.7.6.4. LCS Summary

10.7.6.5. MS/MSD Summary

10.7.6.6. ICAL Summary

10.7.6.7. ICV Summary

10.7.6.8. DCC Summary

10.7.6.9. Analytical Run Log

10.7.6.10. Sample Preparation Log

10.7.6.11. Non-Conformance Report (if any)

10.8. **Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the project specific requirements (PSR).

10.8.1.1. If any of the checkpoints below indicate a problem, re-analysis is required.

- ✓ Check that all samples required for analysis are analyzed.
- ✓ Check that samples are extracted and analyzed within holding time.
- ✓ Check that all calibration requirements are fulfilled.
- ✓ Check the chromatogram of all positively identified Aroclor patterns that they are qualitatively identified and qualitatively accurate.
- ✓ Check surrogate recoveries against required limits.

- ✓ Check that concentration of target analytes are within calibration range.

10.8.2. Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.3. Submit the analysis package for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Refer to Form 8082FM for daily routine maintenance check points.

10.9.2. Record instrument maintenance performed in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back in control.

10.9.3. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality. The table below is a list of preventive maintenance activities that are essential to consider in performing this SOP.

Maintenance Activity	Description	Frequency
Autosampler Check	Inspect and clean syringe. Check autosampler response.	Daily prior to analysis
Verification	Check instrument parameters to ensure normal operating conditions. Check liner as necessary. Check instrument performance (e.g., daily calibration check, instrument blank, DDT/Endrin breakdown).	Daily prior to analysis
Documentation	Record maintenance in instrument service logs.	Daily prior to analysis
Leak Test	Perform inlet pressure decay test.	Every 6 months or as necessary
System Cleaning	Remove dust from fans and vent covers, inspect and clean inlet and detector where applicable.	Every 6 months or as necessary
Check Flow Path Components	Check and replace the following as necessary: tubing assembly, union, sample probe, and loop.	Once a year or as necessary
Complete Inspection	Perform general inspection of the complete system. Inspect autosampler cabling and configuration setting.	Once a year

11.0 QUALITY CONTROL

11.1. Preparative Batch

11.1.1. A preparative batch shall consist of a method blank, LCS, MS/MSD and a maximum of 20 field samples of similar matrix.

11.1.2. In the absence of MS/MSD, LCS/LCD is prepared.

11.1.3. Surrogate standard shall be added to all samples, including method blank LCS/LCD and MS/MSD. Check PSR for QC Control Limits.

11.1.4. Perform QC check prior to utilizing the surrogate and LCS/MS spike standards by analyzing the prepared standard at the spiking level. Results should be within $\pm$ 20% of the expected value.

11.2. Analytical Batch QC

11.2.1. Instrument Blank must be analyzed daily to establish zero baseline or background value.

11.2.2. A continuing calibration shall be performed before any other analysis is done. The continuing calibration procedure and the acceptance criteria are discussed in Section 10.3.2 and Appendix 1.

11.3. Method QC

11.3.1. Analyst demonstration of proficiency is a must prior to performing this analysis.

11.3.2. A valid LOD and LOQ must exist prior to sample analysis.

11.3.3. A valid ICAL must exist prior to sample analysis.

11.3.4. Instrument performance must be checked prior to sample analysis. Check Appendix 1 for acceptance criteria.

11.3.5. Prepare and analyze QC samples, to include, method blank, LCS (LCD), and MS/MSD. QC Control Limits shall follow the Project Specific Requirement (PSR) in each analytical folder.

12.0 CORRECTIVE ACTION

12.1. Corrective actions associated with this analytical procedure are described in the Summary of Quality Control Procedures in Appendix 1. Document out-of-control event and corrective action in the analytical logbook. If the problem persists, consult the supervisor.

12.2. If Initial calibration is non-compliant, consider the following suggestions to correct the problem:

12.2.1. If %RSD is out of acceptance criteria, review result and identify presence of an outlier.

12.2.2. If one of the standard returns a bias-low or bias-high on all of the analytes, then that point is considered an outlier, prepare a standard at that ICAL point and reanalyze.

12.2.3. If the highest ICAL point appears to be saturated, drop the highest point.

12.2.4. If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.

Note: The lowest calibration point identifies the limit of quantitation (LOQ). Therefore, check that the LOQ is in conformance to the current projects to which the ICAL will be used.

12.2.5. If instrumentation problem is suspected, consider the following suggestions to correct the problem:

12.2.5.1. Check the connections and make sure they are air-tight and perform maintenance as needed.

12.2.5.2. Check the gas flow

12.2.5.3. Prepare a fresh standard and repeat calibration

12.2.6. If the problem persists, inform the supervisor.

12.3. If the ICV is non-compliant, consider the following suggestions to correct the problem:

12.3.1. Re-analyze ICV (to rule out poor injection)

12.3.2. If ICV is still out of acceptance criteria, prepare a fresh standard and re-analyze to rule out any preparation error

12.3.3. If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration

12.3.4. If the problem persists, inform the supervisor

12.4. If the instrument blank is non-compliant, consider the following suggestions to correct problem:

12.4.1. Rule out instrument contamination by performing the instrument daily maintenance, such as changing septum, cleaning liner, cleaning or using new auto sampler syringe.

12.4.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.

12.4.3. Rule out preparation contamination by preparing a new instrument blank

12.4.4. If the problem persists, inform the supervisor.

12.5. If Continuing Calibration is non-compliant, consider the following suggestions to correct the problem:

12.5.1. Change the liner

12.5.2. Clean injection port

12.5.3. Prepare new standard

12.5.4. Cut or replace column

12.5.5. Clean the detector

12.5.6. Rule out leaks by checking all connections

12.5.7. If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL

12.6. If Method Blank is non-compliant, consider the following suggestions to correct the problem:

12.6.1. Rule out instrument contamination by checking instrument blank

12.6.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01

12.6.3. Rule out glassware contamination used for extraction as described in EMAX-QC07

12.6.4. Re-extract MB and the associated samples with reagents free of contamination or with newly

opened reagents

12.6.5. If the problem persists, inform the supervisor

12.7. If LCS is non-compliant, perform the following suggestions to correct the problem:

12.7.1. If result is bias-high or bias-low, check the LCS Standard by analyzing at the spike level

12.7.2. If LCS check is within 80-120% of expected value, check calibration of the micropipette or syringe used for spiking. Re-extract and re-analyze the LCS and the associated samples.

12.7.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS Standard, re-extract and re-analyze LCS and the associated samples.

12.8. Initiate a Non-Conformance Report (NCR) when the following circumstances occur:

- Anomalies other than specified in Appendix 1 is observed.
- Sample is out of technical holding time.

12.8.1. Refer to EMAX-QA08 for NCR details.

12.9. For other problems encountered, inform the supervisor immediately for further instructions.

13.0 POLLUTION PREVENTION

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2. Prepare all standard s in fume hoods.

14.0 WASTE MANAGEMENT

14.1. No samples shall be dumped on the laboratory sink.

14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.

14.3. Place all waste generated during analytical process in properly labeled satellite waste containers for proper collection.

14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. **Definition of Terms**

15.1.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.

15.1.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

15.1.2.1. **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.

15.1.2.2. **Analytical Batch** – is compose of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.3. Detection Limit (DL) – is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

15.1.4. Limit of Detection (LOD) – is defined as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative result rate (Type II error) is 1%.

15.1.5. Limit of Quantitation (LOQ) – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

15.1.6. Safety Data Sheet (SDS) – is written information concerning a chemical physical properties, toxicity, health hazards, fire hazard and reactivity data including storage, spill and handling precautions.

15.1.7. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.8. Calibration Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.9. Characteristic Peaks – are major identifying and quantifying peaks for each type of Aroclor.

15.1.10. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.1.11. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.

15.1.12. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.

15.1.13. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.

15.1.14. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

15.1.15. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.16. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.17. Matrix – is a component or form of a sample.

15.1.18. Matrix Spike (MS) – is a sample spiked with a verified known amount of target anayte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.

15.1.19. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

15.1.20. Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard; used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.

15.1.21. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.

15.1.22. Reagent Soil – organic-free Ottawa sand or equivalent.

15.2. **Application of QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing PCB analysis by GC/MS. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

15.3. **Department of Defense (DoD) and Department of Energy (DoE) Projects**

15.3.1. Samples from DoD and DoE sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, shall be applied.

16.0 REFERENCES

16.1. "Test Methods for Evaluating Solid Waste, Physical and Chemical Methods" (SW846) Method 8082A Rev. 1, Feb. 2007 and Method 8082 Rev. 0, Dec. 1996

16.2. "Test Methods for Evaluating Solid Waste, Physical and Chemical Methods" (SW846) Method 8000B, Rev. 2 Dec. 1996 and Method 8000C Rev. 3, March 2003

16.3. EMAX Quality Systems Manual, as updated

17.0 APPENDICES

17.1. **Figures**

17.1.1.	Figure 1	Peak Evaluation Technique
17.1.2.	Figure 2A	Typical PCB Patterns
17.1.3.	Figure 2B	Typical PCT Patterns
17.1.4.	Figure 3A	Typical 1016/1260 Chromatogram
17.1.5.	Figure 3B	Typical 5460 Chromatogram
17.1.6.	Figure 4	Typical Initial Calibration Summary

17.1.7. Figure 5 Typical Retention Time Window Summary

17.1.8. Figure 6 Typical Sample Result Summary

17.1.9. Figure 7 Typical LCS/LCSD Summary

17.1.10. Figure 8 Typical MS/MSD Summary

17.1.11. Figure 9 Typical Case Narrative

17.2. **Tables**

17.2.1. Table 1 Intermediate Standard Preparation

17.2.2. Table 2 Initial Calibration Standard Preparation

17.2.3. Table 3 Calibration Check Standard Preparation

17.2.4. Table 4 Spike Standard and Surrogate Standard Preparation

17.2.5. Table 5 Compound List

17.2.6. Table 6 Established Limit of Detection (LOD) and Limit of Quantitation (LOQ)

17.3. **Appendices**

17.3.1. Appendix 1 Summary of Quality Control Procedures

17.3.2. Appendix 2 Demonstration of Capability

17.4. **Forms**

17.4.1. 8082FPA Sample Preparation Log

17.4.2. 8082FP Analytical Run Log

17.4.3. 8082FM Instrument Maintenance Log

Figure 1: PEAK EVALUATION TECHNIQUE

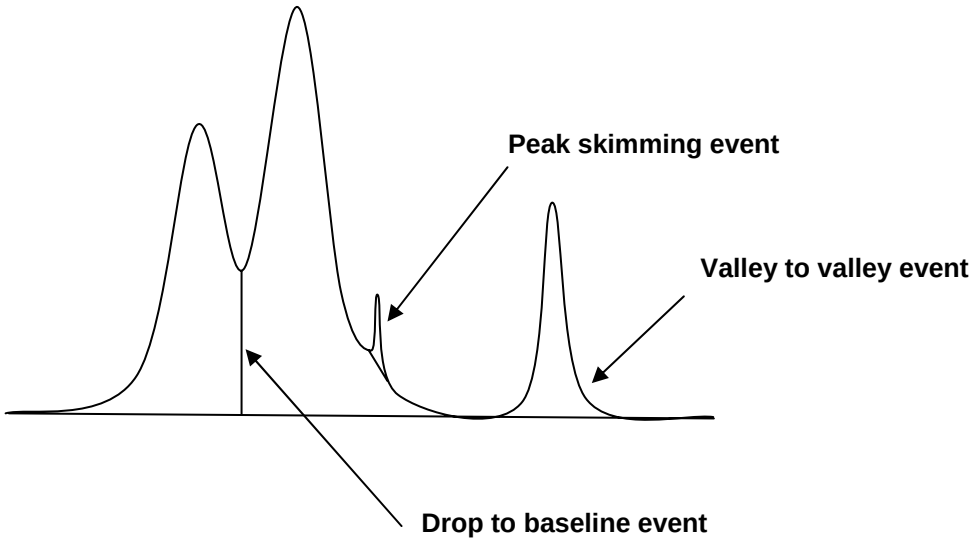


Figure 2A: TYPICAL PCB PATTERNS

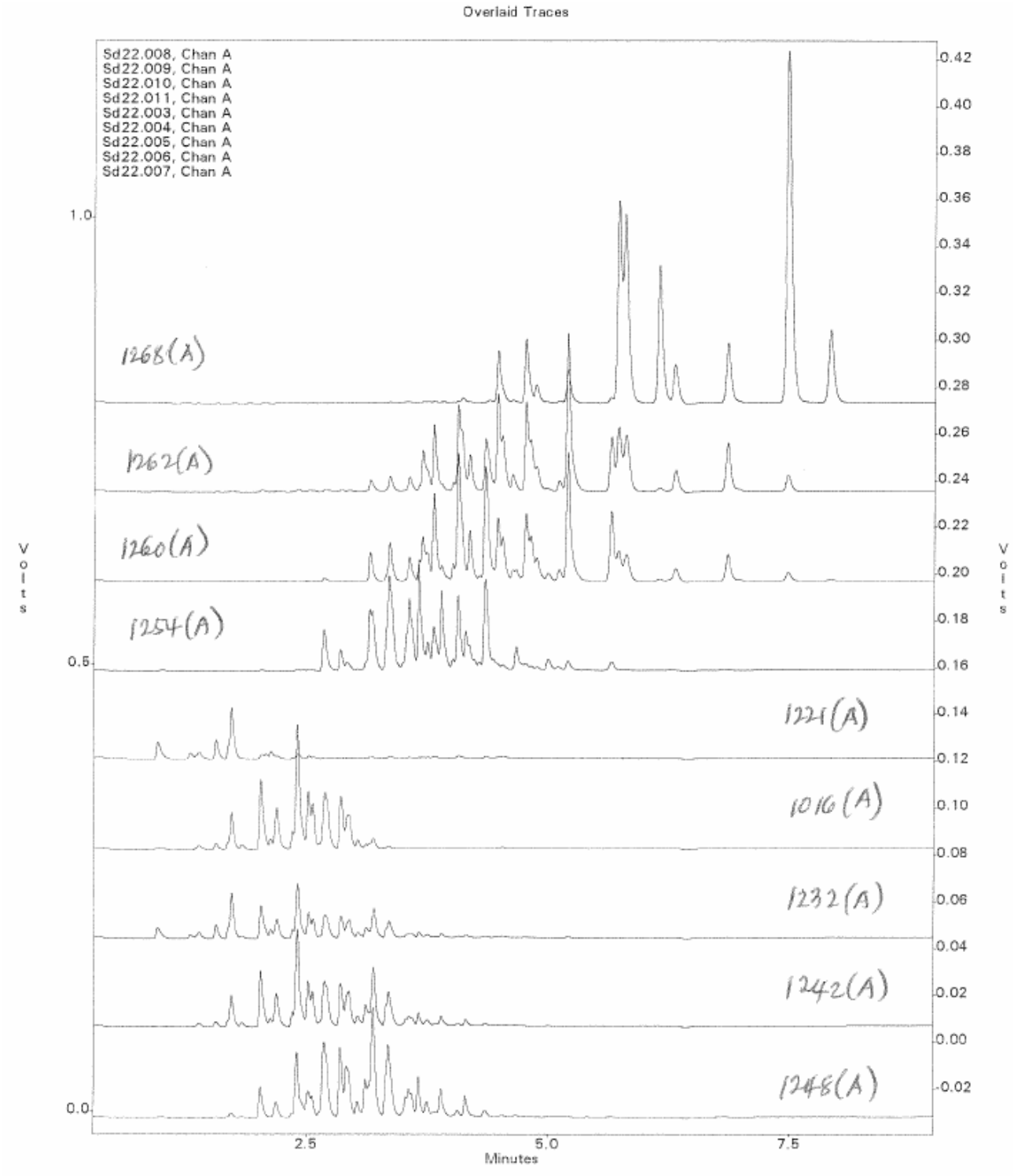


Figure 2A: TYPICAL PCB PATTERNS

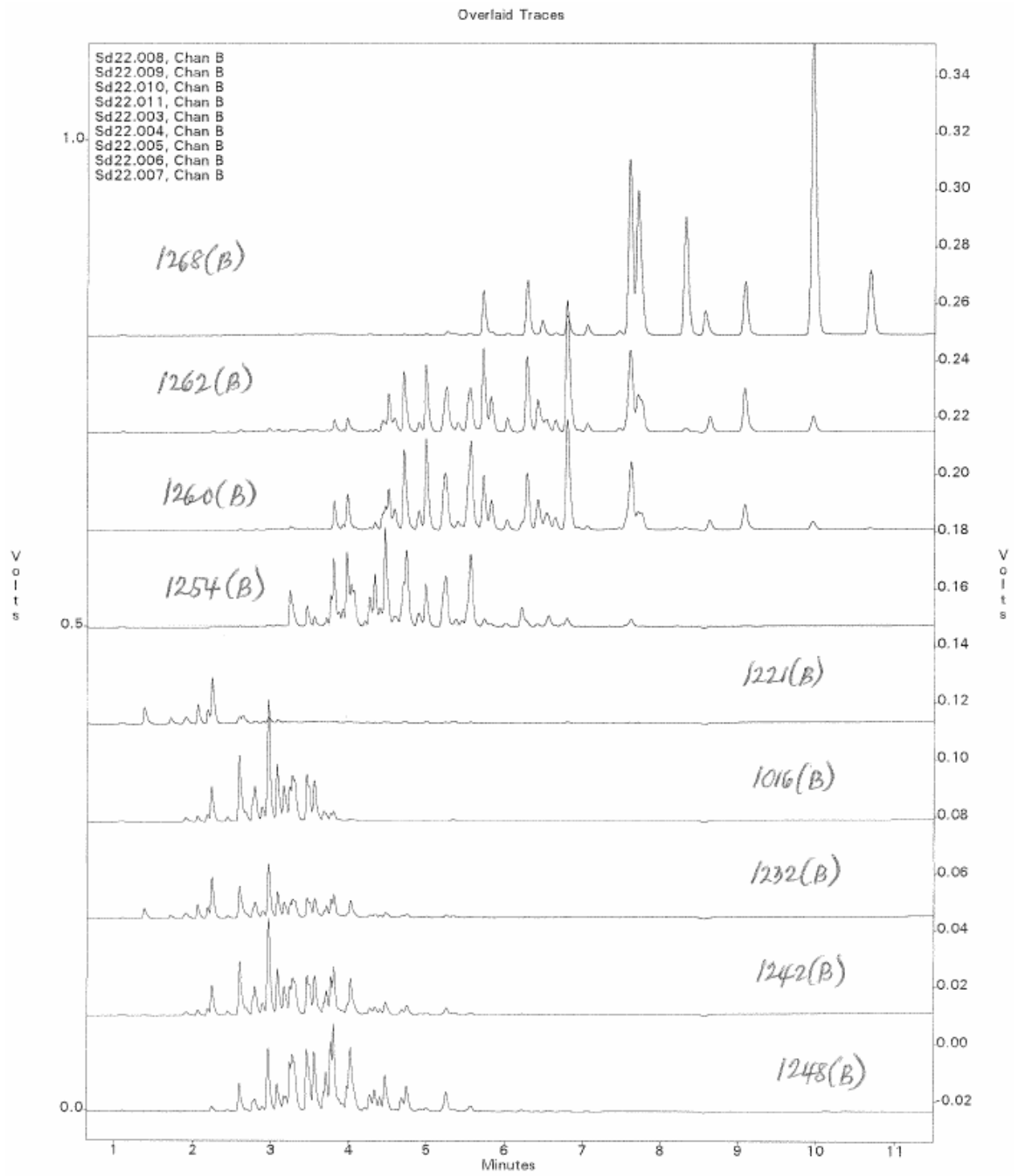


Figure 2B: TYPICAL PCT PATTERNS

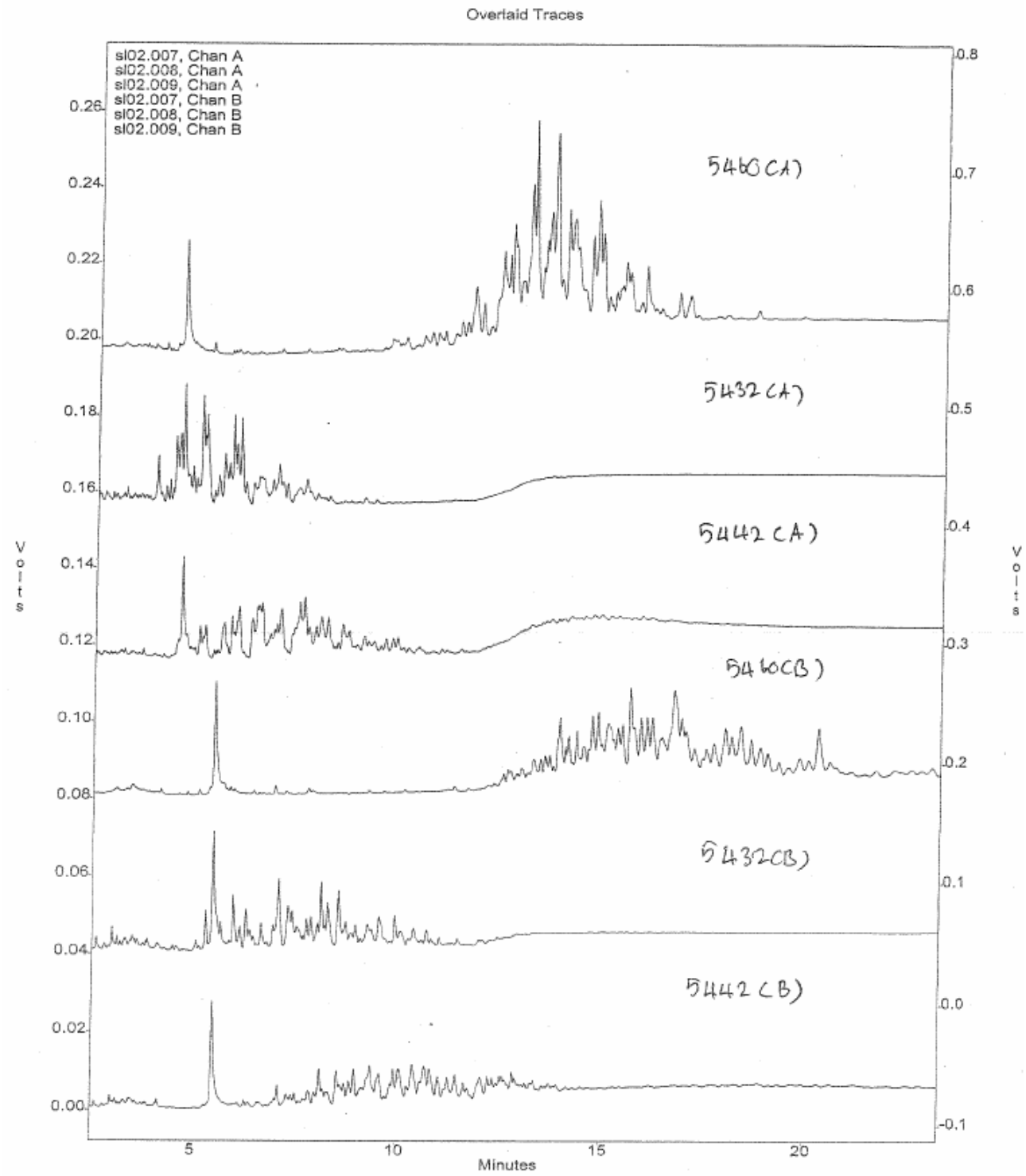


Figure 3A: TYPICAL 1016 / 1260 CHROMATOGRAM

EPA 8082 by GC/ECD
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\sd22\sd22.019
Method : c:\ezchrom\methods\6008d22.met
Sample ID : I6008D2201 500 PPB
Acquired : Apr 22, 2013 16:28:14
Printed : Apr 23, 2013 09:56:16
User : Supakit
Channel A Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc.(ppb)	ICODE
10	TCX	1.397	209505	8027.4	26.10	VV
13	1016-1	1.727	117262	1105.5	106.07	SV
15	1016-2	2.030	208123	2115.1	98.40	VV
17	1016-3	2.193	144013	1406.5	102.39	VV
20	1016-4	2.523	147943	1483.4	99.73	VV
25	1016-5	2.857	158991	1530.6	103.88	VV
37	1260-1	3.817	243941	2385.4	102.26	VV
41	1260-2	4.067	324054	3403.5	95.21	VS
45	1260-3	4.350	381390	4078.6	93.51	VV
50	1260-4	4.773	214219	1885.8	113.60	VV
56	1260-5	5.657	244643	2242.9	109.08	VV
64	DCB	7.923	244745	9822.7	24.92	VV

Channel A Group Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc.(ppb)	ICODE
G1	PCB1016		776332	0.0	510.48	
G2	PCB1260		1408247	0.0	513.66	

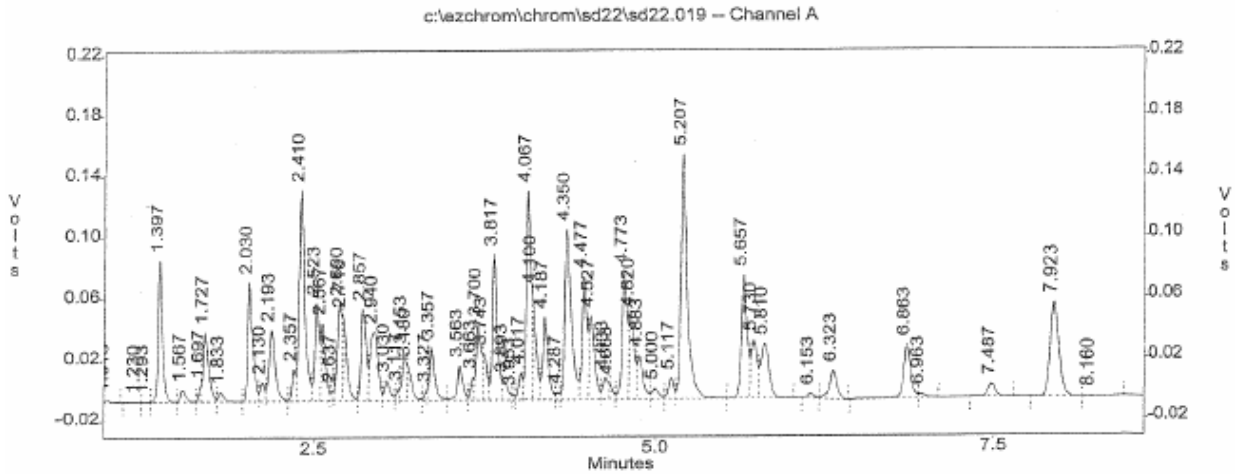


Figure 3A: TYPICAL 1016 / 1260 CHROMATOGRAM

EPA 8082 by GC/ECD
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\sd22\sd22.019
Method : c:\ezchrom\methods\6008d22.met
Sample ID : I6008D2201 500 PPB
Acquired : Apr 22, 2013 16:28:14
Printed : Apr 23, 2013 09:56:16
User : Supakit
Channel B Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc.(ppb)	ICODE
7	TCX	1.790	172868	6723.8	25.71	BV
11	1016-1	2.250	96727	909.1	106.40	SV
17	1016-2	2.977	318468	3162.3	100.71	VV
18	1016-3	3.090	146352	1445.4	101.25	VV
21	1016-4	3.470	96205	884.6	108.76	VS
23	1016-5	3.567	138939	1220.9	113.80	VV
36	1260-1	4.710	242972	2525.2	96.22	VV
38	1260-2	4.993	274421	2771.6	99.01	VV
47	1260-3	6.290	219416	1956.9	112.12	SV
51	1260-4	6.810	452413	3935.2	114.97	VV
55	1260-5	7.627	314588	2861.2	109.95	VV
63	DCB	10.680	213337	8570.9	24.89	BB

Channel B Group Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc.(ppb)	ICODE
G1	PCB1016		796691	0.0	530.92	
G2	PCB1260		1503810	0.0	532.27	

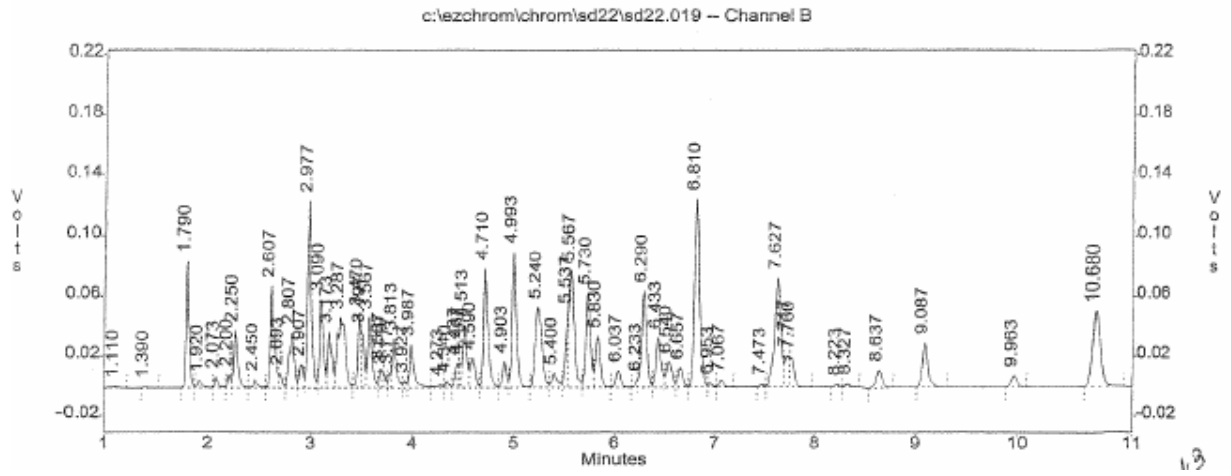


Figure 3B: TYPICAL 5460 CHROMATOGRAM

EPA 8082 by GC/ECD
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\sl06\sl06.006
Method : c:\ezchrom\methods\4608l02.met
Sample ID : I4608L0202
Acquired : Dec 06, 2013 21:01:25
Printed : Dec 09, 2013 09:04:41
User : Supakit
Channel A Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc.(ppb)	ICODE
45	5460-1	11.787	42879	382.9	111.99	vv
57	5460-2	13.240	117349	1162.2	100.98	vv
61	5460-3	13.753	204346	1900.6	107.52	vv
68	5460-4	14.790	76920	737.2	104.34	vv
79	5460-5	15.997	58866	523.6	112.42	vv

Channel A Group Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc.(ppb)	ICODE
G1	PCB5460		500360	0.0	537.24	

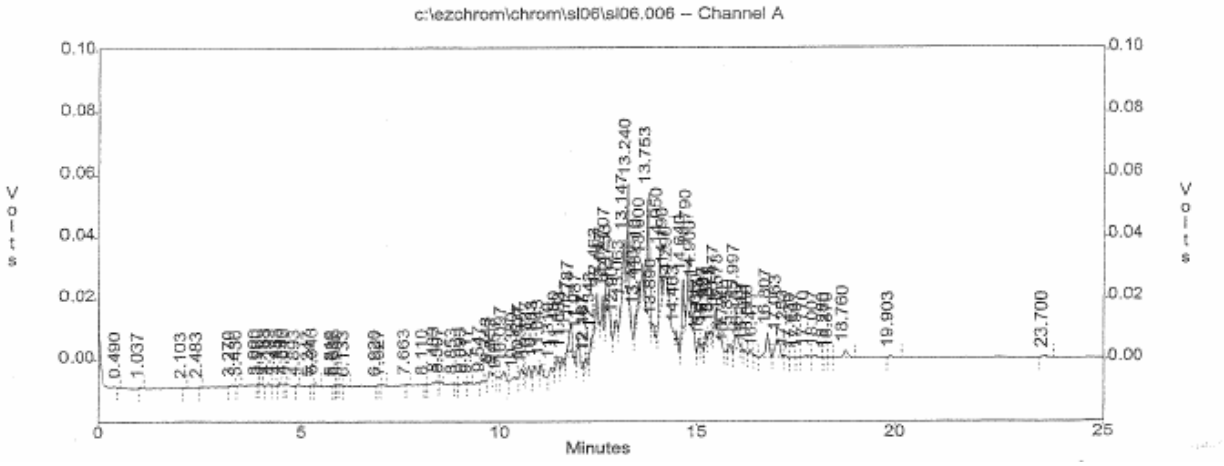


Figure 3B: TYPICAL 5460 CHROMATOGRAM

EPA 8082 by GC/ECD
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\sl06\sl06.006
Method : c:\ezchrom\methods\4608102.met
Sample ID : I4608L0202
Acquired : Dec 06, 2013 21:01:25
Printed : Dec 09, 2013 09:04:42
User : Supakit
Channel B Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc.(ppb)	ICODE
43	5460-1	14.400	30916	272.9	113.27	vv
48	5460-2	14.923	34318	314.8	109.03	vv
57	5460-3	16.143	35980	350.9	102.54	vv
71	5460-4	18.470	62839	598.4	105.01	vv
79	5460-5	20.390	88685	788.4	112.49	vv

Channel B Group Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc.(ppb)	ICODE
G1	PCB5460		252738	0.0	542.35	

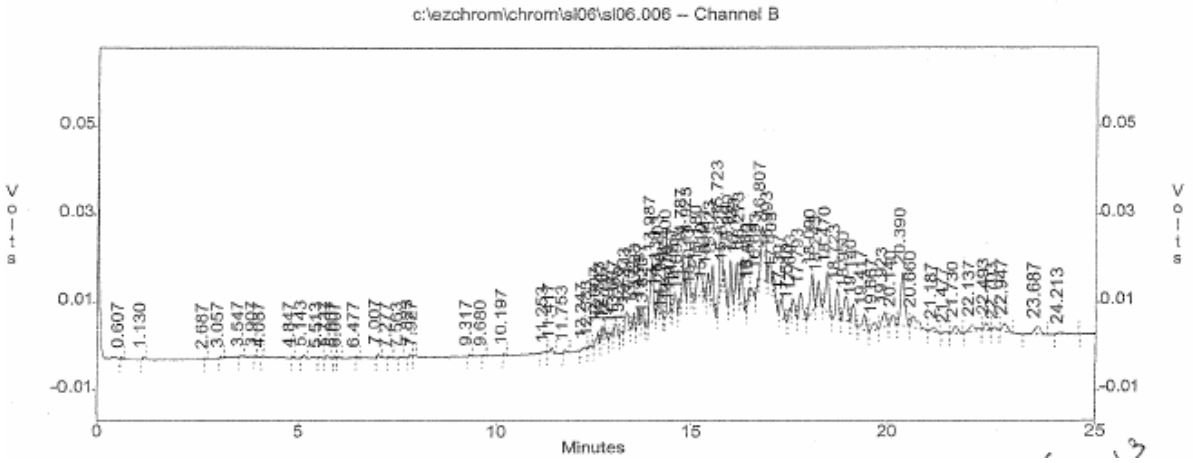


Figure 4: TYPICAL INITIAL CALIBRATION SUMMARY

INITIAL CALIBRATION
METHOD EPA 8082

Lab Name : EMAX Inc
Instrument ID : GCTO08 HP-5890
GC Column : STX-CLPEST
Column size ID : .32MMX30M
LFID & Datetime: SD22012A 04/22/13 14:20
LFID & Datetime: SD22013A 04/22/13 14:38
LFID & Datetime: SD22014A 04/22/13 14:56
LFID & Datetime: SD22015A 04/22/13 15:15
LFID & Datetime: SD22016A 04/22/13 15:33
LFID & Datetime: SD22017A 04/22/13 15:51
LFID & Datetime: SD22018A 04/22/13 16:10
CONC UNIT: PPB

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT 1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X	MEAN	%RSD
PCB1016-1	10.00	1245.80	1264.50	1189.94	1111.22	1010.28	970.48	946.21	1105.489	12.0
PCB1016-2	10.00	2536.40	2564.95	2311.70	2102.99	1854.57	1753.11	1681.63	2115.051	17.3
PCB1016-3	10.00	1425.10	1582.55	1504.94	1450.07	1336.32	1287.07	1259.39	1406.492	8.4
PCB1016-4	10.00	1681.50	1707.10	1566.14	1517.35	1348.08	1288.97	1274.67	1483.401	12.2
PCB1016-5	10.00	1690.30	1689.80	1631.86	1592.19	1427.36	1351.14	1331.22	1530.552	10.2
PCB1260-1	10.00	2497.40	2832.70	2665.04	2481.41	2177.64	2050.89	1993.05	2385.447	13.4
PCB1260-2	10.00	3973.20	3956.45	3780.56	3401.57	3098.52	2868.50	2745.90	3403.528	15.1
PCB1260-3	10.00	4233.80	4253.40	4335.62	4243.01	3950.23	3785.45	3748.53	4078.577	6.0
PCB1260-4	10.00	2101.50	2081.20	2016.72	1916.13	1751.73	1651.76	1681.37	1885.773	10.1
PCB1260-5	10.00	2134.00	2214.35	2293.60	2318.44	2251.94	2258.33	2229.40	2242.865	2.7
SURROGATE	X	1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X	MEAN	%RSD
TCX	2.50	7622.80	7776.60	8055.92	8271.16	8180.68	8129.51	8155.13	8027.399	3.0
DCB	2.50	10453.20	10399.60	10218.48	9930.28	9407.84	9295.45	9054.34	9822.742	5.8

Figure 4: TYPICAL INITIAL CALIBRATION SUMMARY

INITIAL CALIBRATION
METHOD EPA 8082

Lab Name : EMAX Inc
Instrument ID : GCTO08 HP-5890
GC Column : STX-CLPESTII
Column size ID : .32MMX30M
LFID & Datetime: SD22012B 04/22/13 14:20
LFID & Datetime: SD22013B 04/22/13 14:38
LFID & Datetime: SD22014B 04/22/13 14:56
LFID & Datetime: SD22015B 04/22/13 15:15
LFID & Datetime: SD22016B 04/22/13 15:33
LFID & Datetime: SD22017B 04/22/13 15:51
LFID & Datetime: SD22018B 04/22/13 16:10
CONC UNIT: PPB

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT 1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X	MEAN	%RSD
PCB1016-1	10.00	1034.90	1003.55	953.56	920.08	849.59	802.65	799.27	909.085	10.4
PCB1016-2	10.00	3364.00	3348.40	3263.74	3246.16	3052.13	2942.87	2918.57	3162.267	5.9
PCB1016-3	10.00	1584.70	1590.05	1499.06	1500.07	1363.26	1304.63	1275.93	1445.386	9.0
PCB1016-4	10.00	963.20	981.50	1005.26	968.37	838.92	732.73	702.08	884.580	14.3
PCB1016-5	10.00	1367.40	1287.35	1139.18	1278.94	1183.28	1143.45	1146.65	1220.892	7.4
PCB1260-1	10.00	2912.20	2828.30	2682.28	2523.60	2337.13	2210.23	2182.43	2525.167	11.6
PCB1260-2	10.00	3263.30	3140.35	2941.88	2793.55	2541.98	2376.99	2343.39	2771.633	13.2
PCB1260-3	10.00	2107.30	2115.55	2022.06	1964.61	1867.61	1817.69	1803.59	1956.917	6.7
PCB1260-4	10.00	4053.80	4073.20	4062.68	4003.42	3836.75	3766.36	3750.01	3935.174	3.7
PCB1260-5	10.00	2839.40	2874.40	2904.04	2900.47	2847.30	2830.45	2832.44	2861.214	1.1
SURROGATE	X	1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X	MEAN	%RSD
TCX	2.50	6348.80	6425.00	6637.36	6860.32	6902.80	6900.67	6991.45	6723.771	3.8
DCB	2.50	8916.00	8977.60	8898.88	8667.16	8279.26	8188.12	8069.01	8570.861	4.5

Figure 4: TYPICAL INITIAL CALIBRATION SUMMARY

INITIAL CALIBRATION
METHOD EPA 8082

Lab Name : EMAX Inc
Instrument ID : 08 HP-5890
GC Column : STX-CLPESTICIDES
Column size ID : .32MMID X 30MM X 0.32UM DF
LFID & Datetime: SL02010A 12/02/13 22:05
LFID & Datetime: SL02011A 12/02/13 22:40
LFID & Datetime: SL02012A 12/02/13 23:15
LFID & Datetime: SL02013A 12/02/13 23:50
LFID & Datetime: SL02014A 12/03/13 00:25
LFID & Datetime: SL02015A 12/03/13 00:59
LFID & Datetime: SL02016A 12/03/13 01:34
CONC UNIT: PPB

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT 1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X	MEAN	%RSD
PCB5460-1	10.00	345.60	408.85	400.44	387.18	378.08	381.47	378.63	382.893	5.3
PCB5460-2	10.00	1402.30	1398.15	1239.96	1139.28	1034.54	973.24	947.60	1162.152	16.4
PCB5460-3	10.00	2122.80	2141.90	1980.30	1872.49	1786.00	1726.26	1674.43	1900.597	9.8
PCB5460-4	10.00	809.60	825.05	770.08	736.05	694.23	671.68	653.54	737.176	9.1
PCB5460-5	10.00	499.30	538.00	653.86	496.06	496.83	492.63	488.66	523.620	11.4

INITIAL CALIBRATION
METHOD EPA 8082

Lab Name : EMAX Inc
Instrument ID : GCTO08 HP-5890
GC Column : STX-CLPESTICIDES II
Column size ID : .32MMID X 30MM X 0.25UM DF
LFID & Datetime: SL02010B 12/02/13 22:05
LFID & Datetime: SL02011B 12/02/13 22:40
LFID & Datetime: SL02012B 12/02/13 23:15
LFID & Datetime: SL02013B 12/02/13 23:50
LFID & Datetime: SL02014B 12/03/13 00:25
LFID & Datetime: SL02015B 12/03/13 00:59
LFID & Datetime: SL02016B 12/03/13 01:34
CONC UNIT: PPB

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT 1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X	MEAN	%RSD
PCB5460-1	10.00	304.40	303.00	284.96	269.06	256.69	249.62	242.87	272.944	9.2
PCB5460-2	10.00	350.10	358.55	334.00	310.81	293.42	281.35	275.07	314.758	10.6
PCB5460-3	10.00	387.10	393.65	364.36	346.38	331.02	319.98	313.67	350.880	9.1
PCB5460-4	10.00	634.40	657.85	611.10	589.84	573.43	563.00	559.06	598.383	6.3
PCB5460-5	10.00	826.70	839.15	797.92	775.96	766.59	756.85	755.39	788.364	4.3

Figure 5: TYPICAL RETENTION TIME WINDOW SUMMARY

INITIAL CALIBRATION
METHOD EPA 8082

Lab Name : EMAX Inc
Instrument ID : GCT008 HP-5890
GC Column : STX-CLPEST
Column size ID : .32MMX30M
LFID & Datetime: SD22012A 04/22/13 14:20
LFID & Datetime: SD22013A 04/22/13 14:38
LFID & Datetime: SD22014A 04/22/13 14:56
LFID & Datetime: SD22015A 04/22/13 15:15
LFID & Datetime: SD22016A 04/22/13 15:33
LFID & Datetime: SD22017A 04/22/13 15:51
LFID & Datetime: SD22018A 04/22/13 16:10

	RT OF STANDARDS (MIN)							MEAN	RT WINDOW		RTWINDOW
COMPOUND	1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X	RT	FROM	TO	WIDTH
PCB1016-1	1.727	1.727	1.727	1.727	1.723	1.723	1.723	1.725	1.713	1.737	0.012
PCB1016-2	2.033	2.033	2.033	2.030	2.030	2.030	2.030	2.031	2.020	2.042	0.011
PCB1016-3	2.197	2.193	2.193	2.193	2.190	2.193	2.190	2.193	2.182	2.204	0.011
PCB1016-4	2.523	2.520	2.523	2.520	2.520	2.520	2.520	2.521	2.510	2.532	0.011
PCB1016-5	2.857	2.857	2.857	2.857	2.853	2.857	2.853	2.856	2.845	2.867	0.011
PCB1260-1	3.817	3.813	3.817	3.813	3.813	3.813	3.813	3.814	3.798	3.830	0.016
PCB1260-2	4.070	4.067	4.067	4.067	4.063	4.067	4.063	4.066	4.046	4.086	0.020
PCB1260-3	4.350	4.347	4.350	4.347	4.347	4.347	4.347	4.348	4.327	4.369	0.021
PCB1260-4	4.773	4.773	4.773	4.770	4.770	4.773	4.770	4.772	4.746	4.798	0.026
PCB1260-5	5.657	5.657	5.657	5.653	5.657	5.657	5.653	5.656	5.625	5.687	0.031
SURROGATE	1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X	RT	FROM	TO	WIDTH
TCX	1.393	1.393	1.397	1.393	1.393	1.393	1.393	1.394	1.383	1.405	0.011
DCB	7.920	7.920	7.923	7.917	7.920	7.920	7.920	7.920	7.874	7.966	0.046

Figure 5: TYPICAL RETENTION TIME WINDOW SUMMARY

INITIAL CALIBRATION
METHOD EPA 8082

Lab Name : EMAX Inc
Instrument ID : GCT008 HP-5890
GC Column : STX-CLPEST II
Column size ID : .32MMX30M
LFID & Datetime: SD22012B 04/22/13 14:20
LFID & Datetime: SD22013B 04/22/13 14:38
LFID & Datetime: SD22014B 04/22/13 14:56
LFID & Datetime: SD22015B 04/22/13 15:15
LFID & Datetime: SD22016B 04/22/13 15:33
LFID & Datetime: SD22017B 04/22/13 15:51
LFID & Datetime: SD22018B 04/22/13 16:10

COMPOUND	RT OF STANDARDS (MIN) 1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X	MEAN RT	RT WINDOW FROM	TO	RTWINDOW WIDTH
PCB1016-1	2.250	2.250	2.250	2.250	2.250	2.250	2.250	2.250	2.239	2.261	0.011
PCB1016-2	2.977	2.977	2.977	2.977	2.973	2.977	2.977	2.976	2.964	2.988	0.012
PCB1016-3	3.090	3.090	3.093	3.090	3.090	3.090	3.090	3.090	3.078	3.102	0.012
PCB1016-4	3.470	3.467	3.470	3.467	3.467	3.467	3.467	3.468	3.452	3.484	0.016
PCB1016-5	3.567	3.563	3.567	3.567	3.563	3.567	3.563	3.565	3.554	3.576	0.011
PCB1260-1	4.713	4.710	4.713	4.710	4.707	4.710	4.707	4.710	4.684	4.736	0.026
PCB1260-2	4.993	4.993	4.993	4.990	4.990	4.993	4.990	4.992	4.968	5.016	0.024
PCB1260-3	6.287	6.287	6.290	6.287	6.287	6.287	6.287	6.287	6.252	6.322	0.035
PCB1260-4	6.810	6.807	6.810	6.807	6.807	6.810	6.810	6.809	6.770	6.848	0.039
PCB1260-5	7.623	7.623	7.627	7.623	7.623	7.627	7.627	7.625	7.579	7.671	0.046
SURROGATE	1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X	RT	FROM	TO	WIDTH
TCX	1.787	1.790	1.790	1.790	1.787	1.787	1.787	1.788	1.776	1.800	0.012
DCB	10.677	10.677	10.680	10.677	10.673	10.677	10.680	10.677	10.650	10.704	0.027

Figure 5: TYPICAL RETENTION TIME WINDOW SUMMARY

INITIAL CALIBRATION
METHOD EPA 8082

Lab Name : EMAX Inc
Instrument ID : 08 HP-5890
GC Column : STX-CLPESTICIDES
Column size ID : .32MMID X 30MM X 0.32UM DF
LFID & Datetime: SL02010A 12/02/13 22:05
LFID & Datetime: SL02011A 12/02/13 22:40
LFID & Datetime: SL02012A 12/02/13 23:15
LFID & Datetime: SL02013A 12/02/13 23:50
LFID & Datetime: SL02014A 12/03/13 00:25
LFID & Datetime: SL02015A 12/03/13 00:59
LFID & Datetime: SL02016A 12/03/13 01:34

COMPOUND	RT OF STANDARDS (MIN) 1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X	MEAN RT	RT WINDOW FROM	TO	RTWINDOW WIDTH
PCB5460-1	11.757	11.763	11.767	11.773	11.783	11.787	11.793	11.775	11.763	11.787	0.012
PCB5460-2	13.227	13.233	13.233	13.237	13.240	13.247	13.247	13.238	13.231	13.245	0.007
PCB5460-3	13.737	13.743	13.747	13.750	13.753	13.763	13.763	13.751	13.735	13.767	0.016
PCB5460-4	14.767	14.780	14.780	14.787	14.790	14.803	14.803	14.787	14.775	14.799	0.012
PCB5460-5	15.970	15.983	15.990	15.993	16.003	16.017	16.020	15.997	15.986	16.008	0.011

INITIAL CALIBRATION
METHOD EPA 8082

Lab Name : EMAX Inc
Instrument ID : GCT008 HP-5890
GC Column : STX-CLPESTICIDES II
Column size ID : .32MMID X 30MM X 0.25UM DF
LFID & Datetime: SL02010B 12/02/13 22:05
LFID & Datetime: SL02011B 12/02/13 22:40
LFID & Datetime: SL02012B 12/02/13 23:15
LFID & Datetime: SL02013B 12/02/13 23:50
LFID & Datetime: SL02014B 12/03/13 00:25
LFID & Datetime: SL02015B 12/03/13 00:59
LFID & Datetime: SL02016B 12/03/13 01:34

COMPOUND	RT OF STANDARDS (MIN) 1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X	MEAN RT	RT WINDOW FROM	TO	RTWINDOW WIDTH
PCB5460-1	14.380	14.390	14.393	14.393	14.400	14.413	14.413	14.397	14.388	14.406	0.009
PCB5460-2	14.900	14.913	14.917	14.920	14.927	14.940	14.940	14.922	14.911	14.933	0.011
PCB5460-3	16.120	16.133	16.140	16.143	16.153	16.167	16.170	16.147	16.132	16.162	0.015
PCB5460-4	18.443	18.460	18.470	18.470	18.497	18.507	18.513	18.480	18.460	18.500	0.020
PCB5460-5	20.363	20.373	20.393	20.397	20.423	20.433	20.440	20.403	20.366	20.440	0.037

Figure 6: TYPICAL SAMPLE RESULT SUMMARY

METHOD 3546/8082
PCBs

Client : XYZ, INC.	Date Collected: 05/13/14
Project : CLEAN LAND	Date Received: 05/14/14
Batch No. : 14E102	Date Extracted: 05/20/14 13:18
Sample ID: BVBS1265S001	Date Analyzed: 05/22/14 19:37
Lab Samp ID: E102-03	Dilution Factor: 1
Lab File ID: SE22008A	Matrix : SOIL
Ext Btch ID: CPE022S	% Moisture : 3.6
Calib. Ref.: SE22002A	Instrument ID : GCT008

PARAMETERS	RESULTS (ug/kg)	RL (ug/kg)	MDL (ug/kg)
AROCLOR 1016	(ND)|ND	18	8.8|8.8
AROCLOR 1221	(ND)|ND	34	18|18
AROCLOR 1232	(ND)|ND	18	8.8|8.8
AROCLOR 1242	(ND)|ND	18	8.8|8.8
AROCLOR 1248	(ND)|ND	18	8.8|8.8
AROCLOR 1254	(ND)|ND	18	8.8|8.8
AROCLOR 1260	(ND)|ND	18	8.8|8.8
AROCLOR 1262	(ND)|ND	34	18|18
AROCLOR 1268	(ND)|ND	34	18|18
AROCLOR 5432	(ND)|ND	52	26|26
AROCLOR 5442	(ND)|ND	52	26|26
AROCLOR 5460	(ND)|ND	52	26|26

SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
DECACHLOROBIPHENYL	(14.21)|12.68	13.83	(103)|91.7	45-120

Left of | is related to first column ; Right of | related to second column
Final result indicated by ( )
* Out side of QC Limit

Figure 7: **TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA
LCS ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND
BATCH NO.: 14E102
METHOD: METHOD 3546/8082

MATRIX: SOIL % MOISTURE: NA
DILUTION FACTOR: 1 1
SAMPLE ID: MBLK1S
LAB SAMP ID: 60E022SB 60E022SL
LAB FILE ID: SE22005A SE22006A
DATE EXTRACTED: 05/20/1413:18 05/20/1413:18 DATE COLLECTED: NA
DATE ANALYZED: 05/22/1417:52 05/22/1418:27 DATE RECEIVED: 05/20/13
PREP. BATCH: CPE022S CPE022S
CALIB. REF: SE22002A SE22002A

ACCESSION:

PARAMETER	BLNK RSLT (ug/kg)	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	SPIKE AMT (ug/kg)	BSD RSLT (ug/kg)	BS % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Aroclor 1016	(ND)|ND	167	(171)|163	(103)|98	167	(173)|161	(104)|96	(1)|1	50-150	30
Aroclor 1260	(ND)|ND	167	159|(162)	95|(97)	167	158|(165)	95|(99)	1|(2)	50-150	30
Aroclor 5460	(ND)|ND	83.3	(88.3)|84.9	(106)|102	83.3	(90.0)|86.1	(108)|103	(2)|1	50-150	30

SURROGATE PARAMETER	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	QC LIMIT (%)
Decachlorobiphenyl	13.33	16.87|(14.87)	127*|(112)	13.33	16.13|15.01	121*|(113)	45-120

Figure 8: **TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND
BATCH NO.: 14E102
METHOD: METHOD 3546/8082

MATRIX: SOIL % MOISTURE: 3.6

DILUTION FACTOR:	1	1	1		
SAMPLE ID:	BVBS1265S001				
LAB SAMP ID:	E102-03	E102-03M	E102-03S		
LAB FILE ID:	SE22008A	SE22009A	SE22010A		
DATE EXTRACTED:	05/20/1413:18	05/20/1413:18	05/20/1413:18	DATE COLLECTED:	05/13/14
DATE ANALYZED:	05/22/1419:37	05/22/1420:11	05/22/1420:46	DATE RECEIVED:	05/14/14
PREP. BATCH:	CPE022S	CPE022S	CPE022S		
CALIB. REF:	SE22002A	SE22002A	SE22002A		

ACCESSION:

PARAMETER	SMPL RSLT (ug/kg)	SPIKE AMT (ug/kg)	MS RSLT (ug/kg)	MS % REC	SPIKE AMT (ug/kg)	MSD RSLT (ug/kg)	MSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Aroclor 1016	(ND)|ND	173	(155)|176	(90)|102	173	(163)|152	(94)|88	(4)|15	29-135	30
Aroclor 1260	(ND)|ND	173	(165)|165	(95)|95	173	(164)|141	(95)|82	(1)|16	29-135	30
Aroclor 5460	(ND)|ND	86.4	90.1|(93.3)	104|(108)	86.4	(83.9)|79.5	(97)|92	(7)|16	29-135	30

SURROGATE PARAMETER	SPIKE AMT (ug/kg)	MS RSLT (ug/kg)	MS % REC	SPIKE AMT (ug/kg)	MSD RSLT (ug/kg)	MSD % REC	QC LIMIT (%)
Decachlorobiphenyl	13.83	(16.36)|14.64	(118)|106	13.83	(14.44)|12.73	(104)|92.1	45-120

Figure 9: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN LAND

SDG : 14E102

METHOD 3520C/3546/8082
PCBs

A total of six (6) soil samples were received on 05/14/14 for PCBs and PCTs analysis, Method 3546/8082 in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

Holding Time
Samples were analyzed within the prescribed holding time.

Calibration
Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source (ICV). Continuing calibration (CCV) verifications were carried on a frequency specified by the project. All calibration requirements were within acceptance criteria. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank
Method blanks were analyzed at the frequency required by the project. For this SDG, one (1) method blank was analyzed with the samples. Results were compliant to project requirement.

Lab Control Sample
A set of LCS/LCD and lab control sample was analyzed with the samples in this SDG.
Percent recoveries for 60E022SL/C were all within QC limits.

Matrix QC Sample
A set of MS/MSD was analyzed with the samples in this SDG.
Percent recoveries for E102-03M/S were within project QC limits.

Surrogate
Surrogate was added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis
Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter. Sample extracts subjected to appropriate cleanup technique to reduce matrix interference are recorded in extraction log.

Table 1: INTERMEDIATE STANDARD PREPARATION

Standard Name	Stock Standard Conc. (ppm)	Preparation			Final Conc. (µg/L)
		Aliquot (ml)	Solvent	Final Vol. (ml)	
PCB 1016	1000	0.4	Hexane	100	4000
PCB 1260	1000	0.4	Hexane	100	4000
PCB 1221	1000	0.4	Hexane	100	4000
PCB 1232	1000	0.4	Hexane	100	4000
PCB 1242	1000	0.4	Hexane	100	4000
PCB 1248	1000	0.4	Hexane	100	4000
PCB 1254	1000	0.4	Hexane	100	4000
PCB 1262	1000	0.4	Hexane	100	4000
PCB 1268	1000	0.4	Hexane	100	4000
PCT 5460	1000	0.4	Hexane	100	4000
PCT 5432	1000	0.4	Hexane	100	4000
PCT5442	1000	0.4	Hexane	100	4000
Surrogate					
TCX	200	0.1	Hexane	100	200
DCB	200	0.1	Hexane	100	200

Table 2: INITIAL CALIBRATION STANDARD PREPARATION

Standard #	Standard Name	Intermediate Std. (µg/L)	Preparation			Final Conc. (µg/L)
			Aliquot (µl)	Solvent	Final Vol. (µl)	
1	PCB 1660 / PCT 5460	4000	10	Hexane	800	50
	Surrogate	200				2.5
2	PCB 1660 / PCT 5460	4000	20	Hexane	800	100
	Surrogate	200				5
3	PCB 1660 / PCT 5460	4000	50	Hexane	800	250
	Surrogate	200				12.5
4	PCB 1660 / PCT 5460	4000	100	Hexane	800	500
	Surrogate	200				25
5	PCB 1660 / PCT 5460	4000	200	Hexane	800	1000
	Surrogate	200				50
6	PCB 1660 / PCT 5460	4000	300	Hexane	800	1500
	Surrogate	200				75
7	PCB 1660 / PCT 5460	4000	400	Hexane	800	2000
	Surrogate	200				100

Table 3: CALIBRATION CHECK STANDARD PREPARATION

Standard Name	Intermediate Standard Conc. (µg/L)	Preparation			Final Conc. (µg/L)
		Aliquot (µl)	Solvent	Final Vol. (µl)	
PCB 1016	4000	100	Hexane	800	500
PCB 1260	4000	100	Hexane	800	500
PCB 1221	4000	100	Hexane	800	500
PCB 1232	4000	100	Hexane	800	500
PCB 1242	4000	100	Hexane	800	500
PCB 1248	4000	100	Hexane	800	500
PCB 1254	4000	100	Hexane	800	500
PCB 1262	4000	100	Hexane	800	500
PCB 1268	4000	100	Hexane	800	500
PCT 5460	4000	100	Hexane	800	500
PCT 5432	4000	100	Hexane	800	500
PCT 5442	4000	100	Hexane	800	500
Surrogate					
TCX	200	100	Hexane	800	25
DCB	200	100	Hexane	800	25

Table 4: SPIKE STANDARD AND SURROGATE STANDARD PREPARATION

Standard Name	Stock Standard Conc. (mg/L)	Preparation			Final Conc. (µg/L)
		Aliquot (ml)	Solvent	Final Vol. (ml)	
PCB 1016	1000	1.0	50% Isopropanol 50% Hexane	100	10,000
PCB 1260	1000	1.0	50% Isopropanol 50% Hexane		
PCT 5460	1000	1.0	50% Isopropanol 50% Hexane	100	10,000
Surrogate					
TCX	200	2	50% Isopropanol 50% Hexane	1000	400
DCB	200	2	50% Isopropanol 50% Hexane	1000	400

Table 5: COMPOUND LIST

Analytes		Surrogates
PCB 1016	PCT 5432	Decachlorobiphenyl
PCB 1221	PCT 5442	Tetrachloro-m-xylene
PCB 1232	PCT 5460	
PCB 1242		
PCB 1248		
PCB 1254		
PCB 1260		
PCB 1262		
PCB 1268		

Table 6: ESTABLISHED LIMIT OF DETECTION (LOD) AND LIMIT OF QUANTITATION (LOQ)

ANALYTES	AQUEOUS (µg/L)			SOIL (µg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ
PCB 1016	0.45	0.45	0.45	13.2	16.7	33.3
PCB 1221	0.29	0.29	0.29	8.3	16.7	33.3
PCB 1232	0.25	0.25	0.25	9.0	16.7	33.3
PCB 1242	0.25	0.25	0.25	9.3	16.7	33.3
PCB 1248	0.25	0.25	0.25	8.3	16.7	33.3
PCB 1254	0.25	0.25	0.25	8.3	16.7	33.3
PCB 1260	0.31	0.31	0.31	9.9	16.7	33.3
PCB 1262	0.25	0.25	0.25	8.3	16.7	33.3
PCB 1268	0.25	0.25	0.25	8.3	16.7	33.3
PCT 5432	0.5	0.5	0.5	10	20	40
PCT 5442	0.5	0.5	0.5	10	20	40
PCT 5460	0.5	0.5	0.5	10	20	40
Tetrachloro-m-xylene	0.020	0.020	0.020	0.42	0.83	1.67
Decachlorobiphenyl	0.025	0.025	0.025	0.42	0.83	1.67

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
Min. 5 Point Initial Calibration for PCB 1016 + 1260 and PCT 5460	Initially, as needed	RSD for all analytes ≤ 20%. Linear – least squares regression r ≥ 0.995.	Correct then problem then repeat initial calibration.		
Second-source Calibration Verification for PCB 1016/1260 and PCT 5460	Once per 5-point initial calibration	All analytes within ± 20% of expected value from the ICAL.	Correct the problem. If problem persists, repeat initial calibration.		
Initial Calibration Verification Check for PCB 1016/1260 and PCT 5460	Daily, before sample analysis	All analytes within ± 20%	Correct the problem. If problem persists, repeat initial calibration		
Calibration verification for PCB 1016/1260 and PCT 5460	After every 12 hours and at the end of the analytical sequence	All analytes within ± 20% of expected value	Correct the problem then repeat initial calibration verification and re-analyze all samples since last successful calibration verification.		
Method Blank	One per preparation batch (≤ 20 samples per matrix)	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Re-prep and re-analyze method blank and all samples processed with the contaminated blank		
LCS (PCB 1016/1260 and PCT 5460)	One LCS per analytical batch (≤20 samples per matrix)	Within project QC Limits	Correct the problem, then re-prep and re-analyze the LCS and all associated samples.		
Surrogate Spike	Every sample, spiked sample, standard, and method blank	Within project QC Limits	Correct the problem then re-extract and re-analyze sample		
Matrix Spike/ Matrix Spike Duplicate (PCB 1016/1260 and PCT 5460	One MS/MSD per 20 project samples per matrix	Within project QC Limits	If chromatogram is indicative of matrix interference, discuss in case narrative. Otherwise, check for probable source of error and perform corrective action as necessary.		
Confirmation	100% for all positive results	Same as primary column	If quantitation criteria are not met, use confirmation for qualitative identification only.		
Comments: 1. For flagging criteria refer to PSR. Otherwise, if MB is non-compliant, apply "B" to specific analyte(s) on all associated samples, apply "J" to all values between LOD and LOQ.				Reviewed By: Date:	

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
PCBs
METHOD: EPA 8082/ 8082A

MATRIX: WATER

Analytical SOP: EMAX-8082 Rev. 4
Sample Preparation SOP: EMAX-3520 Rev. 5
Conc Unit: µg/L
Sample Amount(mL): 1000
Extract Volume (mL): 10

Instrument ID: 08
Extraction batches: CPE002W - 5/5/14
CPE009W - 5/7/14
Extracted by: J. Muertigue
Analysis date: 5/7/14 & 5/14/14
Analyzed by: R. Zhou

PARAMETER	60E002WL	60E002WC	60E009WL	60E009WC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SE07006A	SE07007A	SE12012A	SE12013A							
PCB 1016	5.11	4.91	5.44	4.66	5	5.03	101	0.3	7	60 - 130	PASSED
PCB 1260	4.65	4.44	5.03	4.46	5	4.65	93	0.3	6	70 - 130	PASSED
PCT 5460	2.34	2.45	2.42	2.41	2.5	2.41	96	0.05	2	50 - 150	PASSED
TCX	0.396	0.385	0.399	0.391	0.4	0.393	98	0.006	2	60 - 130	PASSED
DCB	0.406	0.402	0.433	0.432	0.4	0.418	105	0.016	4	70 - 130	PASSED

PARAMETER	60E002WL	60E002WC	60E009WL	60E009WC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SE07006B	SE07007B	SE12012B	SE12013B							
PCB 1016	5.02	4.83	4.86	4.69	5	4.85	97	0.1	3	60 - 130	PASSED
PCB 1260	4.70	4.68	4.86	4.76	5	4.75	95	0.1	2	70 - 130	PASSED
PCT 5460	2.17	2.28	2.16	2.14	2.5	2.19	88	0.1	3	50 - 150	PASSED
TCX	0.409	0.399	0.405	0.407	0.4	0.405	101	0.004	1	60 - 130	PASSED
DCB	0.372	0.367	0.389	0.384	0.4	0.378	95	0.010	3	70 - 130	PASSED

MATRIX: SOIL

Analytical SOP: EMAX-8082 Rev. 4
Sample Preparation SOP: EMAX-3550 Rev. 4
Conc Unit: µg/Kg
Sample Amount(g): 30
Extract Volume (mL): 10

Instrument ID: 08
Extraction batches: CPF006S - 6/5/14
CPF018S - 6/10/14
Extracted by: J. Villena
Analysis date: 6/6/14 & 6/10/14
Analyzed by: R. Zhou

PARAMETER	60F006SL	60F006SC	60F018SL	60F018SC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SF06006A	SF06007A	SF10006A	SF10007A							
PCB 1016	181	171	163	163	167	169.56	102	8.5	5	70 - 130	PASSED
PCB 1260	159	154	156	155	167	155.63	93	2.2	1	70 - 140	PASSED
PCT 5460	76.5	77.4	84.3	82.9	83.3	80.29	96	3.9	5	50 - 150	PASSED
TCX	14.6	13.6	14.3	14.4	13.3	14.251	107	0.421	3	50 - 140	PASSED
DCB	14.2	14.1	14.9	14.8	13.3	14.508	109	0.446	3	70 - 140	PASSED

PARAMETER	60F006SL	60F006SC	60F018SL	60F018SC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SF06006B	SF06007B	SF10006B	SF10007B							
PCB 1016	190	184	164	170	167	176.93	106	12.0	7	70 - 130	PASSED
PCB 1260	161	159	168	168	167	164.13	98	4.7	3	70 - 140	PASSED
PCT 5460	71.8	73.2	77.5	76.8	83.3	74.84	90	2.8	4	50 - 150	PASSED
TCX	16.2	15.5	15.7	15.7	13.3	15.756	118	0.305	2	50 - 140	PASSED
DCB	12.2	12.1	13.4	13.2	13.3	12.733	95	0.657	5	70 - 140	PASSED

8082FS: SAMPLE PREPARATION LOG

EMAX LABORATORIES, INC.

FileID: CP.xls

EXTRACTION LOG FOR PESTICIDES/PCBs

PrepBatchID	LabSampleID	Aliquot	Unit	DateTime	Ve	EA	EVe	PF	FlorisilCleanup EMAX-3620	SulfurCleanup EMAX-3660	[1]AcidCleanup EMAX-3665	Comments

Ve=ExtractVolume EA= ExpectedAliquot EVe=ExpectedExtractVolume PF=(EA/Aliquot)(Ve/EVe)

☐ ExtractionStarted @

☐ Extraction Ended @

Prepared By:

Comment: [1]Acid cleanup is only for PCB anlaysis.

Checked By:

Date

8082FA: **ANALYTICAL RUN LOG**

EMAX LABORATORIES, INC.

ANALYSIS RUN LOG
for
PESTICIDES/PCBs

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Comments:

Book #: A71-032

Instrument No.: 71

Analytical Sequence:

Method File:

Analytical Batch:

SOP #	Rev. #
☐ EMAX-8081	7
☐ EMAX-8082	3
☐ EMAX-608	1
☐ EMAX-CLP-PEST	
☐ EMAX-	

ICAL REFERENCE

ICAL ID	BATCH	DATE

STANDARDS ID	Conc (µg/L)
Hexane	

Column	A. ZB-MULTI RES I B. ZB-MULTI RES I I

ELECTRONIC DATA ARCHIVAL

Location	Date
☐ EZC_4_Pesticides	
☐ EZC_10_Pesticides	

Analyzed By: Date:

Date Disposed: Disposed By:

This page is checked during data review.

8082FM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: 71 Month: Year: 201_ Book #: M71-004

Date							
Column Head Pressure (8-15psi)							
Column Temperature							
Signal # 1 10-150							
Signal #2 10-150							
Autosampler							
Update Cal. Table							
Computer Date/Time							
Computer Paper							
Septa Change							
Liner Clean-up							
Analyst Initial							
TroubleShooting							
Problems Encountered							
Return to Control (Analyst Initial)							

Code	Problems Encountered	Code	Corrective Action

EMAX
LABORATORIES, INC.
1835 W. 205th Street
Torrance, CA 90501
Tel: (310) 618-8889
Fax: (310) 618-0818

ERRATUM TO	
Document	EMAX-8260
Revision Number	10
Section	5.1.3 & 5.1.4
Date	May 11, 2017
Reference Number	8260.10.1

Section 5.1.3 and 5.1.4 shall be revised as follows:

5.1.3. Samples preserved with HCl to pH<2 must be analyzed within 14 days from the date of sampling. Samples that are not preserved with HCl or with pH≥2 must be analyzed within 7 days from the date of sampling.

5.1.4. If Acrolein and Acrylonitrile are target analytes, samples must be analyzed within 14 days if preserved with $Na_2S_2O_3$ to pH 4-5. Otherwise, analyze within 3 days from sampling date.

PREPARED BY: _____ Date: 05-11-17

Name Tu Nisamaneepong

Title Operations Manager

APPROVED BY: _____ Date: 05-11-17

Name Kenette Pimentel

Title QA Manager

APPROVED BY: _____ Date: 05-16-17

Name Caspar Pang

Title Laboratory Director

EMAX
LABORATORIES, INC.
1835 W. 205th Street
Torrance, CA 90501
Tel: (310) 618-8889

Page 1 of 1

ADDENDUM TO	
Document	ALL ANALYTICAL SOPS
Revision Number	CURRENT REVISIONS
Section	10.2 Instrument Parameters
Date	11 September 2019
Reference Number	AA.7

This applies to all GC/MS, GC, HPLC-MS, HPLC, ICP-MS, ICP and IC Method SOPs

Section 10.2 shall include:

10.2. **Instrument Parameters**

10.2.1. Instrument parameters setup stipulated in the SOPs, are instrument suggested parameters. Fine tune the instrument to obtain optimum instrument condition.

10.2.2. Print and staple a copy of the current instrument parameter on the instrument log for easy access when performing daily instrument routine check.

10.2.3. In the event that instrument parameters necessitate a change, replace the instrument parameter printout with the new parameter setup. Archive the previous instrument parameters at the back of the instrument maintenance log.

PREPARED BY: _____ Date: 9/11/19

Name Farina Madamba

Title QA / QC Coordinator

APPROVED BY: _____ Date: 09.11.19

Name Kenette Pimentel

Title QA Manager

APPROVED BY: _____ Date: 09-11-19

Name Caspar Pang

Title Laboratory Director

STANDARD OPERATING PROCEDURES

VOLATILE ORGANICS BY GC/MS

SOP No.:	EMAX-8260	Revision No.	10	Effective Date:	06-Jun-14
Prepared By:	Souzan Greas			Date:	06-04-14
Approved By:	Kenette Pimentel QA Manager			Date:	06.04.14
Approved By:	Caspar Pang Laboratory Director			Date:	06-04-14

Control Number: 8260-10-

1.0 SCOPE AND APPLICATION

1.1. This analytical method is used to determine the concentration of volatile organic compounds whose boiling points are below 200°C and are water insoluble or slightly water-soluble found in solid or liquid samples. The list of compounds is summarized in Tables 6 and 7. Additional analytes may be added after verification.

1.2. This SOP is an adaptation of SW846 Method 8260B.

2.0 SUMMARY OF METHOD

2.1. A measured sample is extracted using a purge and trap concentrator system. The extract is introduced to a temperature-programmed GC. The analytes are eluted through the GC column separating each analyte relative to its volatility. These analytes are captured and ionized by the mass spectrometer. The ionized fragments are measured by mass to charge ratio. Analyte qualitative identification is based on the characteristic electron impact mass spectra. Analyte quantitative identification is based on the response of the major ion relative to an internal standard using a multi-point calibration curve.

2.2. **Interferences**

2.2.1. Contamination may occur by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through sample container septum during shipment and storage. Trip blanks and storage blanks can serve as means of monitoring.

2.2.2. Glassware and other sample processing materials in which the samples come into contact with are possible sources of contamination. All glassware and other materials used must be purchased pre-cleaned or decontaminated prior to use.

2.2.3. Solvents and reagents are possible sources of contamination. All solvents and reagents must be GC grade and must pass the QC checks prior to use.

2.2.4. Contamination by carry-over can occur whenever high concentration samples are analyzed in sequence with a low concentration sample. To reduce potential carry-over, the concentrator must be thoroughly baked-out between samples and the sample syringe and purging device must be thoroughly rinsed with an appropriate solvent between samples.

2.2.5. Another possible source of contamination is the analytical instrument itself. This can be monitored by analyzing an instrument blank prior to any analysis.

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.

3.1.2. Refer to Table 6 and Table 7 for established DL, LOD and LOQ levels.

4.0 DYNAMIC RANGE

4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point (see Sec. 9.4). All samples analyzed above this concentration are considered "over-range" and requires dilution to properly quantitate.

4.2. The concentration in the diluted sample should be at or above the project reporting limit. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required to properly quantitate.

4.3. **Typical Dynamic Range**

4.3.1. Water: 5 µg/L to 200 µg/L (5 ml purge)

1 µg/L to 40 µg/L (25 ml purge)

4.3.2. Soil: 5 µg/kg to 200 µg/kg

5.0 SAMPLE HOLDING TIME & PRESERVATION

5.1. **Aqueous Samples**

5.1.1. Samples received in the laboratory are expected to be contained in 40 ml vials with teflon lined septa with zero headspace.

***Note: The size of any bubble caused by degassing upon cooling the sample must not exceed 6 mm.*[1]**

5.1.2. Samples must be stored at ≤ 6°C without freezing.

5.1.3. Samples preserved in HCL must be analyzed within 14 days from the date of sampling. Samples with no chemical preservative must be analyzed within 7 days from the date of sampling.

5.1.4. If Acrolein and Acrylonitrile are target analytes, samples must be analyzed within 14 days if preserved with $Na_2S_2O_3$ to pH 4-5. Samples received unpreserved must be analyzed with 3 days from sampling date[2].

5.2. **Soil Samples**

5.2.1. Samples received in glass jars or brass tubes must be stored at ≤ 6°C without freezing. Samples for low level and extracted in methanol for high level must be analyzed within 14 days from sampling date.

5.2.2. Samples received in encore tubes may be frozen, preserved with sodium bisulfate or extracted with methanol prior to analysis.

[1] Referenced from SW846 Method 5030B, Section 6.1.
[2] Reference: 40CFR Table 11 Footnote 10

- Encore samples to be frozen must be analyzed within 14 days from sampling date.
- Encore samples to be preserved with sodium bisulfate for low level and extracted with methanol for high level must be done within 48 hours and analyzed within 14 days from sampling date.
- Preserved samples and extracts must be stored at ≤ 6°C without freezing.

6.0 ASSOCIATED SOPs

6.1. EMAX-5030 Purge and Trap

6.2. EMAX-5035 Closed-System Purge and Trap

6.3. EMAX-DM01 Data Flow and Review

6.4. EMAX-QA04 Detection Limit (DL)

6.5. EMAX-QA05 Training

6.6. EMAX-QA08 Corrective Action

6.7. EMAX-QC01 Quality Control for Chemicals

6.8. EMAX-QC02 Analytical Standard Preparation

6.9. EMAX-QC07 Glassware Cleaning

6.10. EMAX-SM01 Sample Management

6.11. EMAX-SM03 Waste Disposal

6.12. EMAX-SM04 Analytical and QC Sample Labeling

7.0 SAFETY

7.1. Read all SDS of chemicals listed in this SOP.

7.2. Treat all reagents, standards, and samples as potential hazards. Observe standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves at all times when performing this procedure. Perform all sample and standard handling in the fume hood.

7.3. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of the body, rinse the affected body part thoroughly with copious amounts of water. If irritations or any other discomfort related to the incident persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS & REAGENTS

8.1. **Instruments and Supplies**

Gas Chromatography	HP 5890 Series II or equivalent
Detector	HP 5971 MSD or equivalent

Column	RTX 502.2 (0.32 mm x 60 m), 1.8 µm thickness or equivalent after verification that the four gases (chloromethane, bromomethene, chloroethane, and vinyl chloride) can be resolved > 90% from each other in the total ion chromatogram
Data Acquisition Software	ChemStation or equivalent
Purge & Trap Device	OI 4560/Encon Evolution/EST or equivalent
Multiple purging module	Archon/Centurion or equivalent
Gases	Ultra-high purity helium/Air
Syringes	5 ml, 25 ml Luerlok gas-tight
Microsyringes	1, 10, 20, 25, 50, 100 and 1000 µl (Hamilton 702N or equivalent)
Volumetric Flasks	2, 5, 10, 50 and 100 ml with ground glass stopper
Heated Sparge	Archon or Automatic sample heating jacket or equivalent

8.2. **Chemicals and Reagents**

Extraction Solvent	Purge & Trap Grade Methanol or equivalent
Reagent Water	Organic-free water
Reagent Soil	Organic-free Ottawa Sand or equivalent
Preservative	Sodium Bisulfate

9.0 STANDARDS

9.1. Standard preparation for VOA is summarized in Tables 1 to 4. Refer to EMAX-QC02 for proper analytical standard preparation and EMAX-SM04 for proper labeling. Other concentration levels may be prepared as long as it complies with the method and/or project requirements.

9.2. **Stock Standard**

9.2.1. Purchase Stock Standards as certified solutions.

9.2.2. Purchase one set of calibration standard (refer to Table 1) for calibration and a secondary source Stock Standard for calibration verification (refer to Table 2).

9.2.3. Purchase Surrogate Mix at 2500 mg/L and Internal Standard at 2500 mg/L (refer to Table 3).

9.2.4. Purchase BromoFluorobenzene (BFB) as Tuning Standard at 5000 mg/L (refer to Table 4).

9.2.5. After opening, transfer in inert vials with minimal headspace and store at -10°C to -20°C.

9.3. **Intermediate Standards**

9.3.1. Using the stock standard solutions, prepare intermediate standards in methanol according to Tables 1 to 4 and store with minimal headspace in an inert vial.

9.4. **Initial Calibration Standards (ICAL)**

9.4.1. ICAL for 5 ml Purge

9.4.1.1. Using intermediate standards (refer to Tables 1 and 3), prepare multi calibration standards (minimum of five different concentrations) in reagent water as suggested below.

Calibration Pt.	VOA (µg/L)*	Surrogate (µg/L)	Internal Std (µg/L)
1	5	5	50
2	10	10	50
3	20	20	50
4	50	50	50
5	100	100	50
6	200	200	50

** Ketones, Acrolein, Acrylonitrile and tert-Butanol are 5X the indicated concentration and m/p-Xylene is 2x the indicated concentration.*

9.4.2. ICAL for 25 ml Purge

9.4.2.1. Using intermediate standards (refer to Tables 1 and 3), prepare multi calibration standards (minimum of five different concentrations) in reagent water as suggested below:

Calibration Pt.	VOA (µg/L)*	Surrogate (µg/L)	Internal Std (µg/L)
1	0.5	0.5	10
2	1	1	10
3	2	2	10
4	10	10	10
5	20	20	10
6	40	40	10

** Ketones, Acrolein, Acrylonitrile and tert-Butanol are 5X the indicated concentration and m/p-Xylene is 2x the indicated concentration.*

9.5. **Initial Calibration Verification Standard (ICV)**

9.5.1. Using the Intermediate Standard prepared from the secondary source (refer to Tables 2 and 3), spike into 5 ml or 25 ml purge in reagent water as suggested below.

9.5.1.1. ICV for 5 ml purge

ICV	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
5 ml	50	50	50

** Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

9.5.1.2. ICV for 25 ml purge

ICV	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
25 ml	10	10	10

** Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

9.6. **Daily Calibration Check Standard (DCC)**

9.6.1. Using the Intermediate Standard prepared from the same source as the ICAL Standard (refer to Tables 1 and 3), spike into 5 ml or 25 ml purge in reagent water as suggested below.

9.6.1.1. DCC for 5 ml purge

DCC	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
5 ml	50	50	50

** Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

9.6.1.2. DCC for 25 ml purge

DCC	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
25	10	10	10

** Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

9.7. **LCS and Matrix Spike Standard**

9.7.1. For spike standards, use the Intermediate Standard prepared from the secondary source (refer to Tables 2 and 3), spike into the 5 ml or 25 ml purge sample as suggested below (unless otherwise specified by the project). Spike 5 ml or 25 ml reagent water for LCS water or 5 g reagent soil in 5 ml reagent water for LCS soil.

9.7.1.1. LCS and Matrix Spike for 5 ml purge

LCS or MS/MSD	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
5 ml	50	50	50

** Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

9.7.1.2. LCS and Matrix Spike for 25 ml purge

LCS or MS/MSD	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
25 ml	10	10	10

** Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

10.0 PROCEDURES

10.1. Sample Preparation

10.1.1. For aqueous samples, refer to EMAX-5030.

10.1.1.1. Check the pH and presence of residual chlorine from remaining sample. Record samples with pH ≥ 2 and residual chlorine ≥ 5 mg/L in the analysis log.

10.1.2. For soil samples, refer to EMAX-5035.

10.2. Instrument Parameters

10.2.1. From the main gas supply (gas Tanks) regulate gas pressure at 80 psi.

10.1.1. Fine-tune the instrument guided by the parameter conditions suggested below. Adjust the parameter conditions accordingly to obtain optimum condition. Print the instrument parameter and post it on the instrument for daily routine maintenance check.

10.2.2. Typical GC Parameters

Carrier gas flow (column) helium	1 – 5 ml/min
Initial Temp	35°C; hold for 1 min.
Rate 1	8°C/min. to 160°C/min
Rate 2	30°C/min to 230°C/min; hold for 3 min.
Inject Port	200°C
Interface	250°C

10.2.3. Mass Spectrometer Parameter

Scan Start	0.5 min.
Mass Range	35 to 300
Multiplier	1200 to 2700

10.2.4. Typical Purge and Trap Condition

10.2.4.1. Purge samples at 40°C for 11 minutes, desorbed at 250°C for 2 minutes and then bake the trap at 260°C for 11 minutes.

10.3. Calibration

10.3.1. Set GC/MS operating condition as described in Section 10.2.

10.3.2. Perform Tune Check

10.3.2.1. Introduce a BFB to yield 5 – 50 ng by either direct injection or purge and trap in 5 ml or 25 ml organic-free water (using tuning standard). Refer to table 4.

10.3.2.2. Evaluate the tune check by the highest scan on the peak or the average of at least 3 scans (before, at and after the apex) with a background subtraction using a single scan no more than 20 scans prior to the elution of BFB.

10.3.2.3. Check Table 5 for acceptance criteria or follow the manufacturer's recommendation for tuning. A valid tune check expires after 12 hours.

10.3.2.4. If non-compliant refer to Section 12 for corrective action.

10.3.3. Initial Calibration (ICAL)

10.3.3.1. Perform ICAL when one of the conditions occurs.

- Instrument is new
- Instrument undergoes a major repair
- DCC failed to meet the acceptance criteria

10.3.3.2. Optimize the instrument condition prior to ICAL

- Ensure that instrument parameters are set up properly
- Ensure that there is no evidence of leak
- Ensure that instrument maintenance is performed on schedule
- Ensure that instrument tune check and column performance is not indicative that it is at the threshold of failing the acceptance criteria

10.3.3.3. Analyze a multi-point initial calibration curve as suggested in Figure 3 after a valid tune check.

10.3.3.4. Base quantitation of identified compounds on the integrated abundance from the EICP of the assigned primary characteristic ion (refer to Tables 6 and 7). For optimum output, assign internal standard to each compound based on the nearest retention time or as suggested on Tables (6 and 7).

10.3.3.5. Evaluate the ICAL Acceptance

10.3.3.5.1. Check for completeness of target compound list. If there is/are missing compound(s), perform the following:

- Check the established retention time window
- Check the relative intensity of major ions
- Adjust accordingly if necessary

10.3.3.5.2. Evaluate retention time of each analyte with respect to the nearest internal standard. The relative retention time (RRT) of each analyte should agree within ± 0.06 RRT units.

10.3.3.5.3. At a minimum, evaluate System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC) as specified in Appendix 1.

10.3.3.5.4. Check RSD and correlation coefficient. If more than 10% of the compounds included with the initial calibration exceed the 15% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Perform necessary instrument maintenance and repeat calibration. Refer to 10.3.3.2, Section 12 for corrective action.

10.3.3.6. Application of ICAL Curve for Quantitation

10.3.3.6.1. Generate a summary of Relative Response Factors for each analyte at each concentration. Calculate the Average Relative Response Factor (RRFm), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to Eq.-10.5.1.1, Eq.-10.5.1.2, Eq.-10.5.1.6 and Eq.-10.5.1.7 respectively.

10.3.3.6.2. If RSD is ≤ 15% average response factor may be applied.

10.3.3.6.3. Apply Inverse Weighting Factor (1/y or $1/y^2$; y being the instrument response) if it is determined to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of ≤ 15% and correlation coefficient of ≥ 0.995.

10.3.3.6.4. Apply linear least squares regression if past experience or priori knowledge of instrument response is known to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of ≤ 15% and correlation coefficient of ≥ 0.995.

10.3.3.6.5. It may be appropriate to force the regression through zero for specific analytes[3]. When exercising this option [as included in the data acquisition software], make sure that the origin (0,0) is not included as a calibration point but rather the intercept is set to zero. This option shall only be applied if the curve favors better accuracy of quantitation.

10.3.3.7. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

10.3.4. Initial Calibration Verification (ICV)

10.3.4.1. Analyze ICV to verify the concentration of the ICAL standards (refer to Section 9.5).

10.3.4.2. Check for completeness of analytes as described in Section 10.4.3.

10.3.4.3. Compare the retention times of the internal standards to the ICAL mid-point. Excursion of ± 30 seconds indicates instrument malfunction. When non-compliant check the column head pressure, gas supply or leaks. Corrective action is required prior to further analysis.

10.3.4.4. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).

[3] SW846 Method 8000B, Section 7.5.3

10.3.4.5. Refer to Appendix 1 for ICV acceptance criteria and/or corrective action.

10.3.4.6. When non-compliant refer to Section 12 for corrective action.

10.3.5. Daily Continuing Calibration (DCC)

10.3.5.1. Analyze DCC to check the validity of the ICAL (refer to 9.6).

10.3.5.2. Check for completeness of analytes as described in Section 10.4.3.

10.3.5.3. Evaluate System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC) as specified in Appendix 1.

10.3.5.4. Compare the retention times of the internal standards to the ICAL mid-point. Excursion of ± 30 seconds indicates instrument malfunction. When non-compliant check the column head pressure, gas supply or leaks. Corrective action is required prior to further analysis.

10.3.5.5. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).

10.3.5.6. Establish RRF of each analyte, calculate %D (Eq.-10.5.2.1) against the ICAL.

10.3.5.7. Refer to Appendix 1 for DCC acceptance criteria and/or corrective action.

10.3.5.8. When non-compliant refer to Section 12 for corrective action.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. Analyze BFB and evaluate tuning

10.4.1.2. Analyze DCC and check ICAL validity

10.4.1.3. Analyze Lab Control Sample

10.4.1.4. Analyze Lab Control Sample Duplicate (if required)

10.4.1.5. Analyze Method Blank

10.4.1.6. Analyze samples to a maximum number of 12-hours from the time of BFB injection.

10.4.1.7. Analyze a pair of matrix spikes (MS/MSD) for every 20 samples of the same matrix.

10.4.1.8. Record analytical sequence in the analysis log.

10.4.2. Sample Result Evaluation

10.4.2.1. Check the QC criteria as soon as the data is available.

- ✓ Check method blank. If result is non-compliant and analyte in question is not detected in any sample or contamination is < 10X of the sample concentration, results maybe reportable. Verify with the PM if results can be reported.
- ✓ Compare the retention times of each Internal Standards (IS) to the ICAL mid point (must be ± 30 seconds).

- ✓ Compare the area of each IS acquired against the mid point of the ICAL. The Extracted Ion Current Profile (EICP) must be within a factor of two (-50 to +100%).
- ✓ Check concentration of target analytes if calibration range is exceeded.
- ✓ Check surrogate recoveries against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 QC limits.
- ✓ If any of the above checkpoints indicate a problem, re-analysis is required. Note observations on the analysis log. When results arise to questionable result, e.g. inconsistency from the first analysis, consult the Supervisor for further action.

10.4.2.2. Properly fill up the analysis log.

10.4.3. Qualitative Identification

- The intensities of the characteristic ions maximize in the same scan or within one scan of each other.
- The relative retention time (RRT) of the sample component is within 0.06 RRT units of the RRT of the standard component.
- The relative intensity of the characteristic ions agrees within 30% of the relative intensity of these ions in the reference spectrum.
- Check the chromatogram for possible misidentified analytes. Investigate visible peaks in the chromatogram that were not identified in the data output. Manually integrate the peak if necessary. For manual integration refer to EMAX-DM01.
- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.[4]

10.4.3.1. For samples containing components not associated with the calibration standards, perform a library search for purposes of tentative identification[5] (TIC). Execute LSC (Chem Station program) to initiate the library search using NIST/EPA/MSDC mass spectral library. Visually inspect each extracted mass ion chromatograph to determine the identification of the unknown before final reporting following the guidelines below.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within + 20%. Example: for an ion with an abundance of 50% of the standard spectra, the corresponding sample ion abundance must be between 30 and 70%.

[4] SW846 Method 8260B, Section 7.6.1.4

[5] Library search is performed only when indicated in the PSR.

- Molecular ions present in reference spectrum should be present in sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting analytes.
- Ions present in the reference spectrum but not present in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting analytes. Data system library reduction programs can sometimes create these discrepancies.

10.4.3.2. **Reporting TICs**

- If the library search produces a match at or above 85%, report the analyte.
- If the library search produces more than one analyte at or above 85%, report the first analyte (highest).
- If the library search produces no matches at or above 85%, the compound should be reported as unknown.

10.4.4. Quantitation

10.4.4.1. Apply the appropriate quantitation method (Section 10.3.3.6). Calculate the concentration of any positively identified target analyte using Eq.-10.5.3. Apply the dilution factor for diluted samples to calculate for the final concentration of the sample.

10.4.5. Manual Integration

10.4.5.1. Refer to EMAX-DM01, Manual Integration Section.

10.4.6. Dealing with Carryover

10.4.6.1. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.

10.4.6.2. If there is no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.

10.4.6.3. If there is any target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule out carry over. If carry over is confirmed, proceed with data reduction and report the data from re-analysis.

10.4.6.4. To clean-up the autosampler purge line consider purging a 25 ml or 5 ml sample spiked with 100 µl of methanol and let it run like a blank sample. If improved result is noted repeat this process until no evidence of contamination is observed. Otherwise inform the Supervisor for further instruction.

10.5. **Calculations**

10.5.1. Initial Calibration

10.5.1.1. **Calculate for the Relative Response Factor (RRF)**

$$RRF = \frac{A_X C_{IS}}{A_{IS} C_X}$$ Eq.-10.5.1.1

where:

A_X – Area of characteristic ion for the compound being measured
A_{IS} – Area of characteristic ion for the specific internal standard
C_X – Concentration of the compound being measured
C_{IS} – Concentration of the specific internal standard

10.5.1.2. **Calculate for the Average Relative Response Factor (RRF_m)**

$$RRF_m = \frac{\sum RRF}{n}$$ Eq.-10.5.1.2

where:

$\sum RRF$ – Summation of response factors
n – Number of measurements

10.5.1.3. **Calculate for Least Square Linear Regression**

$$y = ax + b$$ Eq.-10.5.1.3

where:

y – Response ratio (A_X/A_{IS})
x – Amount ratio (C_X/C_{IS})
a – x1 = slope of the line

$$a = \frac{\sum (x - \bar{x})(y - \bar{y})}{\sum (x - \bar{x})^2}$$

where:

$\bar{x}$ = average of amount ratios
$\bar{y}$ = average of response ratios

b – x0 = intercept of the line

$$b = \bar{y} - a * \bar{x}$$

10.5.1.4. **Calculate for Inverse Weighting Factor**

$$y = ax + b$$ Eq.-10.5.1.4

where:

y – Response ratio (A_X/A_{IS})
x – Amount ratio (C_X/C_{IS})
a – x1 = slope of the line

$$a = \frac{\sum (x - x_a)(y - y_a)}{\sum (x - x_a)^2}$$

where:

$$x_a = \sum \left[x(1/x) / \sum (1/x) \right]$$
$$y_a = \sum \left[y(1/x) / \sum (1/x) \right]$$

or $x_a = \sum\left[x(1/x^2)/\sum(1/x^2)\right]$

$y_a = \sum\left[y(1/x^2)/\sum(1/x^2)\right]$

b – x0 = intercept of the line

$b = y_a - a * x_a$

10.5.1.5. **Calculate Inverse Quadratic**

$y = ax^2 + bx + c$

where:

y – Resp_Ratio =**x0** + **x1** * Amt_Ratio + **x2** * (Amt_Ratio)^2

x – Amt_Ratio

c – **x0**=Det 0/Det b

b – **x1**=Det 1/Det b

a – **x2**=Det 2/Det b

$$W_i = \frac{\frac{1}{x_i}}{\sum_{i=1}^{n}\frac{1}{x_i}}$$

where:

X_i = amount ratio= Conc of Std/Conc of IS

Y_i = response ratio=Resp of Std/Resp of IS

Wi= 1/X)/SUM(1/X)

<X>= SUM(W_i*X_i)

<Y>= SUM(W_i*Y_i)

<XX>= SUM(W_i*(X_i)^2)

<XXX>= SUM(W_i*(X_i)^3)

<XXXX>= SUM(W_i*(X_i)^4)

<YY>= SUM(W_i*(Y_i)^2)

<XY>= SUM(W_i*X_i*Y_i)

<XXY>= SUM(W_i*(X_i)^2*Y_i)

<Yd2>= SUM((Y_i-<Y>)^2*W_i)

Ye= x0+x1*X_i+x2*X_i^2-<Y>)

<Ye2>= SUM(Ye^2*W_i)

Det b	1	<X>	<XX>
	<X>	<XX>	<XXX>
	<XX>	<XXX>	<XXXX>

Det 0	1	<X>	<XX>
	<X>	<XX>	<XXX>
	<Y>	<XY>	<XXY>

Det 1	1	<X>	<XX>
	<Y>	<XY>	<XXY>
	<XX>	<XXX>	<XXXX>

Det 2	<Y>	<XY>	<XXY>
	<X>	<XX>	<XXX>
	<XX>	<XXX>	<XXXX>

r2= <Ye2>/<Yd2>

ccf2= $(r2)^{1/2}$

10.5.1.6. **Calculate the Standard Deviation (SD)**

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}} \qquad \text{Eq.-10.5.1.6}$$

where:

x_i – Result at i^{th} measurement

$\bar{x}$ – Mean of the n measurements

n – Number of measurements

10.5.1.7. **Calculate the % relative standard deviation (%RSD)**

$$\%RSD = \frac{SD}{RRF_m} * 100\% \qquad \text{Eq.-10.5.1.7}$$

where:

SD – Standard deviation

RRF_m – Average response factor

10.5.1.8. **Calculate the relative retention time (RRT)**

$$\text{RRT} = \frac{\text{Retention Time of the Analyte}}{\text{Retention Time of the Internal Standard}} \qquad \text{Eq.-10.5.1.8}$$

10.5.2. Calibration Check/Continuing Calibration

10.5.2.1. **Calculate Percent Difference (%D)**

$$\%D = \frac{\left[RRF_c - RRF_m\right]}{RRF_m} * 100\% \qquad \text{Eq.-10.5.2.1}$$

where:

RRF_c – Response factor from continuing calibration standard
RRF_m – Average response factor

10.5.2.2. **% Drift**

$$\%Drift = \frac{\left[found\ Conc. - true\ Conc\right]}{true\ Conc.} * 100\% \qquad \text{Eq.-10.5.2.2}$$

10.5.3. Calculation of Sample Concentration (Water and Soil/Sediment Samples)

10.5.3.1. When a compound is identified, the quantitation of that compound shall be based on the integrated abundance from the EICP of the primary characteristic ion.

10.5.3.2. **Water Samples**

$$Concentration\ (ug/L) = \frac{(Ax)(Is)}{(Ais)(RRF_m)}\ x\ DF \qquad \text{Eq.-10.5.3.2}$$

where:

A_x – Area of characteristic ion for the compound to be measured
I_s – Concentration of internal standard added in µg/L
A_{is} – Area of characteristic ion for the internal standard
RRF_m – Average response factor
DF – Dilution factor = $\frac{purge\ volume\ in\ ml\ \ (5\ ml\ or\ 25\ ml)}{sample\ \ amount\ \ in\ ml}$

10.5.3.3. **Soil/Sediment Samples (Dry weight basis)**

$$Concentration\ (ug/kg) = \frac{(Ax)(Is)}{(Ais)(RRF_m)(DW)}\ x\ DF \qquad \text{Eq.-10.5.3.3}$$

where:

A_x – Area of characteristic ion for the compound to be measured
I_s – Concentration of internal standard added in µg/L
A_{is} – Area of characteristic ion for the internal standard
RRF_m – Average response factor
DF – Dilution factor = $\frac{5\ g}{\left(sample\ \ amount\ \ in\ g\right)}$
DW – % solid = $\frac{100 - \%moisture}{100}$

10.5.3.4. **Extracted Soil/Sediment Samples (Dry weight basis)**

$$Concentration\ (ug/kg) = \frac{(Ax)(Is)}{(Ais)(RRF_m)(DW)}\ x\ DF \qquad \text{Eq.-10.5.3.4}$$

where:

A_x – Area of characteristic ion for the compound to be measured

I_s – Concentration of internal standard added in µg/L
A_{is} – Area of characteristic ion for the internal standard
RRF_m – Average response factor
DF – Dilution factor $\frac{(purged \quad volume \quad in \ \mu L)(5\,g)}{(extract \quad aliquot \quad in \ \mu L)(sample \quad amount \quad in \ g)}$
DW – % solid = $\frac{100 - \%moisture}{100}$

10.5.4. Alternatively, the regression line (area ratio of Ax/Ais versus concentration using first degree) fitted to the initial calibration may be used for determination of the sample concentration when RSD of the analyte is greater than 15% (Section 10.3.3.6) .

10.5.5. Concentration of TIC is estimated by the same method as target compounds with the following assumptions:

10.5.5.1. The area Ax and Ais are derived from total ion chromatogram. Ais refers to the closest internal standard (IS) free of interference.

10.5.5.2. RRF of the TIC is 1.

10.5.6. Method Proficiency

10.5.6.1. **Percent Recovery**

$$\%\,\mathrm{Recovery} = \frac{(C_f - C)}{C_s} \times 100 \qquad \text{Eq.-10.5.6.1}$$

where:

C_f – Concentration found
C – Concentration of sample (use 0 for LCS)
C_s – Concentration of spike

10.5.6.2. **Relative Percent Difference (RPD)**

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \qquad \text{Eq.-10.5.6.2}$$

where:

C_1 – Measured concentration of the first sample aliquot
C_2 – Measured concentration of the second sample aliquot

10.6. **Data Reduction**

10.6.1. Make a copy of the analysis log.

10.6.2. Print a copy of the sample weight log (if any).

10.6.3. Highlight the data to be reported.

10.6.4. Print a copy of the raw data and the QC report.

10.6.5. Collate the reportable raw data separating the QC results from the sample results.

10.6.6. Keep all other data generated with the analytical folder marked with “For record only”.

10.7. **Report Generation**

10.7.1. Generate the method.txt file using WDB1C.exe.

10.7.2. Generate the sample results using F1NV3C.exe or F1NV3C4.exe.

10.7.3. Generate the QC summary using QCV3CN.exe or QCV3CN4.exe.

10.7.4. Generate the Instrument Performance Check (ICAL and DCC) using F5VOA.exe.

10.7.5. Generate the IS and RT summary using F8VC.exe.

10.7.6. Generate Lab Chronicle using LABCHRN1.exe

10.7.7. Generate Case Narrative using CN1.exe

10.7.8. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration and re-analyses.

10.7.8.1. Case Narrative

10.7.8.2. Lab Chronicle

10.7.8.3. Sample Results

10.7.8.4. Method Blank Results

10.7.8.5. LCS/LCSD Summary

10.7.8.6. MS/MSD Summary

10.7.8.7. Instrument Performance Check (ICAL)

10.7.8.8. ICAL Summary

10.7.8.9. ICV Summary

10.7.8.10. Instrument Performance Check (DCC)

10.7.8.11. IS and RT Summary

10.7.8.12. DCC Summary

10.7.8.13. Analysis Log

10.7.8.14. Sample Weight Log (if any)

10.7.8.15. Non-Conformance Report (If any)

10.8. **Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01and the PSR.

10.8.1.1. If any of the checkpoints below indicates a problem, re-analysis is required.

- ✓ Check internal standard area. They should be within -50 to +100% of ICAL midpoint to be acceptable, otherwise follow PSR.

- ✓ Check retention time of each IS to the ICAL midpoint. They should be within ± 30 seconds to be acceptable, otherwise follow PSR.
- ✓ Check surrogate recoveries against project specific criteria (PSR). In the absence of PSR, default to in-house QC limits.
- ✓ Check concentration of target analytes if calibration range is exceeded.

10.8.1.2. Review the attached logs that they are properly filled.

10.8.1.3. Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.8.1.4. Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.2. Submit the analytical folder for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Perform instrument routine preventive maintenance and record on instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction that may affect data quality.

10.9.2. The table below list suggested routine maintenance schedule.

Task	Every Day	Every Week	Every Month	Every 3 Months	Every 6 Months	As Needed
Tune Check	✓					
Check gas cylinders pressure	✓					
Check the foreline pump oil level		✓				
Check the calibration vial						✓
Check and if necessary, change injection port liners, septa and O-rings.					✓	
Replace the foreline pump oil					✓	
Replace the diffusion pump fluid					✓	
Replace the traps and filters					✓	
Clean the ion source						✓
Change the carrier gas trap(s) and purifier						✓
Replace column						✓
AutoTune the MSD						✓
Replace the worn out parts						✓

11.0 QUALITY CONTROL

11.1. **Analytical Batch QC**

11.1.1. Perform tune check to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to calibration and check for any contamination.

11.1.2. Perform initial calibration (ICAL) to establish a calibration curve for the quantification of the analytes of interest.

11.1.3. Establish retention time window position for each analyte every after ICAL for proper qualitative identification.

11.1.4. Perform initial continuing calibration verification (ICV) every after ICAL to verify accuracy of ICAL.

11.1.5. Perform continuing calibration verification (CCV) every 12 hours to verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.

11.1.6. Evaluate relative retention time for each analytes in every sample to be within ± 0.06 RRT units.

11.1.7. Verify internal standard (IS) for quantitative accuracy and that its Retention time is within ± 30 seconds from retention time of the midpoint standard in the ICAL and EICP area is within -50% to +100% of ICAL midpoint standard.

11.1.8. Evaluate surrogate recovery to monitor instrument response on every sample.

11.2. **Preparation Batch QC**

11.2.1. Reagent water used for IB shall be of the same source for all QC samples and sample dilutions.

11.2.2. Analyze MB, LCS, MS/MSD and < 20 field samples.

11.2.3. Solvents and reagents must undergo quality control check prior to its use. Refer to EMAX-QC01 for details.

11.2.4. Properly treat lab wares used in the sample preparation as specified in EMAX-QC07.

11.3. **Method QC**

11.3.1. All analytes reported must have a valid DL, LOD and LOQ as described in EMAX-QA04.

11.3.2. All analysts conducting this analysis must demonstrate capability (IDOC/DOC) as described in EMAX-QA05.

11.4. Refer to Appendix 1 for all related Quality Control parameters, frequency and acceptance criteria.

12.0 CORRECTIVE ACTION

12.1. Corrective action for each Quality Control procedure is summarized in Appendix 1.

12.2. **Analytical Batch QC**

12.2.1. Tune Check – If tune check is non-compliant consider the following suggestion to correct the problem:

- Check the abundance of mass 95 and 174. If it is significantly less than previous tune checks, it is indicative of insufficient amount of BFB injected. Probable causes are:

improper spiking, leaks, standard degradation or low vacuum system. Repeat tune check ensuring that BFB was properly spiked or rule out leaks, prepare a fresh BFB standard and repeat the tune check.

- If problem persist, re-tune the instrument and repeat tune check.
- If problem is unresolved, inform the supervisor for further action.

12.2.2. Initial Calibration

12.2.2.1. If the %RSD is out of acceptance criteria, consider the following suggestions to correct the problem.

- If one of the standards returns a bias low or bias high on all of the analytes then that point is considered an out-liner. Prepare a standard at that ICAL point and re-analyze.
- If the highest ICAL point appears to be saturated, drop the highest point.
- If the lowest point returns a bias low or bias high response or the peaks are not distinct and sharp, drop the lowest point.

Note : The lowest calibration point identifies the limit of quantitation (LOQ). Therefore, check that the LOQ is in conformance to the current projects where the ICAL will be used.

12.2.2.2. If instrument problem is suspected, consider the following suggestion to correct the problem:

- Check the connection and make sure they are air tight and perform maintenance as needed.
- Check the gas flow.
- Re-tune the MS.
- Prepare a fresh standard and repeat calibration.
- Clean the MS source and repeat calibration.
- If problem is unresolved, inform the supervisor for further action.

12.2.3. Initial Calibration Verification (ICV) – If the ICV is non-compliant, consider the following suggestions to correct the problem:

- Re-analyze ICV to rule out poor purge.
- If ICV is still out of acceptance criteria, prepare a fresh standard and re-analyze to rule out any preparation error.
- If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration.
- If problem is unresolved, inform the supervisor for further action.

12.2.4. Daily Calibration Check (DCC) – If DCC is non-compliant consider the following suggestions to correct the problem:

- If majority of the analyte response are low and no evidence of leak in the system is apparent, it is indicative of a bad purge or leak in the vial. Re-analyze DCC.
- If problem persist, rule out standard degradation. Prepare a fresh standard and repeat DCC.
- Otherwise execute instrument maintenance and perform ICAL.

12.2.5. Instrument Blank – If instrument blank is non-compliant, consider the following suggestions to correct the problem:

- If trace level of THMs is observed, it is indicative that water filters need replacement. Otherwise, bake the trap at the manufacturer's recommended temperature for about 30 minutes.
- If contamination is high, flush the sample line with methanol and replace the trap.
- If problem is unresolved, inform the supervisor for further action.

12.3. **Preparation Batch QC**

12.3.1. For insufficient amount of sample(s), inform the supervisor immediately for further action.

12.3.2. If MB is non-compliant, consider the suggestions as described in Instrument Blank.

12.3.3. If LCS is non-compliant, consider the following suggestions to correct the problem:

- If result is bias low or high, prepare a fresh standard and re-analyze LCS and the associated samples.
- If problem is unresolved, inform the supervisor for further advice.

12.3.4. If MS is non-compliant consider the following suggestion to correct the problem:

- Check the standard log and analytical log and verify that the spike amount value used for calculation is correct.
- If LCS is within acceptance criteria then and the right amount of spike amount used for calculation is correct, then it is indicative of matrix interference. Discuss the probable matrix interference in the case narrative.

12.4. Discuss water samples that are labeled preserved having a pH ≥ 2 and/or residual chlorine ≥ 5 mg/L in the case narrative.

12.5. A Non-Conformance Report (NCR) is required when the following circumstances occur.

- Anomalies other than specified in Appendix 1, is observed.
- Sample is out of technical holding time.

12.5.1. Refer to EMAX-QA08 for NCR details.

13.0 POLLUTION PREVENTION

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2. Prepare all standards in fume hoods.

14.0 WASTE MANAGEMENT

14.1. No samples shall be dumped on the laboratory sink.

14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.

14.3. Place all waste generated during analytical process in properly labeled satellite waste containers for proper collection.

14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. **Definition of Terms**

15.1.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.

15.1.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

15.1.2.1. **Preparation Batch** - is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.

15.1.2.2. **Analytical batch** - is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.3. Detection Limit (DL) – is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

15.1.4. Limit of Detection (LOD) – is defined as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative result rate (Type II error) is 1 %.

15.1.5. Limit of Quantitation (LOQ) – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

15.1.6. Safety Data Sheet (SDS) – is written information concerning a chemical physical properties, toxicity, health hazards, fire hazard and reactivity data including storage, spill and handling precautions.

15.1.7. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.8. Calibration Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.9. Carry-over – are contaminants retained in the instrument/apparatus from a highly contaminated sample that is passed into the succeeding sample(s).

15.1.10. Calibration Check Compounds (CCC) – evaluate the integrity of the system. Variability of these compounds may indicate system leak or reactive sites in the column.

15.1.11. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.1.12. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.

15.1.13. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.

15.1.14. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.

15.1.15. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

15.1.16. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.17. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.18. Matrix – is a component or form of a sample.

15.1.19. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.

15.1.20. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

15.1.21. Response Factor – is the ratio of the peak area of the target compound in the sample or sample extract.

15.1.22. Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard; used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.

15.1.23. SPCC – System performance check compounds are compounds that are used to check compound stability and to check for degradation cause by contaminated lines or active sites in the system.

15.1.24. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.

15.1.25. Reagent Soil – organic-free Ottawa sand or equivalent.

15.2. **Application of EMAX QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing Volatile analysis by GC/MS. The standard analyte list and RL are presented in Tables 6 & 7. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

15.3. **Department of Defense (DoD) and Department of Energy (DoE) Projects**

15.3.1. Samples from DoD and DoE sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, is applied.

16.0 REFERENCES

16.1. U.S. EPA Method 8260B; SW846, as updated

16.2. EMAX Quality Systems Manual, as updated

17.0 APPENDICES

17.1. **Tables**

17.1.1.	Table 1	Initial Calibration Intermediate Standards Preparation
17.1.2.	Table 2	Initial Calibration Verification/LCS/MS/MSD Intermediate Standards Preparation
17.1.3.	Table 3	Surrogate/Internal Standards Preparation
17.1.4.	Table 4	Tuning Solution Standards Preparation
17.1.5.	Table 5	BFB Key Ion Abundance Criteria
17.1.6.	Table 6	Typical Analyte List, Quantitation Ions, IS, Surrogates, Calibration Standards, Detection Limits for 5 ml Purge
17.1.7.	Table 7	Typical Analyte List, Quantitation Ions, IS, Surrogates, Calibration Standards, Detection Limits for 25 ml Purge

17.2. **Figures**

17.2.1.	Figure 1	Peak Evaluation Techniques
17.2.2.	Figure 2	Typical Chromatogram
17.2.3.	Figure 3	Typical ICAL Summary
17.2.4.	Figure 4	Typical Instrument Performance Check (Tuning)
17.2.5.	Figure 5	Typical Instrument Performance Check (Tuning) Summary
17.2.6.	Figure 6	Typical Internal Standard Area and Retention Time Summary
17.2.7.	Figure 7	Typical Sample Result Summary
17.2.8.	Figure 8	Typical LCS/LCSD Summary
17.2.9.	Figure 9	Typical MS/MSD Summary
17.2.10.	Figure 10	Typical Case Narrative

17.3. **Appendices**

17.3.1.	Appendix 1	Summary of Quality Control Procedures
17.3.2.	Appendix 2	Demonstration of Capability for 25 ml
17.3.3.	Appendix 3	Demonstration of Capability for 5 ml
17.3.4.	Appendix 4	Demonstration of Capability for 5 g

17.4. **Forms**

17.4.1.	8260FS	Sample Preparation Log
17.4.2.	8260FA	Analytical Run Log
17.4.3.	8260FM	Instrument Maintenance Log

Table 1: INITIAL CALIBRATION INTERMEDIATE STANDARDS PREPARATION

ICAL/DCC Intermediate Standard	Stock Standard			Preparation (Solvent: Methanol)		Final Conc. (mg/L)
	Standard Name	Source	Conc. (mg/L)	Aliquot (µl)	Final Vol. (ml)	
I	1-Chlorohexane	AccuStandard	2000	50	2	50
	2-Chloroethylvinylether	CPI	2000	50	2	50
	Oxygenate Gasoline Additive	AccuStandard	2000-10000	50	2	50 - 250
	Custom VOA Mix	CPI	2000, 20000, 40000	50	2	50, 500, 1000
II	VOC Gas Mix	Ultra Scientific	2000	250	2	250
	Vinyl Acetate	CPI	2500	200	2	250
III	Carbon Disulfide	CPI	5000	100	2	250
IV	VOA Calibration Mix 1	Restek	5000	100	2	250
	Acrolein / Acrylonitrile	AccuStandard	5000	100	2	250

Table 2: INITIAL CALIBRATION VERIFICATION/LCS/MS/MSD INTERMEDIATE STANDARDS PREPARATION

ICV / LCS / MS Intermediate Standard	Stock Standard			Preparation (Solvent: Methanol)		Final Conc. (mg/L)
	Standard Name	Source	Conc. (mg/L)	Aliquot (µl)	Final Vol. (ml)	
I	1-Chlorohexane	Ultra Scientific	1000	100	2	50
	2-Chloroethylvinylether	AccuStandard	2000	50	2	50
	California Oxygenate Mix	Restek	2000 - 10000	50	2	50 – 250
	Custom 8260 Mega Mix	Restek	2000, 20000, 40000	50	2	50, 500, 1000
II	Volatile Organic Cpds Mix 6	Supelco	2000	250	2	250
	Vinyl Acetate	Restek	2000	250	2	250
III	Carbon Disulfide Solution	Ultra scientific	5000	20	2	50
IV	TCL Volatile Mix 1	Supelco	2000	250	2	250
	Acrolein / Acrylonitrile	Ultra Scientific	2000	250	2	250

Table 3: SURROGATE / INTERNAL STANDARDS PREPARATION

Intermediate Standard	Stock Standard			Preparation (Solvent: Methanol)		Final Conc. (mg/L)
	Standard Name	Source	Conc. (mg/L)	Aliquot (µl)	Final Vol. (ml)	
Surrogate	8260 Surrogate Mix	Restek	2500	200	2	250
Internal Standard	Custom 8260 Internal Standard Mix, 3-30	CPI	2500	200	2	250

Table 4: TUNING SOLUTION STANDARDS PREPARATION

BFB Intermediate Standard	Stock Standard			Preparation (Solvent: Methanol)		Final Conc. (mg/L)
	Standard Name	Source	Conc. (mg/L)	Aliquot (µl)	Final Vol. (ml)	
Tuning Compound	BFB	Restek	5000	20	2	50

Table 5: BFB KEY ION ABUNDANCE CRITERIA

M/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Table 6: TYPICAL TARGET ANALYTE LIST FOR 5-ml PURGE

ANALYTES	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC	LCS/MS	WATER (µg/L)			SOIL (µg/Kg)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8	EV(µg/L)	EV(µg/L)	DL	LOD	LOQ	DL	LOD	LOQ
1,1,1,2-Tetrachloroethane	131	133, 119, 117	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1,1-Trichloroethane	97	99, 61	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1,2,2-Tetrachloroethane	83	85	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1,2-Trichloro-1,2,2-trifluoroethane	151	153	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
1,1,2-Trichloroethane	97	83, 85, 99	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1-Dichloroethane	63	65, 83	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1-Dichloroethene	61	63, 96	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1-Dichloropropene	110	112	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,2,3-Trichlorobenzene	180	182, 145	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
1,2,3-Trichloropropane	110	61, 77	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
1,2,4-Trichlorobenzene	180	182, 145	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
1,2,4-Trimethylbenzene	105	120	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.55	1	5
1,2-Dibromo-3-chloropropane	157	155, 75	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
1,2-Dibromoethane	107	109	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,2-Dichlorobenzene	146	111, 148	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,2-Dichloroethane	62	64	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,2-Dichloropropane	63	41, 76	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,3,5-Trimethylbenzene	105	120, 119	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.59	1	5
1,3-Dichlorobenzene	146	111, 148	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.52	1	5
1,3-Dichloropropane	76	78	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,4-Dichlorobenzene	146	111, 148	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1-Chlorohexane	91	93, 55, 56	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.58	1	5
2,2-Dichloropropane	77	97, 79	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
2-Butanone (MEK)	43	72	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	2.5	5	10
2-Chloroethyl vinyl ether	63	65, 106	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
2-Chlorotoluene	91	126	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.82	1	5
2-Hexanone (MBK)	43	58, 100	IS2	Sur3	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	2.86	5	10
4-Chlorotoluene	91	126	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.61	1	5	0.67	1	5
4-Methyl-2-pentanone (MIBK)	43	58, 85, 100	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	2.75	5	10
Acetone	43	58, 42	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	3.06	5	10
Acrolein	56	55	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	2.5	5	10
Acrylonitrile	53	52, 51	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	2.5	5	10
Benzene	78	77, 52	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Bromobenzene	156	77, 158	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Bromochloromethane	49	128, 130	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Bromodichloromethane	83	85	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Bromoform	173	171, 175	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
Bromomethane	94	96	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1.1	2	5	1.81	2	5

Table 6: TYPICAL TARGET ANALYTE LIST FOR 5-ml PURGE

ANALYTES	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC	LCS/MS	WATER (µg/L)			SOIL (µg/Kg)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8	EV(µg/L)	EV(µg/L)	DL	LOD	LOQ	DL	LOD	LOQ
Carbon disulfide	76	78	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Carbon tetrachloride	119	117	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.54	1	5
Chlorobenzene	112	51, 77, 114	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Chloroethane	64	49, 66	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
Chloroform	83	85, 47	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Chloromethane	50	52	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1.02	2	5
cis-1,2-Dichloroethene	96	61, 98	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
cis-1,3-Dichloropropene	75	77, 39, 110	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Dibromochloromethane	129	127	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Dibromomethane	93	95, 174	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Dichlorodifluoromethane	85	87	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1.3	2	5	1.16	2	5
Dichlorofluoromethane	67	69	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Diisopropyl ether (DIPE)	45	87	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Ethyl Methacrylate	69	99, 41	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
Ethylbenzene	91	106	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Ethyl-tert-butyl ether (ETBE)	59	87	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Hexachlorobutadiene	225	223, 227	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
Iodomethane	142	127	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
Isopropylbenzene	105	120, 79, 103	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.64	1	5
m/p-Xylenes	91	106	IS2	Sur3	4	10	20	40	100	160	200	400	100	100	1	2	10	1	2	10
Methylene chloride	49	84, 86	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
Methyl-t-butyl ether (MTBE)	73	57	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Naphthalene	128	127	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
n-Butylbenzene	91	92, 134	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.73	1	5	0.7	1	5
n-Propylbenzene	91	65, 120	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.51	1	5	0.65	1	5
o-Xylene	91	106	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
p-Isopropyltoluene	119	91, 134	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.56	1	5	0.62	1	5
sec-Butylbenzene	105	134	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.67	1	5
Styrene	104	78, 103	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
tert-Amylmethyl ether (TAME)	87	55,73	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
tert-Butyl alcohol (TBA)	59	41	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	7.1	10	25	9.18	10	20
tert-Butylbenzene	134	91, 119	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.62	1	5
Tetrachloroethene	164	129, 131, 166	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.52	1	5	0.5	1	5
Toluene	91	92	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
trans-1,2-Dichloroethene	61	96, 98	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
trans-1,3-Dichloropropene	75	77, 39	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
trans-1,4-Dichloro-2-butene	53	88	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
Trichloroethene	130	97, 132, 95	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5

Table 6: TYPICAL TARGET ANALYTE LIST FOR 5-ml PURGE

ANALYTES	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC	LCS/MS	WATER (µg/L)			SOIL (µg/Kg)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8	EV(µg/L)	EV(µg/L)	DL	LOD	LOQ	DL	LOD	LOQ
Trichlorofluoromethane	101	103	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.86	1	5	1.06	2	5
Vinyl acetate	43	86	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1.26	2	5
Vinyl chloride	62	64	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.61	1	5	1	2	5
Dibromofluoromethane (Sur0)	111	113, 192	IS1		2	5	10	20	50	80	100	200	50	50	1	2	5	0.5	1	5
1,2-Dichloroethane-d4 (Sur1)	65	102	IS1		2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
4-Bromofluorobenzene (Sur2)	95	174, 176	IS3		2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
Toluene-D8 (Sur3)	98	100	IS2		2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
1,4-Difluorobenzene (IS1)	114	88																		
Chlorobenzene-d5 (IS2)	117	82, 119																		
1,2-Dichlorobenzene-d4 (IS3)	152	150																		

Note: Since, retention time of Dibromofluoromethane (Sur0) and 1,2-Dichloroethane-d4 (Sur1) is too close (~43 sec) hence, Dibromofluoromethane (Sur0) is only used when required by the project.

Table 7: TYPICAL TARGET ANALYTE LIST FOR 25-ml PURGE

Analytes	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)									ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER (µg/L)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8	9			DL	LOD	LOQ
1,1,1,2-Tetrachloroethane	131	133, 119, 117	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,1,1-Trichloroethane	97	99, 61	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,1,2,2-Tetrachloroethane	83	85	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
1,1,2-Trichloro-1,2,2-trifluoroethane	151	153	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.17	0.3	0.5
1,1,2-Trichloroethane	97	83, 85, 99	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,1-Dichloroethane	63	65, 83	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,1-Dichloroethene	61	63, 96	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,1-Dichloropropene	110	112	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,2,3-Trichlorobenzene	180	182, 145	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
1,2,3-Trichloropropane	110	61, 77	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
1,2,4-Trichlorobenzene	180	182, 145	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
1,2,4-Trimethylbenzene	105	120	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
1,2-Dibromo-3-chloropropane	157	155, 75	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
1,2-Dibromoethane	107	109	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,2-Dichlorobenzene	146	111, 148	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,2-Dichloroethane	62	64	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,2-Dichloropropane	63	41, 76	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,3,5-Trimethylbenzene	105	120, 119	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.13	0.2	0.5
1,3-Dichlorobenzene	146	111, 148	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
1,3-Dichloropropane	76	78	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,4-Dichlorobenzene	146	111, 148	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1-Chlorohexane	91	93, 55, 56	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.14	0.2	0.5
2,2-Dichloropropane	77	97, 79	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.16	0.2	0.5
2-Butanone (MEK)	43	72	IS1	Sur0/1	1.5	2.5	5	10	25	50	100	150	200	50	50	2	4	10
2-Chloroethyl vinyl ether	63	65, 106	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
2-Chlorotoluene	91	126	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.12	0.2	0.5
2-Hexanone (MBK)	43	58, 100	IS2	Sur3	1.5	2.5	5	10	25	50	100	150	200	50	50	2.3	4	5
4-Chlorotoluene	91	126	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
4-Methyl-2-pentanone (MIBK)	43	58, 85, 100	IS1	Sur0/1	1.5	2.5	5	10	25	50	100	150	200	50	50	2.2	4	5
Acetone	43	58, 42	IS1	Sur0/1	1.5	2.5	5	10	25	50	100	150	200	50	50	2.6	5	10
Acrolein	56	55	IS1	Sur0/1	1.5	2.5	5	10	25	50	100	150	200	50	50	2.5	5	10
Acrylonitrile	53	52, 51	IS1	Sur0/1	1.5	2.5	5	10	25	50	100	150	200	50	50	2.5	5	10
Benzene	78	77, 52	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5

Table 7: TYPICAL TARGET ANALYTE LIST FOR 25-ml PURGE

Analytes	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)									ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER (µg/L)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8	9			DL	LOD	LOQ
Bromobenzene	156	77, 158	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Bromochloromethane	49	128, 130	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
Bromodichloromethane	83	85	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Bromoform	173	171, 175	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
Bromomethane	94	96	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.16	0.3	0.5
Carbon disulfide	76	78	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Carbon tetrachloride	119	117	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Chlorobenzene	112	51, 77, 114	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Chloroethane	64	49, 66	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.27	0.3	0.5
Chloroform	83	85, 47	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Chloromethane	50	52	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
cis-1,2-Dichloroethene	96	61, 98	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
cis-1,3-Dichloropropene	75	77, 39, 110	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Dibromochloromethane	129	127	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Dibromomethane	93	95, 174	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Dichlorodifluoromethane	85	87	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
Dichlorofluoromethane	67	69	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Diisopropyl ether (DIPE)	45	87	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
Ethyl Methacrylate	69	99, 41	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
Ethylbenzene	91	106	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Ethyl-tert-butyl ether (ETBE)	59	87	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
Hexachlorobutadiene	225	223, 227	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.22	0.3	0.5
Iodomethane	142	127	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
Isopropylbenzene	105	120, 79, 103	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
m/p-Xylenes	91	106	IS2	Sur3	0.6	1	2	4	10	20	40	60	80	20	20	0.21	0.4	1
Methylene chloride	49	84, 86	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
Methyl-t-butyl ether (MTBE)	73	57	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.13	0.2	0.5
Naphthalene	128	127	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
n-Butylbenzene	91	92, 134	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.17	0.2	0.5
n-Propylbenzene	91	65, 120	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.13	0.2	0.5
o-Xylene	91	106	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
p-Isopropyltoluene	119	91, 134	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.14	0.2	0.5
sec-Butylbenzene	105	134	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.13	0.2	0.5

Table 7: TYPICAL TARGET ANALYTE LIST FOR 25-ml PURGE

Analytes	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)									ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER (µg/L)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8	9			DL	LOD	LOQ
Styrene	104	78, 103	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
tert-Amylmethyl ether (TAME)	87	55,73	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
tert-Butyl alcohol (TBA)	59	41	IS1	Sur0/1	1.5	2	5	10	25	50	100	150	200	50	50	2.5	5	10
tert-Butylbenzene	134	91, 119	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.13	0.2	0.5
Tetrachloroethene	164	129, 131, 166	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.2	0.5
Toluene	91	92	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
trans-1,2-Dichloroethene	61	96, 98	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
trans-1,3-Dichloropropene	75	77, 39	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
trans-1,4-Dichloro-2-butene	53	88	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
Trichloroethene	130	97, 132, 95	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Trichlorofluoromethane	101	103	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
Vinyl acetate	43	86	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
Vinyl chloride	62	64	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.12	0.2	0.5
Dibromofluoromethane (Sur0)	111	113, 192	IS1		0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
1,2-Dichloroethane-d4 (Sur1)	65	102	IS1		0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
4-Bromofluorobenzene (Sur2)	95	174, 176	IS3		0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
Toluene-D8 (Sur3)	98	100	IS2		0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
1,4-Difluorobenzene (IS1)	114	88																
Chlorobenzene-d5 (IS2)	117	82, 119																
1,2-Dichlorobenzene-d4 (IS3)	152	150																

Note: Since retention time of Dibromofluoromethane (Sur0) and 1,2-Dichloroethane-d4 (Sur1) is too close (~43 sec) hence, Dibromofluoromethane (Sur0) is only used when required by the project.

Figure 1: PEAK EVALUATION TECHNIQUES

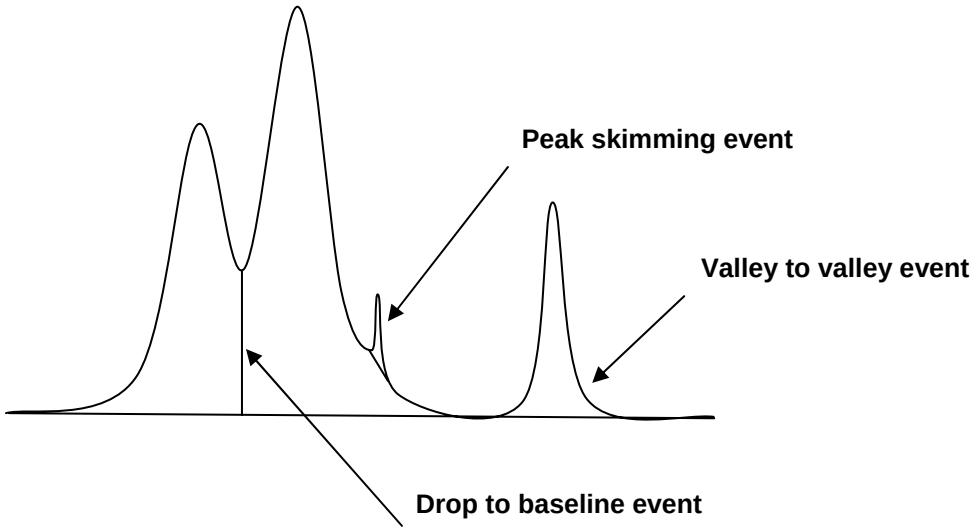
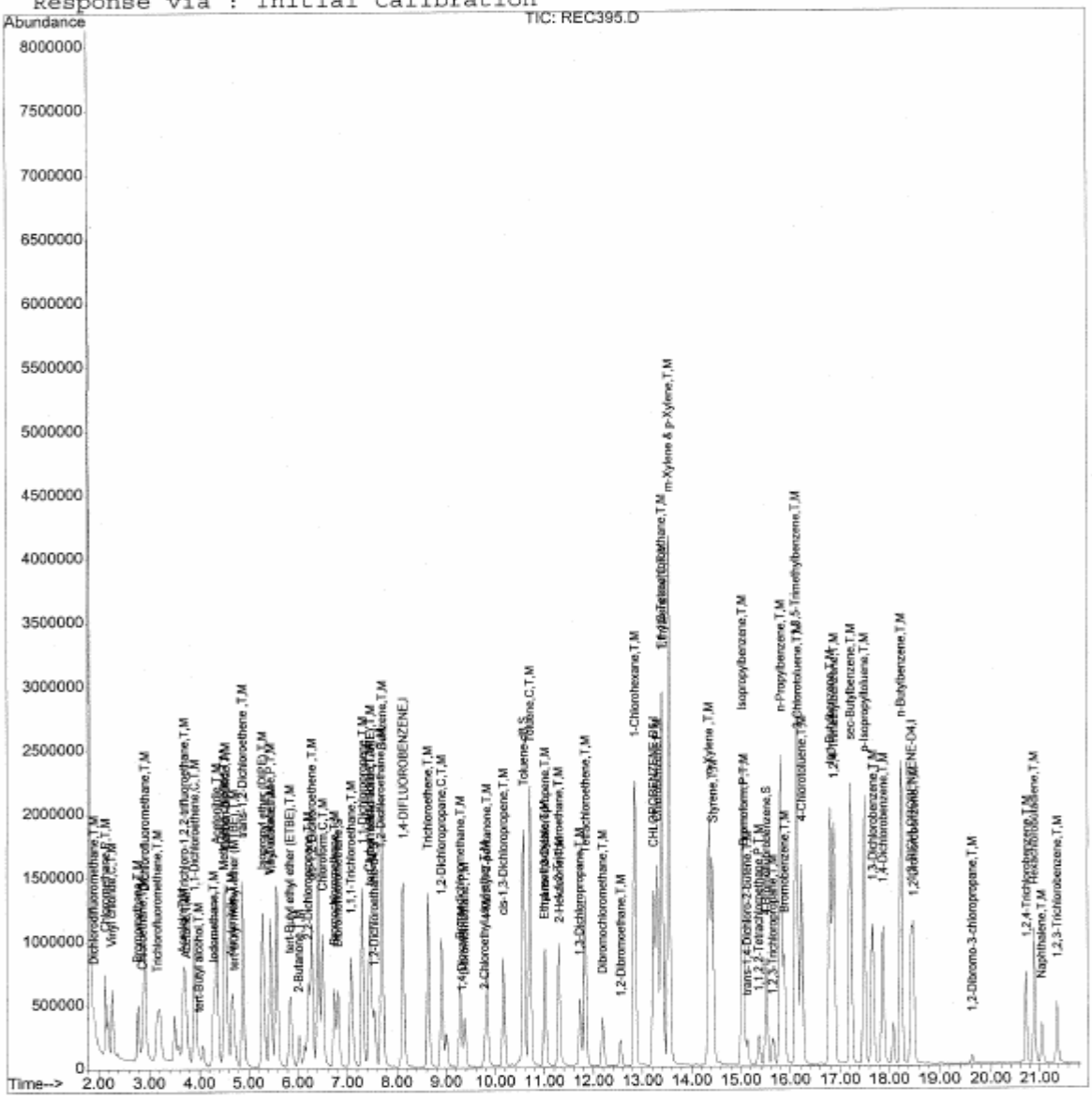


Figure 2: **TYPICAL CHROMATOGRAM**

Quantitation Report

Data File : D:\HPCHEM\1\DATA\11E25\REC395.D Vial: 2
Acq On : 25 May 2011 1:40 pm Operator: AS
Sample : IVO67E2402 10ppb Inst : TO67
Misc : 10ppb 8260/50ppb KET-ACR-ACN-TBA Multiplr: 1.00
MS Integration Params: LSCINT1.P
Quant Time: May 25 16:32 2011 Quant Results File: VO67E24.RES

Method : D:\HPCHEM\1\METHODS\VO67E24.M (RTE Integrator)
Title : METHOD 8260B 4.0
Last Update : Wed May 25 16:26:28 2011
Response via : Initial Calibration



REC395.D VO67E24.M Wed May 25 16:38:57 2011 Page 4

Figure 3: **TYPICAL ICAL SUMMARY**

INITIAL_CALIBRATION - RELATIVE_RESPONSE_FACTOR

Instrument ID :03
Beginning DateTime :03/12/13 07:57
Spike Units :PPB
IC File :RCB023

Column Spec :ZB-624 ID :0.25MM
Ending DateTime :03/12/13 12:58
HPChem Method :V003C12

M	IDX	Parameters	.3 07:57 RCB018	.5 08:31 RCB019	1 09:05 RCB020	2 09:39 RCB021	5 10:11 RCB022	10 10:45 RCB023	20 11:18 RCB024	30 11:52 RCB025	50 12:25 RCB026	100 12:58 RCB027	Av_RRF	%_RSD	Av_Rt_M
	1	1,4-DIFLUOROBENZENE	1	1	1	1	1	1	1	1	1	1	1	0	13.1167
	2	Chlorotrifluoroethylene	------	------	------	------	------	------	------	------	------	------	0.000	0.00	0.0000
	3	Dichlorodifluoromethane	0.312	0.311	0.326	0.327	0.302	0.284	0.298	0.281	0.271	0.265	0.298	7.35	4.0527
	4	Chloromethane	0.497	0.479	0.499	0.498	0.449	0.423	0.434	0.414	0.395	0.379	0.447	10.02	4.5516
	5	Vinyl chloride	0.295	0.288	0.315	0.308	0.286	0.271	0.286	0.277	0.270	0.273	0.287	5.39	4.8866
	6	2-Chloro-1,1,1-trifluoroethane	------	------	------	------	------	------	------	------	------	------	0.000	0.00	0.0000
	7	Bromomethane	0.307	0.306	0.320	0.311	0.275	0.265	0.267	0.247	0.210	------	0.279	12.91	5.7735
	8	Chloroethane	0.209	0.223	0.239	0.233	0.210	0.202	0.209	0.197	0.184	0.165	0.207	10.58	6.0466
	9	Dichlorofluoromethane	0.670	0.653	0.680	0.650	0.648	0.592	0.632	0.621	0.635	0.625	0.641	3.96	6.5916
	10	Trichlorofluoromethane	0.321	0.351	0.385	0.402	0.365	0.363	0.384	0.376	0.364	0.363	0.367	5.95	6.6749
5	11	Acrolein	0.012	0.010	0.011	0.011	0.012	0.013	0.013	0.013	0.013	0.013	0.012	9.23	7.7679
	12	1,1,2-Trichloro-1,2,2-trifluoroethane	0.214	0.199	0.207	0.193	0.190	0.173	0.180	0.176	0.185	0.180	0.190	7.16	7.8901
	13	1,1-Dichloroethene	0.613	0.612	0.620	0.579	0.570	0.534	0.548	0.523	0.543	0.514	0.566	6.97	7.9392
5	14	Acetone	------	------	0.045	0.035	0.032	0.031	0.029	0.027	0.029	0.028	0.032	18.18	8.1020
	15	Iodomethane	0.553	0.519	0.566	0.525	0.512	0.471	0.490	0.476	0.484	0.486	0.508	6.41	8.3338
	16	Carbon disulfide	1.259	1.220	1.291	1.367	1.204	1.194	1.218	1.179	1.183	1.151	1.227	5.20	8.4588
	17	Methyl acetate	------	------	------	------	------	------	------	------	------	------	0.000	0.00	0.0000
	18	Methylene chloride	------	------	0.548	0.484	0.450	0.419	0.422	0.397	0.422	0.401	0.443	11.51	9.0085
5	19	tert-Butyl alcohol	0.009	0.009	0.011	0.010	0.010	0.009	0.010	0.009	0.010	0.009	0.010	6.11	9.2093
	20	tert-Butyl methyl ether (MTBE)	0.430	0.405	0.440	0.420	0.409	0.367	0.381	0.371	0.404	0.374	0.400	6.43	9.4267
	21	trans-1,2-Dichloroethene	0.681	0.638	0.664	0.629	0.618	0.585	0.584	0.553	0.577	0.535	0.606	7.83	9.4834
5	22	Acrylonitrile	0.036	0.036	0.041	0.040	0.038	0.039	0.037	0.037	0.038	0.036	0.038	5.13	9.5161
	23	Isopropyl ether (DIPE)	1.387	1.263	1.335	1.285	1.234	1.171	1.151	1.110	1.121	1.024	1.208	9.29	10.2472
	24	1,1-Dichloroethane	0.768	0.739	0.749	0.730	0.706	0.676	0.668	0.644	0.653	0.605	0.694	7.62	10.2785
	25	Vinyl acetate	------	0.332	0.283	0.324	0.337	0.355	0.379	0.413	0.347	0.317	0.343	10.92	10.3003
	26	tert-Butyl ethyl ether (ETBE)	0.796	0.755	0.728	0.707	0.718	0.694	0.681	0.696	0.732	0.705	0.721	4.69	10.8667
	27	2,2-Dichloropropane	0.395	0.372	0.378	0.353	0.339	0.345	0.323	0.310	0.300	0.263	0.338	11.78	11.2345
5	28	2-Butanone	0.083	0.072	0.068	0.069	0.064	0.064	0.055	0.056	0.058	0.053	0.064	14.51	11.2761
	29	cis-1,2-Dichloroethene	0.469	0.440	0.442	0.430	0.415	0.405	0.384	0.379	0.390	0.365	0.412	8.05	11.2687
5	30	2-Butanol	------	------	------	------	------	------	------	------	------	------	0.000	0.00	0.0000
	31	Bromochloromethane	0.307	0.309	0.315	0.309	0.309	0.274	0.277	0.276	0.288	0.257	0.292	6.89	11.6797
	32	Tetrahydrofuran	------	------	0.048	0.045	0.037	0.034	0.033	0.036	0.036	0.034	0.038	13.93	11.7372
	33	Chloroform	0.705	0.690	0.687	0.691	0.672	0.607	0.605	0.606	0.620	0.579	0.646	7.24	11.7705
	34	Dibromofluoromethane	0.326	0.367	0.356	0.342	0.340	0.327	0.311	0.315	0.311	0.294	0.329	6.83	12.0326
	35	1,1,1-Trichloroethane	0.523	0.515	0.500	0.484	0.512	0.471	0.477	0.472	0.470	0.435	0.486	5.50	12.0386
	36	Cyclohexane	------	------	------	------	------	------	------	------	------	------	0.000	0.00	0.0000
	37	1,1-Dichloropropene	0.209	0.194	0.192	0.189	0.183	0.178	0.181	0.182	0.174	0.159	0.184	7.23	12.2738
	38	Carbon tetrachloride	0.467	0.452	0.446	0.423	0.441	0.427	0.429	0.426	0.426	0.403	0.434	4.16	12.2857
	39	1,2-Dichloroethane-d4	0.270	0.266	0.275	0.269	0.258	0.251	0.225	------	------	------	0.259	6.60	12.5581
	40	Benzene	1.724	1.622	1.501	1.421	1.497	1.348	1.382	1.392	1.393	------	1.476	8.50	12.6150
	41	tert-Amyl methyl ether (TAME)	0.100	0.103	0.107	0.100	0.109	0.098	0.097	0.102	0.105	0.098	0.102	4.11	12.6595
	42	1,2-Dichloroethane	0.299	0.308	0.290	0.294	0.303	0.287	0.279	0.277	0.287	0.256	0.288	5.20	12.6729
	43	Trichloroethene	0.428	0.404	0.405	0.389	0.419	0.382	0.382	0.375	0.400	0.366	0.395	4.98	13.5291
	44	Methylcyclohexane	------	------	------	------	------	------	------	------	------	------	0.000	0.00	0.0000
	45	1,2-Dichloropropane	0.387	0.398	0.375	0.376	0.407	0.377	0.381	0.361	0.376	0.342	0.378	4.76	13.8776
20	46	1,4-Dioxane	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	9.47	14.0562
	47	Dibromomethane	0.163	0.146	0.158	0.146	0.166	0.142	0.149	0.144	0.158	0.145	0.152	5.80	14.0935
	48	Bromodichloromethane	0.438	0.407	0.437	0.429	0.470	0.419	0.416	0.430	0.448	0.413	0.431	4.33	14.2617

Figure 3 (cont.): **TYPICAL ICAL SUMMARY**

	49	2-Chloroethyl vinyl ether	------	0.052	0.053	0.059	0.064	0.062	0.064	0.067	0.077	0.073	0.063	13.09	14.5606
	50	cis-1,3-Dichloropropene	0.511	0.509	0.493	0.489	0.553	0.509	0.513	0.510	0.537	0.492	0.512	3.91	14.8291
5	51	4-Methyl-2-pentanone	0.182	0.169	0.170	0.164	0.180	0.168	0.154	0.160	0.170	0.144	0.166	6.86	14.9541
	52	CHLOROBENZENE-D5	1	1	1	1	1	1	1	1	1	1	1	0	17.1073
	53	Toluene-d8	1.471	1.484	1.407	1.468	1.417	1.414	1.426	1.350	1.404	------	1.427	2.94	15.1712
	54	Toluene	1.975	1.822	1.622	1.764	1.814	1.638	1.732	1.646	1.710	------	1.747	6.46	15.2622
	55	Ethyl methacrylate	0.274	0.248	0.237	0.247	0.287	0.268	0.263	0.270	0.286	0.266	0.264	6.15	15.5111
	56	trans-1,3-Dichloropropene	0.383	0.368	0.358	0.376	0.420	0.392	0.406	0.404	0.429	0.403	0.394	5.77	15.5483
	57	1,1,2-Trichloroethane	0.222	0.209	0.198	0.196	0.213	0.196	0.195	0.206	0.217	0.201	0.205	4.79	15.8267
	58	Tetrachloroethene	0.438	0.389	0.368	0.385	0.382	0.355	0.371	0.366	0.383	0.352	0.379	6.42	15.9995
5	59	2-Hexanone	0.119	0.137	0.116	0.121	0.117	0.125	0.112	0.121	0.125	0.106	0.120	6.87	16.0248
	60	1,3-Dichloropropane	0.416	0.397	0.392	0.417	0.411	0.384	0.385	0.389	0.415	0.377	0.398	3.79	16.0531
	61	Dibromochloromethane	0.252	0.234	0.233	0.261	0.259	0.256	0.262	0.267	0.287	0.268	0.258	6.22	16.3941
	62	1,2-Dibromoethane	0.207	0.208	0.195	0.197	0.212	0.193	0.202	0.205	0.224	0.208	0.205	4.47	16.5847
	63	1-Chlorohexane	0.897	0.862	0.785	0.803	0.835	0.761	0.806	0.779	0.829	0.757	0.811	5.53	16.9495
	64	Chlorobenzene	1.152	1.065	0.937	0.954	0.990	0.958	0.962	0.991	1.007	0.911	0.993	7.08	17.1475
	65	Ethylbenzene	2.251	2.178	2.021	1.994	2.182	1.965	2.014	2.014	1.839	------	2.051	6.29	17.1930
	66	1,1,1,2-Tetrachloroethane	0.319	0.317	0.311	0.307	0.333	0.304	0.308	0.319	0.318	0.294	0.313	3.37	17.2264
2	67	m-Xylene & p-Xylene	1.713	1.626	1.515	1.469	1.497	1.439	1.490	1.387	------	------	1.517	6.90	17.3227
	68	o-Xylene	1.694	1.653	1.478	1.477	1.519	1.428	1.437	1.428	1.440	------	1.506	6.65	17.8581
	69	Styrene	1.028	1.012	0.971	0.933	1.027	0.971	0.981	0.978	0.998	0.874	0.977	4.73	17.8787
	70	1,2-DICHLOROBENZENE-D4	1	1	1	1	1	1	1	1	1	1	1	0	20.6245
	71	Bromoform	0.330	0.326	0.329	0.333	0.339	0.334	0.362	0.385	0.425	0.401	0.356	9.92	18.2613
	72	Isopropylbenzene	5.562	5.517	5.180	5.026	5.022	4.609	5.010	5.013	5.337	------	5.142	5.76	18.2767
	73	4-Bromofluorobenzene	1.603	1.546	1.384	1.356	1.335	1.258	1.308	1.333	1.419	1.324	1.386	7.83	18.5621
	74	1,1,2,2-Tetrachloroethane	0.746	0.756	0.716	0.675	0.696	0.618	0.665	0.707	0.749	0.704	0.703	6.08	18.7036
	75	trans-1,4-Dichloro-2-butene	0.187	0.180	0.176	0.186	0.198	0.177	0.197	0.205	0.223	0.201	0.193	7.69	18.7438
	76	n-Propylbenzene	8.651	8.030	7.852	7.211	7.500	6.892	7.592	7.274	------	------	7.625	7.21	18.7857
	77	1,2,3-Trichloropropane	0.177	0.170	0.160	0.153	0.158	0.140	0.147	0.149	0.157	0.138	0.155	7.93	18.8019
	78	Bromobenzene	1.165	1.071	1.055	1.014	1.052	0.931	0.983	1.022	1.109	1.010	1.041	6.31	18.8227
	79	1,3,5-Trimethylbenzene	4.694	4.553	4.332	4.080	4.256	3.733	4.106	4.10	4.355	3.299	4.151	9.67	18.9612
	80	2-Chlorotoluene	5.150	4.552	4.413	4.224	4.372	3.923	4.126	4.108	4.517	3.691	4.308	9.28	18.9999
	81	4-Chlorotoluene	4.441	3.866	3.719	3.646	3.838	3.506	3.604	3.728	4.047	3.296	3.769	8.31	19.1265
	82	tert-Butylbenzene	1.011	0.999	0.944	0.893	0.936	0.837	0.894	0.889	0.955	0.865	0.922	6.14	19.4213
	83	1,2,4-Trimethylbenzene	4.627	4.271	3.973	4.095	4.026	3.564	3.677	3.874	4.208	3.401	3.972	9.12	19.4794
	84	sec-Butylbenzene	6.845	6.467	6.537	6.165	6.138	5.570	5.934	5.860	6.013	------	6.170	6.32	19.6962
	85	p-Isopropyltoluene	4.982	4.670	4.483	4.208	4.407	4.011	4.268	4.060	4.454	------	4.394	6.93	19.8402
	86	1,3-Dichlorobenzene	2.358	2.168	2.115	2.002	2.047	1.927	2.037	1.985	2.204	2.008	2.085	6.15	19.9961
	87	1,4-Dichlorobenzene	2.379	2.019	1.958	1.903	1.942	1.756	1.873	1.837	2.027	1.854	1.955	8.73	20.1063
	88	n-Butylbenzene	5.550	4.899	5.079	4.650	4.817	4.593	4.886	4.619	4.980	------	4.897	6.06	20.3928
	89	1,2-Dichlorobenzene	1.816	1.636	1.646	1.560	1.576	1.448	1.505	1.471	1.578	1.464	1.570	7.08	20.6528
	90	1,2-Dibromo-3-chloropropane	0.063	0.069	0.073	0.072	0.076	0.068	0.073	0.073	0.078	0.074	0.072	6.04	21.7696
	91	1,2,4-Trichlorobenzene	0.972	0.844	0.786	0.841	0.831	0.798	0.763	0.733	0.817	0.766	0.815	8.11	23.0070
	92	Hexachlorobutadiene	------	0.740	0.722	0.700	0.678	0.604	0.560	0.503	0.559	0.527	0.621	14.43	23.1360
	93	Naphthalene	1.249	1.099	1.013	1.042	1.036	0.961	0.917	0.843	0.957	0.895	1.001	11.58	23.4983
	94	1,2,3-Trichlorobenzene	0.700	0.631	0.619	0.614	0.617	0.556	0.499	0.460	0.505	0.476	0.568	14.08	23.9168

Spike Amount = Nominal Amount * M
Ave_%RSD : 7.5 Max_%RSD : 18.2

Use Least Square Linear Regression with weighting factor of inverse concentration for comps with %_RSD > 15
Resp_Ratio = xo + x1 * Amt_Ratio

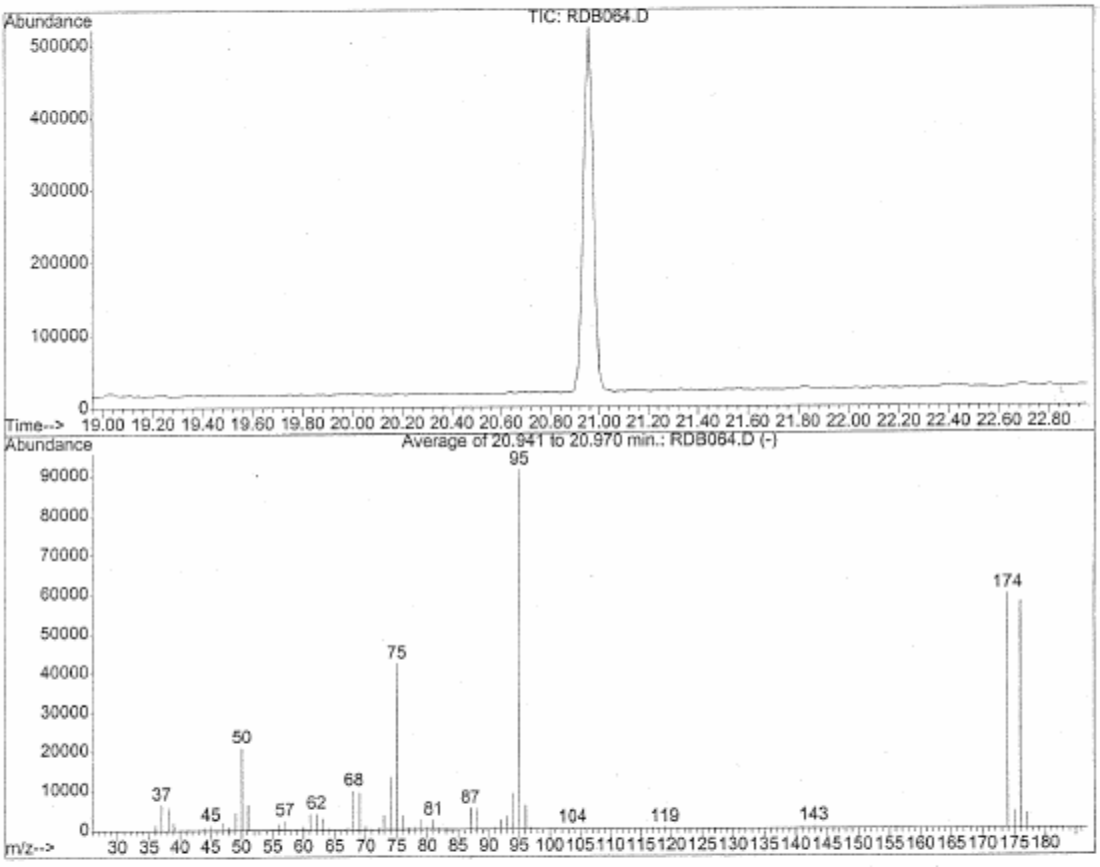
IDX	Parameter	x0	x1	CCF
14	Acetone	0.00872	0.02772	0.9996

Figure 4: TYPICAL INSTRUMENT PERFORMANCE CHECK (TUNING)

BFB

Data File : D:\HPCHEM\1\DATA\11D07\RDB064.D
Acq On : 7 Apr 2011 7:43 pm
Sample : BFB03D04
Misc : T/CHECK
MS Integration Params: 524INT.P
Method : D:\HPCHEM\1\METHODS\VO03D07.M (RTE Integrator)
Title : METHOD 8260 25mL

Vial: 2
Operator: MW
Inst : TO03
Multiplr: 1.00



AutoFind: Scans 1163, 1164, 1165; Background Corrected with Scan 1156

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
50	95	15	40	22.9	20916	PASS
75	95	30	60	46.4	42347	PASS
95	95	100	100	100.0	91301	PASS
96	95	5	9	6.6	6055	PASS
173	174	0.00	2	0.0	0	PASS
174	95	50	100	65.3	59619	PASS
175	174	5	9	7.4	4406	PASS
176	174	95	101	96.7	57635	PASS
177	176	5	9	6.7	3884	PASS

RDB064.D VO03D07.M Fri Apr 08 09:01:53 2011

Figure 5: TYPICAL INSTRUMENT PERFORMANCE CHECK (TUNING) SUMMARY

5A
VOLATILE ORGANIC INSTRUMENT PERFORMANCE CHECK
BROMOFLUOROBENZENE (BFB)

Lab Name: EMAX Inc Contract: CLEAN WATER PROJECT
Lab Code: EMXT Case No.: SAS No.: SDG No.: YYMNNN
Lab File ID: RDC521 BFB Injection Date : 04/29/14
Instrument ID: 67 BFB Injection Time : 09:52
GC Column:RTX502.2ID:0.25mm (mm) Heated Purge: (Y/N) N

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% of mass 95	20.69
75	30.0 - 60.0% of mass 95	45.82
95	Base peak, 100% relative abundance	100.00
96	5.0 - 9.0% of mass 95	6.39
173	Less than 2.0% of mass 174	0.00(0.0)1
174	Greater than 50% of mass 95	70.43
175	5.0 - 9.0% of mass 174	5.04(7.2)1
176	95.0 - 101.0% of mass 174	67.25(95.5)1
177	5.0 - 9.0% of mass 176	4.57(6.8)2

1-Value is % mass 174 2-Value is % mass 176

THIS CHECK APPLIES TO THE FOLLOWING SAMPLES, MS, MSD,BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
1	VSTD010	CVO67D0914	RDC523	04/29/14	11:14
2	MBLK1W	VO67D21B	RDC527	04/29/14	13:15
3	LCS1W	VO67D21L	RDC524	04/29/14	11:44
4	LCD1W	VO67D21C	RDC525	04/29/14	12:15
5	XXX-59XX001	MNNN-02	RDC534	04/29/14	16:55
6	XXX-59XX001MS	MNNN-02M	RDC535	04/29/14	17:26
7	XXX-59XX001MSD	MNNN-02S	RDC536	04/29/14	18:57

Figure 6: TYPICAL INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

8A
VOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name : EMAX INC. Project : CLEAN WATER PROJECT
Lab Code : EMXT SDG No. : YYMNNN
Lab File ID : RDC168 Date Analyzed : 04/09/14
Instrument ID: 67 Time Analyzed : 18:54
GC Column : RTX502.2ID:0.25mm (mm) Heated Purge : No

		IS1(DBF)		IS2(CBZ)		IS3(DCB)	
		AREA #	RT #	AREA #	RT #	AREA #	RT #
	12 HOUR STD	1321331	8.08	1022177	13.17	321226	18.40
	UPPER LIMIT	2642662	8.58	2044354	13.67	642452	18.90
	LOWER LIMIT	660666	7.58	511089	12.67	160613	17.90
	SAMPLE ID						
1	VSTD010	1155410	8.07	908594	13.15	276831	18.38
2	MBLK1W	1259632	8.07	956984	13.15	261376	18.38
3	LCS1W	1216852	8.07	957974	13.15	290568	18.38
4	LCD1W	1262335	8.07	983468	13.15	300886	18.38
5	XXX-59GW001	1202358	8.07	934767	13.16	277693	18.38
6	XXX-59GW001MS	1216852	8.07	957974	13.17	290568	18.38
7	XXX-59GW001MSD	1262335	8.07	983468	13.17	300886	18.38

IS1 (DFB) = 1,4-Difluorobenzene
IS2 (CBZ) = Chlorobenzene-d5
IS3 (DCB) = 1,2-Dichlorobenzene-d4

AREA UPPER LIMIT = + 100% of internal standard area
AREA LOWER LIMIT = - 50% of internal standard area
RT UPPER LIMIT = + 0.5 minutes (30 sec) of internal standard RT
RT LOWER LIMIT = - 0.5 minutes (30 sec) of internal standard RT

Column used to flag internal standard area values with an asterisk
* Values outside of QC limits.

Figure 7: TYPICAL SAMPLE RESULT SUMMARY

METHOD SW5030C/8260B
VOLATILE ORGANICS BY GC/MS

Client : XYZ INC.	Date Collected: 04/28/14
Project : CLEAN WATER PROJECT	Date Received: 04/29/14
Batch No. : YYMNNN	Date Extracted: 04/29/14 16:55
Sample ID: XX-59XX001	Date Analyzed: 04/29/14 16:55
Lab Samp ID: MNNN-02	Dilution Factor: 1
Lab File ID: RDC534	Matrix : WATER
Ext Btch ID: VO67D21	% Moisture : NA
Calib. Ref.: RDC168	Instrument ID : 67

PARAMETERS	RESULTS (ug/L)	LOQ (ug/L)	DL (ug/L)	LOD (ug/L)
BENZENE	0.11J	1.0	0.10	0.20
BROMODICHLOROMETHANE	ND	1.0	0.10	0.20
BROMOFORM	1.5	1.0	0.15	0.30
BROMOMETHANE	ND	1.0	0.16	0.30
CARBON TETRACHLORIDE	ND	1.0	0.10	0.20
CHLOROBENZENE	ND	1.0	0.10	0.20
CHLOROETHANE	ND	1.0	0.27	0.30
CHLOROFORM	ND	1.0	0.10	0.20
CHLOROMETHANE	ND	2.0	0.15	0.30
DIBROMOCHLOROMETHANE	0.20J	1.0	0.10	0.20
1,2-DICHLOROBENZENE	ND	1.0	0.10	0.20
1,3-DICHLOROBENZENE	ND	1.0	0.11	0.20
1,4-DICHLOROBENZENE	ND	1.0	0.10	0.20
DICHLORODIFLUOROMETHANE (FREON 12)	ND	1.0	0.15	0.30
1,1-DICHLOROETHANE	ND	1.0	0.10	0.20
1,2-DICHLOROETHANE	ND	1.0	0.10	0.20
1,1-DICHLOROETHENE	ND	1.0	0.10	0.20
1,2-DICHLOROETHENE (TOTAL)	0.30J	1.0	0.10	0.20
1,2-DICHLOROPROPANE	ND	1.0	0.10	0.20
TRANS-1,3-DICHLOROPROPENE	ND	1.0	0.11	0.20
CIS-1,3-DICHLOROPROPENE	ND	1.0	0.10	0.20
ETHYLBENZENE	ND	1.0	0.10	0.20
METHYLENE CHLORIDE	ND	5.0	0.25	0.50
1,1,2,2-TETRACHLOROETHANE	ND	1.0	0.11	0.20
TETRACHLOROETHENE	0.19J	1.0	0.15	0.20
TOLUENE	62	1.0	0.10	0.20
1,1,1-TRICHLOROETHANE	ND	1.0	0.10	0.20
1,1,2-TRICHLOROETHANE	ND	1.0	0.10	0.20
TRICHLOROFLUOROMETHANE (FREON 11)	ND	1.0	0.15	0.30
TRICHLOROETHENE	14	1.0	0.10	0.20
VINYL CHLORIDE	ND	2.0	0.12	0.20
XYLENES (TOTAL)	ND	1.0	0.10	0.50

SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
1,2-DICHLOROETHANE-D4	8.44	10.00	84.4	70-120
4-BROMOFLUOROBENZENE	9.41	10.00	94.1	75-120
TOLUENE-D8	10.3	10.00	103	85-120
DIBROMOFLUOROMETHANE	9.95	10.00	99.5	85-115

Figure 8: TYPICAL LCS/LCSD SUMMARY

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT:	XYZ INC.				
PROJECT:	CLEAN WATER PROJECT				
BATCH NO.:	YYMNNN				
METHOD:	SW5030C/8260B				
MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	MBLK1W				
LAB SAMP ID:	VO67D21B	VO67D21L	VO67D21C		
LAB FILE ID:	RDC527	RDC524	RDC525		
DATE EXTRACTED:	04/29/1413:15	04/29/1411:44	04/29/1412:15	DATE COLLECTED:	NA
DATE ANALYZED:	04/29/1413:15	04/29/1411:44	04/29/1412:15	DATE RECEIVED:	04/29/14
PREP. BATCH:	VO67D21	VO67D21	VO67D21		
CALIB. REF:	RDC168	RDC168	RDC168		

ACCESSION:

PARAMETER	BLNK RSLT (ug/L)	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Benzene	ND	10.0	9.32	93	10.0	9.17	92	2	80-120	30
Bromodichloromethane	ND	10.0	9.65	97	10.0	9.37	94	3	75-120	30
Bromoform	ND	10.0	10.1	101	10.0	9.80	98	3	70-130	30
Bromomethane	ND	10.0	10.6	106	10.0	9.80	98	7	30-145	30
Carbon Tetrachloride	ND	10.0	8.35	83	10.0	8.20	82	2	65-140	30
Chlorobenzene	ND	10.0	9.78	98	10.0	9.68	97	1	80-120	30
Chloroethane	ND	10.0	11.0	110	10.0	10.1	101	8	60-135	30
Chloroform	ND	10.0	9.84	98	10.0	9.56	96	3	65-135	30
Chloromethane	ND	10.0	10.2	102	10.0	9.58	96	7	40-125	30
Dibromochloromethane	ND	10.0	9.64	96	10.0	9.49	95	2	60-135	30
1,2-Dichlorobenzene	ND	10.0	10.3	103	10.0	10.1	101	2	70-120	30
1,3-Dichlorobenzene	ND	10.0	10.1	101	10.0	9.91	99	2	75-125	30
1,4-Dichlorobenzene	ND	10.0	10.0	100	10.0	9.87	99	1	75-125	30
Dichlorodifluoromethane (Freon 12)	ND	10.0	9.93	99	10.0	9.39	94	6	30-155	30
1,1-Dichloroethane	ND	10.0	9.27	93	10.0	8.99	90	3	70-135	30
1,2-Dichloroethane	ND	10.0	7.97	80	10.0	7.88	79	1	70-130	30
1,1-Dichloroethene	ND	10.0	7.58	76	10.0	7.41	74	2	70-130	30
1,2-Dichloroethene (Total)	ND	20.0	17.1	85	20.0	16.6	83	3	70-125	30
1,2-Dichloropropane	ND	10.0	9.90	99	10.0	9.70	97	2	75-125	30
Trans-1,3-Dichloropropene	ND	10.0	8.39	84	10.0	8.49	85	1	55-140	30
cis-1,3-Dichloropropene	ND	10.0	9.23	92	10.0	8.93	89	3	70-130	30
Ethylbenzene	ND	10.0	9.84	98	10.0	9.67	97	2	75-125	30
Methylene Chloride	ND	10.0	8.28	83	10.0	8.40	84	1	55-140	30
1,1,2,2-Tetrachloroethane	ND	10.0	10.5	105	10.0	10.3	103	2	65-130	30
Tetrachloroethene	ND	10.0	9.31	93	10.0	9.20	92	1	45-150	30
Toluene	ND	10.0	9.57	96	10.0	9.47	95	1	75-120	30
1,1,1-Trichloroethane	ND	10.0	8.74	87	10.0	8.56	86	2	65-130	30
1,1,2-Trichloroethane	ND	10.0	10.3	103	10.0	10.3	103	0	75-125	30
Trichlorofluoromethane (Freon 11)	ND	10.0	10.8	108	10.0	9.93	99	9	60-145	30
Trichloroethene	ND	10.0	9.46	95	10.0	9.19	92	3	70-125	30
Vinyl Chloride	ND	10.0	10.3	103	10.0	9.61	96	7	50-145	30
Xylenes (Total)	ND	30.0	28.5	95	30.0	28.0	93	2	80-120	30

SURROGATE PARAMETER	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	QC LIMIT (%)
1,2-Dichloroethane-d4	10.0	8.20	82	10.0	8.24	82	70-120
4-Bromofluorobenzene	10.0	9.69	97	10.0	9.70	97	75-120
Toluene-d8	10.0	10.4	104	10.0	10.4	104	85-120
Dibromofluoromethane	10.0	9.92	99	10.0	9.85	98	85-115

Figure 9: TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ INC.
PROJECT: CLEAN WATER PROJECT
BATCH NO.: YYMNNN
METHOD: SW5030C/8260B

MATRIX: WATER % MOISTURE: NA

DILUTION FACTOR:	1	1	1		
SAMPLE ID:	XXX-59XX001	XXX-59XX001MS	XXX-59XX001MSD		
LAB SAMP ID:	MNNN-02	MNNN-02M	MNNN-02S		
LAB FILE ID:	RDC534	RDC535	RDC536		
DATE EXTRACTED:	04/29/1416:55	04/29/1417:26	04/29/1418:15	DATE COLLECTED:	NA
DATE ANALYZED:	04/29/1416:55	04/29/1417:26	04/29/1418:15	DATE RECEIVED:	04/29/14
PREP. BATCH:	VO67D21	VO67D21	VO67D21		
CALIB. REF:	RDC168	RDC168	RDC168		

ACCESSION:

PARAMETER	BLNK RSLT (ug/L)	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Benzene	ND	10.0	9.32	93	10.0	9.17	92	2	80-120	30
Bromodichloromethane	ND	10.0	9.65	97	10.0	9.37	94	3	75-120	30
Bromoform	ND	10.0	10.1	101	10.0	9.80	98	3	70-130	30
Bromomethane	ND	10.0	10.6	106	10.0	9.80	98	7	30-145	30
Carbon Tetrachloride	ND	10.0	8.35	83	10.0	8.20	82	2	65-140	30
Chlorobenzene	ND	10.0	9.78	98	10.0	9.68	97	1	80-120	30
Chloroethane	ND	10.0	11.0	110	10.0	10.1	101	8	60-135	30
Chloroform	ND	10.0	9.84	98	10.0	9.56	96	3	65-135	30
Chloromethane	ND	10.0	10.2	102	10.0	9.58	96	7	40-125	30
Dibromochloromethane	ND	10.0	9.64	96	10.0	9.49	95	2	60-135	30
1,2-Dichlorobenzene	ND	10.0	10.3	103	10.0	10.1	101	2	70-120	30
1,3-Dichlorobenzene	ND	10.0	10.1	101	10.0	9.91	99	2	75-125	30
1,4-Dichlorobenzene	ND	10.0	10.0	100	10.0	9.87	99	1	75-125	30
Dichlorodifluoromethane (Freon 12)	ND	10.0	9.93	99	10.0	9.39	94	6	30-155	30
1,1-Dichloroethane	ND	10.0	9.27	93	10.0	8.99	90	3	70-135	30
1,2-Dichloroethane	ND	10.0	7.97	80	10.0	7.88	79	1	70-130	30
1,1-Dichloroethene	ND	10.0	7.58	76	10.0	7.41	74	2	70-130	30
1,2-Dichloroethene (Total)	ND	20.0	17.1	85	20.0	16.6	83	3	70-125	30
1,2-Dichloropropane	ND	10.0	9.90	99	10.0	9.70	97	2	75-125	30
Trans-1,3-Dichloropropene	ND	10.0	8.39	84	10.0	8.49	85	1	55-140	30
cis-1,3-Dichloropropene	ND	10.0	9.23	92	10.0	8.93	89	3	70-130	30
Ethylbenzene	ND	10.0	9.84	98	10.0	9.67	97	2	75-125	30
Methylene Chloride	ND	10.0	8.28	83	10.0	8.40	84	1	55-140	30
1,1,2,2-Tetrachloroethane	ND	10.0	10.5	105	10.0	10.3	103	2	65-130	30
Tetrachloroethene	ND	10.0	9.31	93	10.0	9.20	92	1	45-150	30
Toluene	ND	10.0	9.57	96	10.0	9.47	95	1	75-120	30
1,1,1-Trichloroethane	ND	10.0	8.74	87	10.0	8.56	86	2	65-130	30
1,1,2-Trichloroethane	ND	10.0	10.3	103	10.0	10.3	103	0	75-125	30
Trichlorofluoromethane (Freon 11)	ND	10.0	10.8	108	10.0	9.93	99	9	60-145	30
Trichloroethene	ND	10.0	9.46	95	10.0	9.19	92	3	70-125	30
Vinyl Chloride	ND	10.0	10.3	103	10.0	9.61	96	7	50-145	30
Xylenes (Total)	ND	30.0	28.5	95	30.0	28.0	93	2	80-120	30

SURROGATE PARAMETER	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	QC LIMIT (%)
1,2-Dichloroethane-d4	10.0	8.20	82	10.0	8.24	82	70-120
4-Bromofluorobenzene	10.0	9.69	97	10.0	9.70	97	75-120
Toluene-d8	10.0	10.4	104	10.0	10.4	104	85-120
Dibromofluoromethane	10.0	9.92	99	10.0	9.85	98	85-115

Figure 10: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ INC.

Project : CLEAN WATER PROJECT

SDG : YYDNNN

METHOD SW5030C/8260B
VOLATILE ORGANICS BY GC/MS

A total of two (2) water samples were received on 04/29/14 for Volatile Organics by GC/MS analysis, Method 5030C/8260B in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods and Project QAPP Clean Water Project.

Holding Time
Samples were analyzed within the prescribed holding time.

Instrument Performance and Calibration
Instrument tune check was performed prior to calibration. Instrument mass ratios were within specification. Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using secondary source (ICV). Continuing calibration (CCV) was carried on at a frequency required by the project. All project calibration requirements were satisfied. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank
Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Results were compliant to project requirement.

Lab Control Sample
A set of LCS/LCD was analyzed with the samples in this SDG.
Percent recoveries for VO67D21L/C were all within QC limits.

Matrix QC Sample
Matrix QC sample was analyzed at the frequency prescribed by the project.
Percent recoveries and RPDs for MNNN-02M/S were within project QC limits.

Surrogate
Surrogates were added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis
Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in Table 5	Retune instrument and verify		
Multi point Initial Calibration(ICAL) minimum of 5 points	Initially; as needed	SPCCs : RF $\geq$ 0.1 for Bromoform, Chloromethane and 1,1-Dichloroethane RF $\geq$ 0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane CCCs: RSD ≤ 30% for the following analytes: Chloroform, 1,1-DCE, 1,2-DCP, Ethylbenzene, Toluene and Vinyl Chloride. 1.) if RRF is applied, then RSD ≤ 15% 2.) If 1st order is applied, then r ≥ 0.995 with min 5 pt ICAL 3.) If 2nd order is applied, then r ≥ 0.99 with min 6 pt ICAL	Check for outliers. Otherwise, optimize the instrument then repeat initial calibration.		
Initial calibration verification (ICV)	After initial calibration	All analytes within ± 20% of expected value except for the following compounds due to erratic chromatographic behavior: Bromomethane, Chloroethane, Chloromethane, Dichlorodifluoromethane but must be within $\pm$ 35% of expected value.	Verify second source standard. Prepare fresh standard and rerun ICV. If that fails, Optimize instrument and repeat ICAL.		
Evaluation of relative retention times (RRT)	Each sample	Within ± 0.06 RRT units	Correct the problem then reanalyze all samples analyzed since the last retention time check		
Continuing Calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time	SPCCs: Min. RF same as ICAL CCC : %Diff $\leq$ 20% (when using RFs) or drift (when using least squares regression or non-linear calibration)	Correct the problem then repeat initial calibration		
Internal Standard (IS)	All samples	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning		
Method blank (MB)	One per preparation batch	No analytes detected > ½ LOQ	Rule out instrument contamination by re-analyzing the MB. If problem persist refer to PSR. In the absence of PSR, report NDs and results >10X of the MB concentration. Otherwise, cure contamination source, re-prep and re-analyze method blank and all associated samples.		
LCS	One LCS per preparation	Within project QC Limits	Re-prep and re-analyze the LCS and all associated samples		
MS/MSD	One MS/MSD per every 20 project samples per matrix	Within project QC Limits	Check if sample was properly spiked. If indicative of matrix interference, discuss in case narrative, otherwise re-prep and re-analyze the sample		
Surrogate	Every Sample, MB, LCS, MS/MSD, DCC	Within project QC Limits	Correct the problem then re-analyze		
Comments: This QCP is applicable in the absence of the PSR Report values between LOD and LOQ. Refer to PSR for Flagging Criteria.			Reviewed by: Date:		

Appendix 2: DEMONSTRATION OF CAPABILITY for 25 ml

DEMONSTRATION OF CAPABILITY
METHOD: SW 8260

Unit: µg/L
Sample Amount(ml): 25
Sample Purge(ml): 25

Date Analyzed: 01/31 & 02/01/12
Analyzed by: D. Nguyen

PARAMETER	RAY133	RAY134	RBY004	RBY005	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
	VOF5A10L	VOF5A10C	VOF5B01L	VOF5B01C							
Acetone	46.2	49.8	45.0	49.4	50	47.6	95	2.378	5	60 - 130	Passed
Acetonitrile	83.4	86.1	88.7	83.0	100	85.3	85	2.632	3	50 - 130	Passed
Acrolein	47.0	50.2	42.0	48.0	50	46.8	94	3.502	7	10 - 160	Passed
Acrylonitrile	50.0	52.1	45.0	51.3	50	49.6	99	3.188	6	60 - 150	Passed
Benzene	9.39	10.0	9.71	9.62	10	9.69	97	0.255	3	70 - 130	Passed
Bromobenzene	9.65	10.4	9.85	10.1	10	10.0	100	0.344	3	70 - 130	Passed
Bromochloromethane	9.08	9.78	9.18	9.48	10	9.4	94	0.315	3	70 - 130	Passed
Bromodichloromethane	9.58	10.1	9.81	10.0	10	9.9	99	0.239	2	70 - 130	Passed
Bromoform	9.70	10.3	9.00	10.0	10	9.76	98	0.568	6	60 - 130	Passed
Bromomethane	9.49	10.2	10.6	10.3	10	10.14	101	0.465	5	50 - 140	Passed
tert-Butyl alcohol	51.8	54.4	42.6	48.4	50	49.3	99	5.096	10	50 - 150	Passed
2-Butanone (MEK)	49.3	51.8	42.1	50.5	50	48.4	97	4.344	9	70 - 130	Passed
n-Butylbenzene	10.0	10.8	11.1	10.4	10	10.6	106	0.472	4	70 - 130	Passed
sec-Butylbenzene	9.94	10.8	10.8	10.5	10	10.5	105	0.397	4	70 - 130	Passed
tert-Butylbenzene	8.78	9.46	9.31	8.95	10	9.1	91	0.315	3	70 - 130	Passed
Carbon disulfide	9.24	10.2	8.45	8.74	10	9.15	92	0.755	8	50 - 130	Passed
Carbon tetrachloride	9.24	10.0	10.0	9.65	10	9.7	97	0.343	4	60 - 130	Passed
Chlorobenzene	9.08	9.68	9.39	9.22	10	9.3	93	0.257	3	70 - 130	Passed
2-Chloroethyl vinyl ether	9.55	9.91	8.16	9.38	10	9.3	93	0.760	8	10 - 160	Passed
Chloroethane	9.11	9.81	9.89	10.1	10	9.72	97	0.414	4	60 - 130	Passed
Chloroform	9.02	9.65	9.54	9.39	10	9.4	94	0.272	3	70 - 130	Passed
1-Chlorohexane	8.77	9.38	9.10	8.85	10	9.0	90	0.278	3	70 - 130	Passed
Chloromethane	9.12	9.52	9.39	9.53	10	9.39	94	0.191	2	50 - 130	Passed
2-Chlorotoluene	9.86	10.7	10.1	10.7	10	10.3	103	0.433	4	70 - 130	Passed
4-Chlorotoluene	9.70	10.4	11.1	9.9	10	10.3	103	0.611	6	70 - 130	Passed
Isopropyl ether (DIPE)	9.76	10.4	9.33	9.68	10	9.8	98	0.435	4	70 - 130	Passed
Dibromochloromethane	9.85	10.4	9.62	10.2	10	10.0	100	0.346	3	70 - 130	Passed
1,2-Dibromo-3-chloropropane	9.14	9.80	8.25	8.90	10	9.02	90	0.641	7	60 - 130	Passed
1,2-Dibromoethane	9.90	10.5	9.39	10.2	10	10.0	100	0.487	5	70 - 130	Passed
Dibromomethane	9.82	10.3	8.87	9.58	10	9.6	96	0.584	6	70 - 130	Passed
1,1-Dichloroethane	9.15	9.83	9.42	9.31	10	9.4	94	0.292	3	70 - 130	Passed
1,2-Dichloroethane	9.45	10.0	9.52	9.89	10	9.7	97	0.261	3	70 - 130	Passed
1,2-Dichlorobenzene	8.98	9.68	9.33	9.26	10	9.3	93	0.290	3	70 - 130	Passed
1,3-Dichlorobenzene	9.61	10.4	9.92	9.88	10	9.9	99	0.327	3	70 - 130	Passed
trans-1,4-Dichloro-2-Butene	10.6	11.5	9.58	10.8	10	10.6	106	0.812	8	50 - 140	Passed
1,4-Dichlorobenzene	9.82	10.6	10.1	10.1	10	10.2	102	0.340	3	70 - 130	Passed
Dichlorodifluoromethane	9.14	10.0	9.22	9.67	10	9.50	95	0.383	4	50 - 140	Passed
1,1-Dichloroethene	9.17	9.8	9.40	9.27	10	9.4	94	0.289	3	60 - 130	Passed
cis-1,2-Dichloroethene	9.40	10.1	9.71	9.60	10	9.7	97	0.285	3	70 - 130	Passed
trans-1,2-Dichloroethene	9.27	9.83	9.34	9.21	10	9.4	94	0.282	3	60 - 130	Passed
Dichlorofluoromethane	8.27	9.12	9.84	9.25	10	9.12	91	0.646	7	70 - 130	Passed
1,1-Dichloropropene	9.42	10.2	9.79	9.72	10	9.8	98	0.319	3	70 - 130	Passed
1,2-Dichloropropane	9.43	10.0	9.48	9.68	10	9.6	96	0.256	3	70 - 130	Passed
1,3-Dichloropropane	9.82	10.4	9.54	10.1	10	10.0	100	0.359	4	70 - 130	Passed

Appendix 2 (cont.): DEMONSTRATION OF CAPABILITY for 25 ml

DEMONSTRATION OF CAPABILITY
METHOD: SW 8260

Unit: µg/L
Sample Amount(ml): 25
Sample Purge(ml): 25

Date Analyzed: 01/31 & 02/01/12
Analyzed by: D. Nguyen

PARAMETER	RAY133	RAY134	RBY004	RBY005	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
	VOF5A10L	VOF5A10C	VOF5B01L	VOF5B01C							
2,2-Dichloropropane	9.07	10.4	10.2	9.65	10	9.8	98	0.598	6	60 - 140	Passed
cis-1,3-Dichloropropene	10.0	10.7	10.0	10.3	10	10.3	103	0.339	3	70 - 130	Passed
trans-1,3-Dichloropropene	9.30	9.75	9.06	9.42	10	9.4	94	0.288	3	70 - 130	Passed
tert-Butyl ethyl ether (ETB	11.1	11.8	10.1	10.8	10	11.0	110	0.685	6	70 - 130	Passed
Ethyl Methacrylate	9.30	9.68	8.15	9.21	10	9.1	91	0.656	7	70 - 130	Passed
Ethylbenzene	9.47	10.1	10.0	9.68	10	9.8	98	0.300	3	70 - 130	Passed
2-Hexanone (MBK)	50.6	52.3	42.7	50.6	50	49.0	98	4.298	9	70 - 140	Passed
Hexachlorobutadiene	8.21	8.92	8.99	8.08	10	8.6	86	0.469	5	70 - 130	Passed
Iodomethane	9.16	10.0	9.31	9.21	10	9.43	94	0.417	4	50 - 150	Passed
Isopropylbenzene	10.4	11.2	11.0	10.8	10	10.9	109	0.380	3	70 - 130	Passed
p-Isopropyltoluene	10.1	11.0	11.0	10.4	10	10.6	106	0.423	4	70 - 130	Passed
Methylene Chloride	8.56	9.09	8.74	8.87	10	8.8	88	0.225	3	60 - 130	Passed
4-Methyl-2-pentanone (MIBK)	52.9	54.7	45.5	53.6	50	51.7	103	4.184	8	70 - 130	Passed
tert-Butyl methyl ether	10.1	10.7	8.99	10.1	10	10.0	100	0.723	7	60 - 130	Passed
Naphthalene	10.3	10.7	9.41	9.88	10	10.07	101	0.565	6	50 - 140	Passed
n-Propylbenzene	10.6	11.4	11.2	11.1	10	11.1	111	0.369	3	70 - 130	Passed
Styrene	9.63	10.4	10.0	9.8	10	10.0	100	0.311	3	70 - 130	Passed
tert-Amyl methyl ether (TAME)	11.3	12.0	10.2	11.1	10	11.1	111	0.747	7	60 - 140	Passed
1,1,1,2-Tetrachloroethane	9.32	9.91	9.59	9.54	10	9.6	96	0.246	3	70 - 130	Passed
1,1,2,2-Tetrachloroethane	9.92	10.6	9.52	10.5	10	10.1	101	0.501	5	60 - 130	Passed
Tetrachloroethene	9.08	9.77	9.50	9.01	10	9.3	93	0.356	4	60 - 130	Passed
Toluene	9.68	10.3	10.1	9.94	10	10.0	100	0.262	3	70 - 130	Passed
1,1,1-Trichloroethane	8.83	10.0	10.2	9.52	10	9.6	96	0.591	6	70 - 130	Passed
1,1,2-Trichloroethane	9.67	10.1	9.45	10.1	10	9.8	98	0.346	4	70 - 130	Passed
1,2,3-Trichlorobenzene	8.77	9.14	8.46	8.40	10	8.7	87	0.339	4	60 - 130	Passed
1,2,4-Trichlorobenzene	9.04	9.63	8.93	8.60	10	9.1	91	0.432	5	60 - 140	Passed
Trichloroethene	9.21	9.93	9.42	9.29	10	9.5	95	0.326	3	70 - 130	Passed
Trichlorofluoromethane	9.06	9.62	10.1	10.3	10	9.76	98	0.544	6	60 - 140	Passed
1,2,3-Trichloropropane	9.93	10.5	9.45	10.6	10	10.1	101	0.524	5	70 - 130	Passed
1,1,2-Trichloro1,2,2-trifluoroethane	8.70	9.49	9.45	9.05	10	9.2	92	0.376	4	60 - 150	Passed
1,2,4-Trimethylbenzene	9.8	10.6	10.6	10.2	10	10.3	103	0.374	4	70 - 130	Passed
1,3,5-Trimethylbenzene	9.9	10.7	10.5	10.2	10	10.3	103	0.335	3	70 - 130	Passed
Vinyl Acetate	10.9	11.0	9.68	11.1	10	10.66	107	0.662	6	40 - 150	Passed
Vinyl Chloride	8.26	8.56	9.02	9.01	10	8.71	87	0.372	4	60 - 130	Passed
m-Xylene & p-xylene	19.2	20.6	20.4	19.6	20	19.9	100	0.633	3	60 - 140	Passed
o-Xylene	8.80	9.41	9.18	8.93	10	9.1	91	0.270	3	70 - 130	Passed
1,2-Dichloroethane-d4	10.0	10.0	9.9	10.3	10	10.05	101	0.200	2	70 - 130	Passed
Toluene-d8	10.3	10.3	10.4	10.3	10	10.32	103	0.046	0.5	70 - 130	Passed
4-Bromofluorobenzene	10.6	10.6	10.5	10.9	10	10.68	107	0.189	2	70 - 130	Passed
Dibromofluoromethane	10.0	9.93	10.1	10.2	10	10.06	101	0.106	1	70 - 130	Passed

Appendix 3: DEMONSTRATION OF CAPABILITY for 5 ml

DEMONSTRATION OF CAPABILITY
METHOD: SW 8260

Unit: µg/L
Sample Amount(ml): 5
Sample Purge(ml): 5

Date Analyzed: 01/07/13 & 01/08/13
Analyzed by: C. Mendoza

PARAMETER	RAN044 VSF4A07L	RAN045 VSF4A07C	RAN058 VSF4A08L	RAN059 VSF4A08C	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
Acetone	261	261	275	272	250	267	107	7.569	3	60 130	Passed
Acetonitrile	540	546	510	533	500	532	106	15.685	3	30 160	Passed
Acrolein	334	330	337	325	250	331	133	5.396	2	30 160	Passed
Acrylonitrile	269	270	278	271	250	272	109	4.119	2	70 130	Passed
Benzene	51.0	52.3	48.2	49.6	50	50.3	101	1.744	3	70 130	Passed
Bromobenzene	46.8	48.0	45.4	46.2	50	46.6	93	1.104	2	70 130	Passed
Bromochloromethane	51.7	52.2	50.7	51.2	50	51.5	103	0.611	1	70 130	Passed
Bromodichloromethane	49.5	50.8	48.4	49.2	50	49.5	99	1.022	2	70 130	Passed
Bromoform	47.8	48.6	45.6	47.5	50	47.4	95	1.282	3	70 130	Passed
Bromomethane	54.8	51.5	50.4	50.5	50	51.8	104	2.076	4	60 130	Passed
tert-Butyl alcohol	281	286	255	271	250	273	109	13.989	5	60 140	Passed
2-Butanone (MEK)	279	278	284	282	250	281	112	3.049	1	70 130	Passed
n-Butylbenzene	53.8	54.5	49.7	50.6	50	52.2	104	2.387	5	70 130	Passed
sec-Butylbenzene	52.0	52.5	47.9	49.4	50	50.5	101	2.181	4	70 130	Passed
tert-Butylbenzene	52.4	52.3	48.2	49.6	50	50.6	101	2.047	4	70 130	Passed
Carbon disulfide	48.2	46.9	44.2	46.0	50	46.4	93	1.669	4	60 130	Passed
Carbon tetrachloride	52.9	53.1	49.7	51.2	50	51.7	103	1.588	3	70 130	Passed
Chlorobenzene	49.3	50.2	47.1	47.9	50	48.6	97	1.402	3	70 130	Passed
2-Chloroethyl vinyl ether	50.1	49.7	36.4	38.4	50	43.7	87	7.258	17	50 150	Passed
Chloroethane	58.5	52.1	53.0	53.1	50	54.2	108	2.944	5	70 130	Passed
Chloroform	52.1	52.6	50.9	51.3	50	51.7	103	0.749	1	70 130	Passed
1-Chlorohexane	56.0	56.6	50.6	51.8	50	53.7	107	2.974	6	70 130	Passed
Chloromethane	53.8	48.8	48.5	50.0	50	50.3	101	2.456	5	60 130	Passed
2-Chlorotoluene	49.0	48.9	45.9	46.6	50	47.6	95	1.619	3	70 130	Passed
4-Chlorotoluene	51.9	53.0	49.5	49.8	50	51.1	102	1.675	3	70 130	Passed
2-Chloro-1,1,1-trifluoroethane	54.6	53.8	51.2	54.3	50	53.5	107	1.562	3	30 160	Passed
Chlorotrifluoroethylene	46.4	44.7	41.6	45.6	50	44.6	89	2.093	5	30 160	Passed
Dibromochloromethane	48.4	49.7	47.4	47.9	50	48.4	97	1.017	2	70 130	Passed
1,2-Dibromo-3-chloropropane	47.1	47.9	44.3	45.6	50	46.2	92	1.577	3	60 130	Passed
1,2-Dibromoethane	49.2	50.7	47.3	48.4	50	48.9	98	1.452	3	70 130	Passed
Dibromomethane	48.1	49.6	47.1	48.3	50	48.3	97	1.022	2	70 130	Passed
1,1-Dichloroethane	55.3	55.9	52.7	53.4	50	54.3	109	1.554	3	70 130	Passed
1,2-Dichloroethane	49.5	50.1	48.7	49.8	50	49.5	99	0.593	1	70 130	Passed
1,2-Dichlorobenzene	47.0	47.6	45.5	45.6	50	46.4	93	1.084	2	70 130	Passed
1,3-Dichlorobenzene	48.6	49.3	45.9	46.2	50	47.5	95	1.704	4	70 130	Passed
trans-1,4-Dichloro-2-Butene	51.0	50.7	48.7	50.0	50	50.1	100	1.012	2	70 130	Passed
1,4-Dichlorobenzene	49.0	49.7	46.6	46.7	50	48.0	96	1.599	3	70 130	Passed
Dichlorodifluoromethane	51.3	47.0	46.7	47.4	50	48.1	96	2.151	4	60 130	Passed
1,1-Dichloroethene	58.3	58.5	54.2	55.1	50	56.5	113	2.189	4	70 130	Passed
cis-1,2-Dichloroethene	53.3	53.5	50.9	51.2	50	52.2	104	1.358	3	70 130	Passed
trans-1,2-Dichloroethene	55.7	55.7	51.9	52.8	50	54.0	108	1.960	4	70 130	Passed
Dichlorofluoromethane	56.9	57.0	54.7	55.1	50	55.9	112	1.234	2	70 130	Passed
1,1-Dichloropropene	53.1	53.5	48.5	49.7	50	51.2	102	2.483	5	70 130	Passed
1,2-Dichloropropane	51.0	52.2	48.8	50.4	50	50.6	101	1.413	3	70 130	Passed

Appendix 3 (cont.): DEMONSTRATION OF CAPABILITY for 5 ml

DEMONSTRATION OF CAPABILITY
METHOD: SW 8260

Unit: µg/L
Sample Amount(ml): 5
Sample Purge(ml): 5

Date Analyzed: 01/07/13 & 01/08/13
Analyzed by: C. Mendoza

PARAMETER	RAN044 VSF4A07L	RAN045 VSF4A07C	RAN058 VSF4A08L	RAN059 VSF4A08C	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
1,3-Dichloropropane	49.4	50.8	47.8	49.3	50	49.3	99	1.251	3	70 130	Passed
2,2-Dichloropropane	57.2	57.1	54.0	54.5	50	55.7	111	1.706	3	70 140	Passed
cis-1,3-Dichloropropene	51.3	52.8	49.1	50.4	50	50.9	102	1.548	3	70 130	Passed
trans-1,3-Dichloropropene	51.2	52.4	49.6	50.5	50	50.9	102	1.191	2	70 130	Passed
tert-Butyl ethyl ether (ETB	56.5	57.2	53.8	55.1	50	55.7	111	1.523	3	70 130	Passed
Ethyl Methacrylate	52.9	53.6	49.9	51.0	50	52	104	1.679	3	30 160	Passed
Ethylbenzene	51.6	52.9	48.5	49.6	50	50.7	101	1.946	4	70 130	Passed
2-Hexanone (MBK)	275	275	284	277	250	278.0	111	4.403	2	70 130	Passed
Hexachlorobutadiene	47.3	49.2	45.7	45.1	50	46.8	94	1.814	4	70 130	Passed
Iodomethane	40.7	39.4	36.9	39.0	50	39.0	78	1.595	4	60 130	Passed
Isopropyl ether (DIPE)	57.2	58.2	54.5	56.0	50	56.5	113	1.595	3	70 130	Passed
Isopropylbenzene	52.1	52.3	48.0	49.5	50	50.5	101	2.093	4	70 130	Passed
p-Isopropyltoluene	52.4	53.0	48.7	49.2	50	51	102	2.199	4	70 130	Passed
Methyl acetate	52.9	52.1	54.2	55.6	50	53.7	107	1.525	3	30 160	Passed
Methylene Chloride	50.4	51.6	49.6	50.2	50	50.5	101	0.856	2	70 130	Passed
4-Methyl-2-pentanone (MIBK)	278	279	283	276	250	278.7	111	2.713	1	70 130	Passed
tert-Butyl methyl ether	54.7	55.3	52.6	53.8	50	54.1	108	1.140	2	70 130	Passed
Naphthalene	43.7	44.3	40.8	39.8	50	42.2	84	2.180	5	60 140	Passed
n-Propylbenzene	51.9	52.3	47.9	49.3	50	50.4	101	2.098	4	70 130	Passed
Styrene	51.6	53.2	49.6	50.4	50	51.2	102	1.553	3	70 130	Passed
tert-Amyl methyl ether (TAME)	55.3	56.0	53.3	54.2	50	54.7	109	1.212	2	70 130	Passed
1,1,1,2-Tetrachloroethane	48.7	50.4	47.5	48.2	50	48.7	97	1.250	3	30 160	Passed
1,1,2,2-Tetrachloroethane	49.1	49.2	45.7	47.4	50	47.9	96	1.652	3	30 160	Passed
Tetrachloroethene	50.6	51.2	47.0	48.0	50	49.2	98	2.014	4	70 130	Passed
Toluene	51.3	52.4	48.6	49.7	50	50.5	101	1.718	3	70 130	Passed
1,1,1-Trichloroethane	55.0	55.2	52.6	52.9	50	53.9	108	1.405	3	30 160	Passed
1,1,2-Trichloroethane	48.3	50.1	46.3	48.0	50	48.2	96	1.553	3	30 160	Passed
1,2,3-Trichlorobenzene	48.0	49.6	46.0	44.8	50	47.1	94	2.140	5	70 130	Passed
1,2,4-Trichlorobenzene	50.8	51.5	46.7	45.5	50	48.6	97	2.965	6	70 130	Passed
Trichloroethene	50.7	51.5	47.7	48.3	50	49.5	99	1.834	4	70 130	Passed
Trichlorofluoromethane	61.7	55.1	54.8	55.3	50	56.7	113	3.322	6	70 140	Passed
1,2,3-Trichloropropane	47.8	47.7	45.6	47.4	50	47.1	94	1.036	2	70 130	Passed
1,1,2-Trichloro1,2,2-trifluoroethane	57.4	56.8	52.9	53.6	50	55.2	110	2.264	4	70 - 130	Passed
1,2,4-Trimethylbenzene	51.5	52.0	48.2	49.2	50	50.2	100	1.810	4	70 130	Passed
1,3,5-Trimethylbenzene	51.7	52.3	48.0	49.2	50	50.3	101	2.016	4	70 130	Passed
Vinyl Acetate	65.3	59.2	58.1	58.5	50	60.3	121	3.366	6	50 140	Passed
Vinyl Chloride	59.8	52.9	54.5	54.5	50	55.4	111	3.040	5	70 140	Passed
m-Xylene & p-xylene	103	105	97.0	98.7	100	101.0	101	3.765	4	70 130	Passed
o-Xylene	52.3	53.5	49.4	50.7	50	51.5	103	1.786	3	70 130	Passed
Allyl Chloride	57.3	58.3	54.1	55.5	50	56.3	113	1.869	3	30 160	Passed

Appendix 4: DEMONSTRATION OF CAPABILITY for 5 g

DEMONSTRATION OF CAPABILITY
METHOD: SW 8260

Unit: µg/Kg
Sample Amount(g): 5
Sample Purge(ml): 5

Date Analyzed: 03/20 & 03/21/12
Analyzed by: C. Mendoza

PARAMETER	RCP061 VO02C04L	RCP062 VO02C04C	RCP086 VO02C06L	RCP087 VO02C06C	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
Acetone	259	264	288	281	250	273	109	14.006	5	40 - 140	Passed
Acetonitrile	480	506	528	546	500	515	103	28.361	6	50 - 150	Passed
Acrylonitrile	240	245	275	268	250	257	103	17.133	7	10 - 160	Passed
Benzene	50.1	49.3	49.9	49.6	50	49.7	99	0.364	1	70 - 130	Passed
Bromobenzene	49.3	47.4	48.9	49.1	50	48.7	97	0.838	2	70 - 130	Passed
Bromochloromethane	52.6	53.3	55.0	56.1	50	54.2	108	1.592	3	70 - 130	Passed
Bromodichloromethane	50.1	49.8	51.8	52.2	50	51.0	102	1.207	2	70 - 130	Passed
Bromoform	49.2	49.2	50.3	49.6	50	49.6	99	0.501	1	70 - 130	Passed
Bromomethane	46.4	46.6	45.4	46.7	50	46.3	93	0.585	1	60 - 130	Passed
tert-Butyl alcohol	254	266	274	285	250	270	108	13.169	5	40 - 150	Passed
2-Butanone (MEK)	246	251	271	260	250	257	103	11.327	4	60 - 140	Passed
n-Butylbenzene	53.2	51.3	52.5	53.9	50	52.7	105	1.105	2	70 - 130	Passed
sec-Butylbenzene	52.1	51.3	51.9	52.3	50	51.9	104	0.441	1	70 - 130	Passed
tert-Butylbenzene	48.4	48.0	48.7	49.4	50	48.6	97	0.572	1	70 - 130	Passed
Carbon disulfide	48.1	49.8	52.1	42.5	50	48.1	96	4.087	8	60 - 130	Passed
Carbon tetrachloride	52.5	51.2	52.2	51.2	50	51.8	104	0.666	1	70 - 130	Passed
Chlorobenzene	51.3	51.3	49.8	51.2	50	50.9	102	0.728	1	70 - 130	Passed
2-Chloroethyl vinyl ether	57.8	57.8	51.8	51.2	50	54.6	109	3.681	7	50 - 140	Passed
Chloroethane	55.0	56.1	52.3	53.3	50	54.2	108	1.696	3	70 - 140	Passed
Chloroform	55.4	55.5	53.0	54.3	50	54.5	109	1.171	2	70 - 130	Passed
1-Chlorohexane	53.3	51.9	51.8	52.2	50	52.3	105	0.678	1	70 - 130	Passed
Chloromethane	49.5	51.4	50.8	52.4	50	51.0	102	1.201	2	60 - 130	Passed
2-Chlorotoluene	44.7	48.4	45.5	48.0	50	46.6	93	1.814	4	70 - 130	Passed
4-Chlorotoluene	57.2	51.0	56.4	52.5	50	54.3	109	3.031	6	60 - 130	Passed
Dibromochloromethane	49.5	49.7	51.1	52.9	50	50.8	102	1.574	3	70 - 130	Passed
1,2-Dibromo-3-chloropropane	44.9	46.0	47.8	48.2	50	46.7	93	1.570	3	60 - 130	Passed
1,2-Dibromoethane	47.2	48.1	50.3	50.4	50	49.0	98	1.600	3	70 - 130	Passed
Dibromomethane	48.8	48.5	49.8	50.6	50	49.4	99	0.957	2	70 - 130	Passed
1,1-Dichloroethane	55.4	55.2	54.5	55.8	50	55.2	110	0.518	1	70 - 130	Passed
1,2-Dichloroethane	51.3	50.9	51.7	51.1	50	51.3	103	0.310	1	70 - 130	Passed
1,2-Dichlorobenzene	50.3	50.3	49.0	50.0	50	49.9	100	0.602	1	70 - 130	Passed
1,3-Dichlorobenzene	50.6	50.0	50.0	50.8	50	50.3	101	0.429	1	70 - 130	Passed
trans-1,4-Dichloro-2-Butene	48.8	47.7	53.8	52.2	50	50.6	101	2.848	6	60 - 140	Passed
1,4-Dichlorobenzene	51.7	51.0	49.8	50.8	50	50.8	102	0.775	2	70 - 130	Passed
Dichlorodifluoromethane	66.9	67.2	58.9	59.2	50	63.1	126	4.620	7	60 - 130	Passed
1,1-Dichloroethene	51.9	53.0	52.1	54.3	50	52.8	106	1.104	2	60 - 130	Passed
cis-1,2-Dichloroethene	49.5	49.2	53.0	54.8	50	51.6	103	2.725	5	70 - 130	Passed
trans-1,2-Dichloroethene	53.2	52.2	54.2	55.1	50	53.7	107	1.267	2	70 - 130	Passed
Dichlorofluoromethane	49.3	51.4	53.2	56.7	50	52.6	105	3.134	6	70 - 130	Passed
1,1-Dichloropropene	51.5	49.7	50.8	50.5	50	50.6	101	0.753	1	70 - 130	Passed
1,2-Dichloropropane	50.5	49.7	52.0	51.8	50	51.0	102	1.108	2	70 - 130	Passed
1,3-Dichloropropane	47.6	47.9	50.9	51.5	50	49.5	99	2.010	4	70 - 130	Passed
2,2-Dichloropropane	56.3	55.0	54.4	55.9	50	55.4	111	0.857	2	60 - 140	Passed
cis-1,3-Dichloropropene	51.7	51.3	52.3	52.4	50	52.0	104	0.517	1	70 - 130	Passed

Appendix 4 (cont.): DEMONSTRATION OF CAPABILITY for 5 g

DEMONSTRATION OF CAPABILITY
METHOD: SW 8260

Unit: µg/Kg
Sample Amount(g): 5
Sample Purge(ml): 5

Date Analyzed: 03/20 & 03/21/12
Analyzed by: C. Mendoza

PARAMETER	RCP061 VO02C04L	RCP062 VO02C04C	RCP086 VO02C06L	RCP087 VO02C06C	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
trans-1,3-Dichloropropene	52.6	52.3	53.1	53.7	50	52.9	106	0.623	1	70 - 130	Passed
1,4-Dioxane	878	878	947	958	1000	915	92	43.314	5	50 - 150	Passed
tert-Butyl ethyl ether (ETBE)	54.0	53.9	54.2	55.2	50	54.3	109	0.617	1	70 - 130	Passed
Ethyl Methacrylate	49.5	49.8	51.2	51.4	50	50.5	101	0.964	2	70 - 130	Passed
Ethylbenzene	50.5	50.8	50.7	51.3	50	50.8	102	0.355	1	70 - 130	Passed
2-Hexanone (MBK)	241	247	270	256	250	253	101	12.291	5	20 - 160	Passed
Hexachlorobutadiene	52.6	48.4	49.6	49.2	50	49.9	100	1.873	4	70 - 130	Passed
Iodomethane	61.3	61.7	50.7	52.5	50	56.6	113	5.760	10	60 - 140	Passed
Isopropyl ether (DIPE)	53.2	53.2	54.7	55.5	50	54.2	108	1.156	2	70 - 130	Passed
Isopropylbenzene	55.9	53.8	51.7	50.4	50	52.9	106	2.417	5	70 - 130	Passed
p-Isopropyltoluene	53.6	52.7	51.5	51.5	50	52.3	105	1.040	2	70 - 130	Passed
Methylene Chloride	48.6	49.4	49.9	51.6	50	49.9	100	1.276	3	70 - 130	Passed
4-Methyl-2-pentanone (MIBK)	243	249	266	253	250	253	101	9.792	4	50 - 150	Passed
tert-Butyl methyl ether	51.2	51.8	52.3	53.4	50	52.2	104	0.957	2	70 - 130	Passed
Naphthalene	49.7	47.2	48.9	48.7	50	48.6	97	1.018	2	60 - 140	Passed
n-Propylbenzene	50.2	48.4	51.1	49.9	50	49.9	100	1.136	2	70 - 130	Passed
Styrene	52.6	53.1	51.7	53.0	50	52.6	105	0.637	1	70 - 130	Passed
tert-Amyl methyl ether (TAME)	51.9	52.9	52.4	53.9	50	52.8	106	0.850	2	70 - 130	Passed
1,1,1,2-Tetrachloroethane	50.7	51.8	50.8	52.0	50	51.3	103	0.670	1	70 - 130	Passed
1,1,2,2-Tetrachloroethane	48.3	48.6	50.5	49.7	50	49.3	99	1.013	2	70 - 130	Passed
Tetrachloroethene	52.5	50.5	50.0	50.2	50	50.8	102	1.184	2	70 - 130	Passed
Toluene	49.9	49.3	50.1	50.1	50	49.9	100	0.359	1	70 - 130	Passed
1,1,1-Trichloroethane	55.4	55.0	53.7	54.8	50	54.7	109	0.756	1	70 - 130	Passed
1,1,2-Trichloroethane	49.6	49.4	50.3	51.7	50	50.2	100	1.049	2	70 - 130	Passed
1,2,3-Trichlorobenzene	52.9	49.1	50.9	50.8	50	50.9	102	1.545	3	70 - 130	Passed
1,2,4-Trichlorobenzene	56.6	52.9	52.3	54.2	50	54.0	108	1.911	4	70 - 140	Passed
Trichloroethene	51.4	50.6	49.6	49.4	50	50.3	101	0.955	2	70 - 130	Passed
Trichlorofluoromethane	63.3	61.5	57.5	55.8	50	59.6	119	3.486	6	70 - 140	Passed
1,2,3-Trichloropropane	45.2	45.3	48.9	48.3	50	46.9	94	1.942	4	70 - 130	Passed
1,1,2-Trichloro1,2,2-trifluoroethane	57.2	58.5	50.7	54.4	50	55.2	110	3.450	6	70 - 140	Passed
1,2,4-Trimethylbenzene	52.6	51.3	51.3	51.4	50	51.7	103	0.647	1	70 - 130	Passed
1,3,5-Trimethylbenzene	50.7	49.9	51.0	51.1	50	50.7	101	0.516	1	70 - 130	Passed
Vinyl Acetate	50.9	47.8	59.8	57.1	50	53.9	108	5.524	10	20 - 160	Passed
Vinyl Chloride	55.2	57.3	51.7	52.9	50	54.3	109	2.495	5	60 - 140	Passed
m-Xylene & p-xylene	102	101	101	104	100	102.0	102	1.343	1	70 - 130	Passed
o-Xylene	49.9	50.3	51.3	52.6	50	51.0	102	1.217	2	70 - 130	Passed

8260FS: **SAMPLE PREPARATION LOG**

EMAX LABORATORIES, INC.

PRESERVATION LOG FOR VOLATILES

VP.xls

PrepBatchID	Lab Sample ID	W1 (g)	DateTime	Wf (g)	DateTime	ExpWs(g)	Ws(g)	Prep□Factor	Comments

☐ EMAX-5035 Rev. ☐ Balance Calibration Check performed prior to use — Surrogate ID: — Prepared By:

☐ Encore+$NaHSO_4 \cdot H_2O$ ☐ BalanceID 25451062 — BlankSoil Lot#: — Standard Added By:

☐ Frozen Sample ☐ BalanceID 25650797 — NaHSO4H2O: — Witnessed By:

☐ Pre-weighed vial — Expected Ws(g): 5 — Extract Rcvd By:

☐ Volumetric dispenser was checked with 5-ml volumetric flask — Sample Location:

W1=Weight of Vial + Stirrer+NaHSO4•H2O+Solvent Wf=W1 + Sample

8260FA: **ANALYTICAL RUN LOG**

EMAX LABORATORIES, INC.

Page 1

ANALYSIS LOG FOR VOLATILES

SOP ☐ EMAX-8260 Rev.No. __ ☐ EMAX-624 Rev.No. __ ☐ EMAX-8260SIM Rev.No. __ ☐ EMAX-TCPSIM Rev.No. __ ☐ EMAX-M8260SIM Rev.No. __ ☐ EMAX-8260C Rev. No. __

Start Date: ☐ 5-mL Purge ☐ 10-mL Purge ☐ 25-mL Purge Book # A06 -053

BATCH _____

Sample Prep ID	Data File Name	Lab Sample ID	Sample Amount	DF	Matrix W pH < 2	Matrix W Cl_2 < 3ppm	Matrix S	Notes
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

Instrument No.	06
INITIAL CALIBRATION REFERENCE	
DATE	
ICAL ID	

STANDARDS			
NAME	ID	Amount (µl)	Conc. (mg/L)
DCC			
DCC			
DCC			
DCC			
BFB			
IS/SURR.			
ICV/LCS			
ICV/LCS			
ICV/LCS			
ICV/LCS			
Data File Folder			
	LOT #		
pH strip			
Chlorine strip			
Methanol			
$NaHSO_4$			
Reagent Water			
Sand			

Electronic Data Archival Location	Date
HPCHEM_VOA/TO06	

Comments: _____

☐ Refer to sample weight log

Analyzed By: _____

Date Disposed: _____ Disposed By: _____

8260FM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: 05 Month: Year: 201_ Book # : MD3-005

DATE						
Bake Trap (210°C-260°C)						
Column Head Pressure (2-60 psi)						
Injector Port Temperature (160°C-220°C)						
Interface Temperature (250°C-280°C)						
Verify Computer Date and Time						
Check Gas Pressure (Daily)						
Check IS/Surrogate auto injection vial as appropriate (Daily)						
Check the foreline pump oil level (Weekly)						
AutoTune the MSD (As needed)						
Analyst's initial						
Trouble Shooting						
Problem(s) Encountered						
Return-to-Control (Analyst Initial)						

Code	Problems Encountered	Code	Corrrective Action

EMAX
LABORATORIES, INC.
1835 W. 205th Street
Torrance, CA 90501
Tel: (310) 618-8889
Fax: (310) 618-0818

ERRATUM TO	
Document	EMAX-8270
Revision Number	6
Section	Forms: Table 8
Date	September 20, 2016
Reference Number	8270.6.2

Table 8 shall read as attached.

PREPARED BY: _____ Date: 09-20-16

Name Tu Nisamaneepong

Title Operations Manager

APPROVED BY: _____ Date: 09-20-16

Name Kenette Pimentel

Title QA Manager

APPROVED BY: _____ Date: 09-20-16

Name Caspar Pang

Title Laboratory Director

Table 8: TYPICAL TARGET ANALYTE LIST FOR REGULAR LIST

Analyte	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7	8			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
Acenaphthene	153	152	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Acenaphthylene	152	151	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Aniline	93	66	IS1	Sur3	4	10	20	25	40	50			25	40	5.3	10	20	µg/L	83	167	667	µg/Kg
Anthracene	178	152	IS4	Sur4/6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Azobenzene*	77	105	IS4	Sur4/6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	96	167	333	µg/Kg
Benzo(a)anthracene	228.1	113	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzo(a)pyrene	252.1	125	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzo(b)fluoranthene	252.1	124.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.6	5	10	µg/L	86	167	333	µg/Kg
Benzo(e)pyrene	252.1	125	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzo(g,h,i)perylene	276.1	137.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	87	167	333	µg/Kg
Benzo(k)fluoranthene	252.1	124.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzoic acid	105	77	IS2	Sur1/2	4	10	20	25	40	50	80	100	25	80	10	20	40	µg/L	333	667	1333	µg/Kg
Benzyl alcohol	108	79	IS1	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Biphenyl	154	153	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis (2-Chloroethoxy) methane	92.9	94.9	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis (2-Chloroethyl) ether	92.9	62.9	IS1	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis (2-chloroisopropyl) ether	45	76.9	IS1	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis(2-ethylhexyl)adipate	129	57	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	87	167	333	µg/Kg
bis(2-Ethylhexyl)phthalate	149	167	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	115	167	333	µg/Kg
4-Bromophenyl-phenylether	247.9	250	IS4	Sur4/6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	90	167	333	µg/Kg
Butylbenzylphthalate	149	91	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Carbazole	167	139	IS4	Sur4/6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	89	167	333	µg/Kg
4-Chloro-3-methylphenol	107	77	IS2	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Chloroaniline	127	129	IS2	Sur3	4	10	20	25	40	50			25	40	4.2	5	10	µg/L	83	167	333	µg/Kg
2-Chloronaphthalene	162	164	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Chlorophenol	127.9	64	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Chlorophenyl-phenylether	204	206	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Chrysene	228.1	226.1	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dibenzo(a,h)anthracene	278.1	139	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dibenzofuran	168	139	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,2-Dichlorobenzene	145.9	147.9	IS1	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,3-Dichlorobenzene	145.9	147.9	IS1	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,4-Dichlorobenzene	145.9	147.9	IS1	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
3,3'-Dichlorobenzidine	252	254	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	84	167	333	µg/Kg
2,4-Dichlorophenol	161.9	163.9	IS2	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Diethylphthalate	149	177	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,6-Dimethylnaphthalene	156	141	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,4-Dimethylphenol	107	122	IS2	Sur1/2	4	10	20	25	40	50			25	40	2.6	5	10	µg/L	83	167	333	µg/Kg
3,4-Dimethylphenol	107	122	IS2	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dimethylphthalate	163	164	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Di-n-butylphthalate	149	150	IS4	Sur4/6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	97	167	333	µg/Kg

Table 8: TYPICAL TARGET ANALYTE LIST FOR REGULAR LIST

Analyte	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7	8			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
4,6-Dinitro-2-methylphenol	198	121	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,3-Dinitrobenzene	168	75	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,4-Dinitrophenol	184	106.9	IS3	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	86	167	333	µg/Kg
2,4-Dinitrotoluene	165	89	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,6-Dinitrotoluene	165	89	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Di-n-octylphthalate	149	150	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	97	167	333	µg/Kg
Fluoranthene	202	100.9	IS4	Sur4/6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	126	167	333	µg/Kg
Fluorene	166	165	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachlorobenzene	283.8	141.9	IS4	Sur4/6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachlorobutadiene	224.8	222.8	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachlorocyclopentadiene	236.8	234.8	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachloroethane	116.8	200.8	IS1	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Indeno(1,2,3-cd)pyrene	276.1	137.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Isophorone	82	138	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1-Methylnaphthalene	142	141	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Methylnaphthalene	142	141	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1-Methylphenanthrene	192.1	165	IS4	Sur4/6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Methylphenol	107	108	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Methylphenol	107	108	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Naphthalene	128	129	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Nitroaniline	65	92	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
3-Nitroaniline	138	92	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Nitroaniline	138	92	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	120	167	333	µg/Kg
Nitrobenzene	77	123	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Nitrophenol	139	65	IS2	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Nitrophenol	109	139	IS3	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	106	167	667	µg/Kg
N-nitrosodimethylamine	74	42	IS1	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-Nitroso-di-n-propylamine	70	42	IS1	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-Nitrosodiphenylamine	169	168	IS4	Sur4/6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	153	167	333	µg/Kg
Pentachlorophenol	265.8	164.9	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	667	µg/Kg
Perylene	252.1	125	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	86	167	333	µg/Kg
Phenanthrene	178	152	IS4	Sur4/6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Phenol	94	65	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Pyrene	202	100.9	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	160	167	333	µg/Kg
Pyridine	79	52	IS1	Sur3	4	10	20	25	40	50	80	100	25	80	11	20	40	µg/L	333	667	1333	µg/Kg
2,3,4,6-Tetrachlorophenol	231.8	229.8	IS3	Sur5	4	10	20	25	40	50			25	40	3.6	5	10	µg/L	100	167	333	µg/Kg
1,2,4-Trichlorobenzene	179.9	181.9	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,3,4-Trichlorophenol	195.9	96.9	IS3	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,3,5-Trichlorophenol	195.9	96.9	IS3	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	165	167	333	µg/Kg
2,4,5-Trichlorophenol	195.9	197.9	IS3	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	91	167	333	µg/Kg
2,4,6-Trichlorophenol	195.9	197.9	IS3	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg

Table 8: TYPICAL TARGET ANALYTE LIST FOR REGULAR LIST

Analyte	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7	8			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
2,3,5-Trimethylnaphthalene	171.1	155	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzidine	184	92	IS1/4	Sur6	4	10	25	40	50	80	100		25	80	10	20	50	µg/L	863	867	1333	µg/Kg
2-Fluorophenol (Sur1)	112	64	IS1		4	10	20	25	40	50			25	40	1.5	3	5	µg/L	61	100	167	µg/Kg
Phenol-d5 (Sur2)	99	71	IS1		4	10	20	25	40	50			25	40	1.5	3	5	µg/L	52	100	167	µg/Kg
Nitrobenzene-d5 (Sur3)	82	128	IS2		4	10	20	25	40	50			25	40	0.59	1	1.67	µg/L	17	33	56	µg/Kg
2-Fluorobiphenyl (Sur4)	172	171	IS3		4	10	20	25	40	50			25	40	0.5	1	1.67	µg/L	24	33	56	µg/Kg
2,4,6-Tribromophenol (Sur5)	329.8	331.8	IS4		4	10	20	25	40	50			25	40	1.5	3	5	µg/L	55	100	167	µg/Kg
Terphenyl-d14 (Sur6)	244.2	122	IS5		4	10	20	25	40	50			25	40	0.54	1	1.67	µg/L	24	33	56	µg/Kg
1,4-Dichlorobenzene-d4 (IS1)	151.9	115																				
Naphtalene-d8 (IS2)	136	68																				
Acenaphthene-d10 (IS3)	164.1	162.1																				
Phenanthrene-d10 (IS4)	188.1	94																				
Chrysene-d12 (IS5)	240.1	120																				
Perylene-d12 (IS6)	264.1	130																				

Notes:

1. PSR supersedes Table 8 2. Other analytes not listed above must have a minimum response factor of 0.01 3. * Dissociated to Azobenzene

EMAX
LABORATORIES, INC.
1835 W. 205th Street
Torrance, CA 90501
Tel: (310) 618-8889

Page 1 of 1

ADDENDUM TO	
Document	ALL ANALYTICAL SOPS
Revision Number	CURRENT REVISIONS
Section	10.2 Instrument Parameters
Date	11 September 2019
Reference Number	AA.7

This applies to all GC/MS, GC, HPLC-MS, HPLC, ICP-MS, ICP and IC Method SOPs

Section 10.2 shall include:

10.2. **Instrument Parameters**

10.2.1. Instrument parameters setup stipulated in the SOPs, are instrument suggested parameters. Fine tune the instrument to obtain optimum instrument condition.

10.2.2. Print and staple a copy of the current instrument parameter on the instrument log for easy access when performing daily instrument routine check.

10.2.3. In the event that instrument parameters necessitate a change, replace the instrument parameter printout with the new parameter setup. Archive the previous instrument parameters at the back of the instrument maintenance log.

PREPARED BY: _____ Date: 9/11/19

Name Farina Madamba

Title QA / QC Coordinator

APPROVED BY: _____ Date: 09.11.19

Name Kenette Pimentel

Title QA Manager

APPROVED BY: _____ Date: 09-11-19

Name Caspar Pang

Title Laboratory Director

STANDARD OPERATING PROCEDURES

SEMIVOLATILE ORGANICS BY GC/MS

SOP No.:	EMAX-8270	Revision No.	6	Date:	10-Jul-14
Prepared By:	Souzan Grease			Date:	07-09-14
Approved By:	Kenette Pimentel QA Manager			Date:	07.09.14
Approved By:	Caspar Pang Laboratory Director			Date:	07-09-14

Control Number: 8270-06-

1.0 SCOPE AND APPLICATION

1.1. This method is used to determine the concentration of semivolatile organic compounds extracted from many types of solid waste matrices, soils, air sampling media and water samples. Analytes that are listed in Table 9 were determined when this SOP was established. Additional analytes may be added upon completion of similar validation as the analytes that are listed in Table 8 & 9.

1.2. This SOP is an adaptation of method SW846 8270C.

2.0 SUMMARY OF METHOD

2.1. Samples are extracted with methylene chloride. Extracts are concentrated and appropriate cleanup procedure is applied if necessary.

2.2. Internal standards are added to an aliquot of the final extract and are qualitatively and quantitatively analyzed by gas chromatography equipped with mass spectrometry (GC/MS).

2.3. **Interference**

2.3.1. Solvents, reagents, glassware, and other sample processing devices are all possible sources of artifacts and/or interferences to sample analysis. Hence, quality control of chemicals and proper decontamination of re-usable glassware and proofing the analytical instrument to be free from contamination shall be observed.

2.3.2. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interference. Determine source of interference in the preparation and/or cleanup of the samples and Perform corrective action to eliminate the problem.

2.3.3. Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, rinse the sample syringe with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

2.3.4. Another possible source of contamination is the analytical instrument itself. This can be monitored by analyzing an instrument blank prior to any analysis.

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.

3.1.2. Refer to Table 8 & 9 for established limits.

4.0 DYNAMIC RANGE

4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point. All samples analyzed above this concentration are considered "over-range" and requires dilution for proper quantitation.

4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required for proper quantitation.

4.3. The dynamic range established for this method are:

Water (µg/L)	Soil (µg/kg)
10 - 100 µg/L	330 - 3300 µg/kg

5.0 SAMPLE HOLDING TIME & PRESERVATION

5.1. **Sample Preservation**

5.1.1. Store water and soil samples at ≤ 6°C away from light without freezing.

5.1.2. Store all extracts at ≤ 6°C without freezing.

5.2. **Holding Time**

5.2.1. Extract water samples within 7 days from sampling date.

5.2.2. Extract soil samples within 14 days from sampling date.

5.2.3. Analyze all extracts within 40 days from extraction completion date.

6.0 ASSOCIATED SOPs

6.1. EMAX-1311 TCLP for Organic and Inorganic Analytes

6.2. EMAX-3520 Extraction, Continuous Liquid/Liquid

6.3. EMAX-3540 Extraction, Soxhlet

6.4. EMAX-3546 Extraction, Microwave

6.5. EMAX-3550 Extraction, Pulse Sonication

6.6. EMAX-3580 Waste Dilution

6.7. EMAX-3640 Cleanup, GPC

6.8. EMAX-DM01 Data Flow and Review

6.9. EMAX-QA04 Detection Limit (DL)

6.10. EMAX-QA05 Training

6.11. EMAX-QA08 Corrective Action

6.12. EMAX-QC01 Quality Control for Chemicals

6.13. EMAX-QC02 Analytical Standard Preparation

6.14. EMAX-QC07 Glassware Cleaning

6.15. EMAX-SM01 Sample Management

6.16. EMAX-SM03 Waste Disposal

6.17. EMAX-SM04 Analytical and QC Sample Labeling

7.0 SAFETY

7.1. Read all SDS for all chemicals listed in this SOP.

7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.

7.3. If for any reason, solvent and/or other reagents get in contact with the skin or any other part of the body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS & REAGENTS

8.1. **Instruments and Supplies**

Gas Chromatography	Agilent Technologies 7890A with split/splitless injection, Shimadzu GC-17A, or equivalent
Mass Spectrometer	Agilent Technologies 5975C MSD or Shimadzu GCMS – GP 5000 capable of scanning from 1.6 to 1050 amu every 1 second using 70 volts electrode energy in the electron impact ionization mode or equivalent
GC/MS Interface	Capillary-direct into the mass spectrometer source or equivalent
Chromatographic Column	ZB-5MS (20 m x 0.18 mm x 0.32 µm) or equivalent
Data System	MS-ChemStation with Enviroquant software or equivalent
GC Autosampler	Agilent Technologies 7683B series injector or Shimadzu AOC-20i capable of direct injection of 1 µL and 10 µL of extract.
Gases	Ultra high purity helium
Syringes	10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL and 1000 µL syringe Hamilton 202N or equivalent
Vials	Autosampler vials with teflon lined septa

8.2. **Chemicals and Reagents**

Methylene chloride, pesticides grade

Methanol, high purity

9.0 STANDARDS

9.1. Standard Preparation

9.1.1. Follow procedures for all standard preparations and labeling as described in EMAX-QC02 and EMAX-SM04, respectively.

9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

9.2. Stock Standard

9.2.1. Purchase stock standards as certified solutions from a reputable vendor (refer to Table 1 for the listing of all certified solutions). Standards are expected to be at least 96% purity. Read vendor's note if correction has been applied to certified values, otherwise corrections must be applied.

9.2.2. Transfer the stock standard solutions into 2 mL amber vial with Teflon lined screw caps and store at -10°C to -20°C.

9.3. Intermediate Standard

9.3.1. Using stock standard solutions, prepare the intermediate standard in Methylene Chloride according to Table 1.

9.4. Internal Standard

9.4.1. The internal standard shall include 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthalene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12} and Perylene-d_{12} in methylene chloride solution.

9.4.2. Purchase internal standard solutions as certified solution from a reputable vendor at 4,000 µg/mL.

9.4.3. Prepare a 10 mL of 2,000 µg/mL of working internal standard from 4,000 µg/mL (refer to Table 4). Transfer the solution in a properly labeled 10 mL amber vial and store in -10°C to -20°C.

9.5. GC/MS Tuning

9.5.1. The tuning standard shall include decafluorotriphenylphosphine (DFTPP), 4,4-DDT, Pentachlorophenol, and Benzidine.

9.5.2. Purchase tuning standard solution as certified standard at 1000 µg/mL.

9.5.3. Prepare a 500 µL of 50 µg/mL of working standard tuning solution (refer to Table 5). Transfer the solution in a 1 mL amber vial and store in -10°C to -20°C.

9.6. Surrogate Standard

9.6.1. Purchase surrogate stock standards as certified standard.

9.6.1.1. The acid surrogate mixture includes Phenol-d_5, 2-Fluorophenol, and 2,4,6-Tribromophenol at 150 µg/mL.

9.6.1.2. The basic neutral surrogate mixture includes Nitrobenzene-d_5, 2-Fluorobiphenyl, Terphenyl-d_{14}, and 1,2-Dichlorobenzene-d_4 at 50 µg/mL.

9.6.2. For typical extraction [soil: 30 g – 2 mL or water: 1000 mL – 2 mL], add 0.4 mL of surrogate spiking standard to the sample prior to extraction. Spike volume may be adjusted to normalize with the final extract and yield the same concentration.

9.7. **Calibration Standard**

9.7.1. Prepare working standard solutions for initial calibration and daily calibration (refer to Tables 2A-2C). Transfer the solutions in a 1 mL amber vial and store them at ≤ 6°C without freezing.

9.8. **ICAL Verification Standard (Second Source Verification) (ICV)**

9.8.1. Purchase a certified ICV standard from a different vendor. The ICV standard contains the same list of compounds as the stock standard (refer to Table 1B for the standard mix and the corresponding vendors).

9.8.2. Prepare a 500 µL of 25 µg/mL check standard solution (refer to Table 3A-3C). Transfer the solution in a properly labeled 1 mL amber vial and store at ≤ 6°C without freezing.

9.9. **LCS/MS Spiking Standards**

9.9.1. Purchase spiking standards as certified solutions from a different source.

9.9.2. Refer to specific extraction SOP for spiking amount used for LCS/LCSD/MS/MSD, unless otherwise specified by project.

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. For aqueous samples (including TCLP leachates), refer to EMAX-3520 or EMAX-1311. Record in the extraction log if presence of residual chlorine is observed.

10.1.2. For solid samples, refer to EMAX-3550, EMAX-3540 or EMAX-3546.

10.1.3. For waste samples, refer to EMAX-3580.

10.1.4. After extraction, examine the color and consistency of the extract. If the extract appears to be opaque and/or viscous, it is advisable to perform extract cleanup preferably GPC. Refer to EMAX-3640.

10.2. **Instrument Parameters**

10.2.1. Set the instrument parameters as suggested in Table 6. Fine tune the instrument to obtain optimum instrument condition.

10.2.2. Print and display current condition on the instrument for easy access when performing daily instrument routine check.

10.2.3. In the event that instrument parameters necessitate a change, replace the instrument parameter printout with the new parameter setup. Archive the previous instrument parameters in the instrument maintenance log.

10.2.4. Set injection volume to 1 µL to 2 µL.

10.3. **Calibration**

10.3.1. Set GC/MS operating condition as described in Section 10.2.

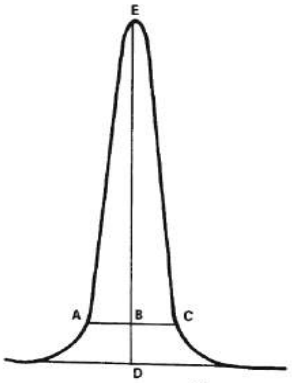
10.3.2. Perform Tune Check

10.3.2.1. Analyze a solution containing 50 µg/mL of tuning standard working solution, DFTTP, DDT, benzidine and pentachlorophenol.

10.3.2.2. A valid Tune Check expires after 12 hours. Evaluate the tune check by averaging three scans (the peak apex scan and the scan immediately preceding and the scan immediately following the apex). Apply a background subtraction using a single scan no more than 20 scans prior to the elution of DFTPP. Do not subtract part of the DFTPP peak or any other discrete peak that does not coelute with DFTPP. Use the DFTPP mass intensity criteria in the manufacturer's instructions as primary tuning acceptance criteria, otherwise refer to Table 7 for acceptance criteria. A valid tune expires after 12 hours.

10.3.2.3. Evaluate column performance and injection port inertness using the data acquisition software.

- Degradation of DDT to DDE and DDD must be less than 20% based on area obtained from the total ion chromatogram.
- Benzidine and Pentachlorophenol must be present at their normal responses. Evaluate the tailing factor of benzidine and pentachlorophenol. Benzidine tailing ≤ 3 and pentachlorophenol ≤ 5. Refer to the attached figure for peak evaluation.



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm
Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

10.3.2.4. If tune check is non-compliant, refer to Section 12 for corrective action.

10.3.3. Initial Calibration (ICAL)

10.3.3.1. Perform ICAL when one of the conditions occurs.

- Instrument is new
- Instrument undergoes a major repair
- DCC failed to meet the acceptance criteria

10.3.3.2. Optimize the instrument condition prior to ICAL.

- Ensure that instrument parameters are set up properly
- Ensure that there is no evidence of leak
- Ensure that instrument maintenance is performed on schedule

- Ensure that instrument tune check and column performance is not indicative that it is at the threshold of failing the acceptance criteria.

10.3.3.3. Analyze a multi-point initial calibration curve as suggested in Figure 3 after a valid tune check.

10.3.3.4. Base quantitation of identified compounds on the integrated abundance from the EICP of the assigned primary characteristic ion (refer to Tables 8 & 9). For optimum output, assign internal standard to each compound based on the nearest retention time or as suggested on tables 8 and 9.

10.3.3.5. **Evaluate the ICAL Acceptance**

10.3.3.5.1. Check for completeness of target compound list. If there is/are missing compound(s), perform the following:

- Check the established retention time window
- Check the relative intensity of major ions
- Adjust accordingly if necessary.

10.3.3.5.2. Evaluate retention time of each analyte with respect to the nearest internal standard. The relative retention time (RRT) of each analyte should agree within ± 0.06 RRT units.

10.3.3.5.3. At a minimum, evaluate System Performance Check Compounds (SPCCs) and Calibration Check Compounds (CCCs) as specified in Appendix 1. The list of SPCCs and CCCs are provided in Tables 10 and 11.

10.3.3.5.4. Check RSD and Correlation Coefficient. If more than 10% of the compounds included with the initial calibration exceed the 15% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Perform necessary instrument maintenance and repeat calibration. Refer to 10.3.3.2., Section 12 for corrective action.

10.3.3.6. **Application of ICAL Curve for Quantitation**

10.3.3.6.1. Generate a summary of Relative Response Factors for each analyte at each concentration. Calculate the Average Relative Response Factor (RRFm), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to Eq.-10.5.1.1, Eq.-10.5.1.2, Eq.-10.5.1.6 and Eq.-10.5.1.7 respectively.

10.3.3.6.2. If RSD is ≤ 15% average response factor may be applied.

10.3.3.6.3. Apply Inverse Weighting Factor ($1/y$ or $1/y^2$; y being the instrument response) if it is determined to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of ≤ 15% and correlation coefficient of ≥ 0.995.

10.3.3.6.4. Apply linear least squares regression if past experience or priori knowledge of instrument response is known to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of ≤ 15% and correlation of determination (COD) ≥ 0.99.

10.3.3.6.5. It may be appropriate to force the regression through zero for specific analytes.[1] When exercising this option [as included in the data acquisition software], make sure that the origin (0,0) is not included as a calibration point but rather the intercept is set to zero. This option shall only be applied if the curve favors better accuracy of quantitation.

10.3.3.7. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

10.3.4. Initial Calibration Verification (ICV)

10.3.4.1. Analyze ICV to verify the concentration of the ICAL standards (Refer to Section 9.8).

10.3.4.2. Check for completeness of analytes as described in Section 10.4.3.

10.3.4.3. Compare the retention times of the internal standards to the ICAL mid-point. Excursion of ± 30 seconds indicate instrument malfunction. Corrective action is required prior to further analysis.

10.3.4.4. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (- 50% to + 100%).

10.3.4.5. Refer to Appendix 1 for ICV acceptance criteria and/or corrective action.

10.3.4.6. When non-compliant refer to Section 12 for corrective action.

10.3.5. Daily Continuing Calibration (DCC)

10.3.5.1. Analyze DCC to check the validity of the ICAL (refer to 9.7).

10.3.5.2. Check for completeness of analytes as described in Section 10.4.3.

10.3.5.3. Evaluate System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC) as specified in Appendix 1.

10.3.5.4. Compare the retention times of the internal standards to the ICAL mid-point. Excursion of ± 30 seconds indicates instrument malfunction. When non-compliant check the column head pressure, gas supply or leaks. Corrective action is required prior to further analysis.

10.3.5.5. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).

10.3.5.6. Establish RRF of each analyte, calculate %D (Eq.-10.5.2.1) against the ICAL.

10.3.5.7. Refer to Appendix 1 for DCC acceptance criteria and/or corrective action,

10.3.5.8. When non-compliant refer to Section 12 for corrective action.

10.4. **Analysis**

10.4.1. Extract Preparation

10.4.1.1. Allow extracts to equilibrate with room temperature.

[1] SW846 Method 8000B, Section 7.5.3

10.4.1.2. Measure 300 µL of extract, transfer into an autosampler vial.

10.4.1.3. Add 6 µL of 2000 µg/mL of internal standard (refer to 9.4).

10.4.1.4. Seal the vial with Teflon-lined septa cap.

10.4.2. Analytical Sequence

10.4.2.1. Analyze instrument blank.

10.4.2.2. Analyze DFTPP and evaluate tuning.

10.4.2.3. Analyze DCC and check ICAL validity.

10.4.2.4. Analyze Method Blank.

10.4.2.5. Analyze Lab Control Sample and Lab Control Sample Duplicate (if requested).

10.4.2.6. Analyze samples to a maximum number of 12-hours from time of DFTPP injection.

10.4.2.7. Analyze a pair of matrix spikes (MS/MSD) for every 20 samples of the same matrix.

10.4.2.8. Record analytical sequence in the analysis log.

10.4.3. Sample Result Evaluation

10.4.3.1. Check the QC criteria as soon as the data is available.

- ✓ Check method blank. If result is non-compliant and analyte in question is not detected in any sample or contamination is < 10X of the sample concentration, results maybe reportable. Verify with the PM if results can be reported.
- ✓ Compare the retention times of each Internal Standards (IS) to the ICAL mid point (must be ± 30 seconds).
- ✓ Compare the area of each IS acquired against the mid point of the ICAL. The Extracted ION Current Profile (EICP) must be within a factor of two (-50 to +100%).
- ✓ Check concentration of target analytes if calibration range is exceeded.
- ✓ Check surrogate recoveries against project specific requirements (PSR). In the absence of PSR, default to Appendix 1 QC limits.
- ✓ If any of the above checkpoints indicate a problem, re-analysis is required. Note observations on the analysis log. When result arise to questionable result, e.g. inconsistency from the first analysis, consult the Supervisor for further action.

10.4.3.2. **Qualitative Identification**

- The intensities of the characteristic ions must maximize in the same scan or within one scan of each other.
- The relative retention time (RRT) of the sample component is within 0.06 RRT units of the RRT of the standard component.
- The relative intensity of the characteristic ions agrees within 30% of the relative intensity of these ions in the reference spectrum.

- Check the chromatogram for possible misidentified analytes. Investigate visible peaks in the chromatogram that were not identified in the data output. Manually integrate the peak if necessary. For manual integration refer to EMAX-DM01.
- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomers peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.[2]

10.4.3.2.1. For samples containing components not associated with the calibration standards, perform a library search for purposes of tentative[3] (TIC). Execute LSC (Chem Station program) to initiate the library search using NIST/EPA/MSDC mass spectral library. Visually inspect each extracted mass ion chromatograph to determine the identification of the unknown before final reporting following the guidelines below.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within + 20%.

  Example: for an ion with an abundance of 50% of the standard spectra, the corresponding sample ion abundance must be between 30 and 70%.
- Molecular ions present in reference spectrum but not present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting analytes.
- Ions present in the reference spectrum but not present in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of the background contamination or co-eluting analytes. Data system library reduction programs can sometimes create these discrepancies.

10.4.3.2.2. **Reporting TICs**

- If the library search produces a match at or above 85%, report the analyte.
- If the library search produces more than one analyte at or above 85%, report the first analyte (highest).

[2] SW846 Method 8270C, Section 7.6.1.4

[3] Library search is performed only when indicated in the PSR.

- If the library search produces no matches at or above 85%, the compound should be reported as unknown.

10.4.3.3. **Quantitation**

10.4.3.3.1. Apply the appropriate quantitation method (Section 10.3.3.6). Calculate the concentration of any positively identified target analyte using Eq.-10.5.3. Apply the dilution factor for diluted samples to calculate for the final concentration of the sample.

10.4.3.4. **Manual Integration**

10.4.3.4.1. Refer to EMAX-DM01, manual integration section.

10.4.3.5. **Dealing with Carryover**

10.4.3.5.1. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.

10.4.3.5.2. If there was no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.

10.4.3.5.3. If there is any target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule out carry over. If carry over is confirmed, proceed with data reduction and report the data from re-analysis.

10.4.3.5.4. To clean-up the system, run a blank sample. If improved result is noted repeat this process until no evidence of contamination is observed. Otherwise inform the Supervisor for further instruction.

10.5. **Calculations**

10.5.1. Initial Calibration

10.5.1.1. **Calculate for Relative Response Factor (RRF)**

$$RRF = \frac{(AX)(CIS)}{(AIS)(CX)} \qquad \text{Eq.-10.5.1.1}$$

where:

AX – Area of characteristic ion for the compound being measured

AIS – Area of characteristic ion for the specific internal standard

CX – Concentration of the compound being measured

CIS – Concentration of the specific internal standard

10.5.1.2. **Calculate for Average Relative Response Factor (RRFm)**

$$RRFm = \frac{\sum RRF}{n} \qquad \text{Eq-10.5.1.2}$$

where:

RRFm – average response factor

$\sum RRF$ – summation of response factors

n – number of measurements

10.5.1.3. **Calculate for Least Square Linear Regression**

$$y = ax + b \qquad \text{Eq.-10.5.1.3}$$

where:

y – Response Ratio *(AX/AIS)*

x – Amount Ratio (CX/CIS)

a – x1 = slope of the line

$$a = \frac{\sum\left(x - \bar{x}\right)\left(y - \bar{y}\right)}{\left(x - \bar{x}\right)^2}$$

where:

$\bar{x}$ – Average of amount ratios

$\bar{y}$ – Average of response ratios

b – x0 = intercept of the line

$$b = \bar{y} - a * \bar{x}$$

10.5.1.4. **Calculate for Inverse Weighting Factor**

$$y = ax + b \qquad \text{Eq.-10.5.1.4}$$

where:

y – Response Ratio *(AX/AIS)*

x – Amount Ratio *(CX/CIS)*

a – x1 = slope of the line

$$a = \frac{\sum\left[\left(x - x_a\right)\left(y - y_a\right)\right]}{\sum\left(x - x_a\right)^2}$$

where:

$$x_a = \sum\left[x\left(1/x\right)/\sum\left(1/x\right)\right]$$

$$y_a = \sum\left[y\left(1/x\right)/\sum\left(1/x\right)\right] \text{ or}$$

$$x_a = \sum\left[x\left(1/x^2\right)/\sum\left(1/x^2\right)\right]$$

$$y_a = \sum\left[y\left(1/x^2\right)/\sum\left(1/x^2\right)\right]$$

b = x0 = intercept of the line

$$b = y_a - a * x_a$$

10.5.1.5. **Calculate Inverse Quadratic**

$$y = ax^2 + bx + c \qquad \text{Eq.-10.5.1.5}$$

where:

- y – Resp_Ratio = **x0 + x1** * Amt_Ratio + **x2** * (Amt_Ratio)^2
- x – Amt_Ratio
- c – **x0** = Det 0/Det b
- b – **x1** = Det 1/Det b
- a – **x2** = Det 2/Det b

$$W_i = \frac{\frac{1}{x_i}}{\sum_{i=1}^{n}\frac{1}{x_i}}$$

where:

- X_i – amount ratio = Conc of Std/Conc of IS
- Y_i – response ratio = Resp of Std/Resp of IS
- **Wi** – 1/X)/SUM(1/X)
- **<X>** – SUM(W_i*X_i)
- **<Y>** – SUM(W_i*Y_i)
- **<XX>** – SUM(W_i*(X_i)^2)
- **<XXX>** – SUM(W_i*(X_i)^3)
- **<XXXX>** – SUM(W_i*(X_i)^4)
- **<YY>** – SUM(W_i*(Y_i)^2)
- **<XY>** – SUM(W_i*X_i*Y_i)
- **<XXY>** – SUM(W_i*(X_i)^2*Y_i)
- **<Yd2>** – SUM((Y_i-<Y>)^2*W_i)
- **Ye** – x0+x1*X_i+x2*X_i^2-<Y>)
- **<Ye2>** – SUM(Ye^2*W_i)

Det b	1	<X>	<XX>
	<X>	<XX>	<XXX>
	<XX>	<XXX>	<XXXX>

Det 0	1	<X>	<XX>
	<X>	<XX>	<XXX>
	<Y>	<XY>	<XXY>

Det 1	1	<X>	<XX>
	<Y>	<XY>	<XXY>
	<XX>	<XXX>	<XXXX>

Det 2	<Y>	<XY>	<XXY>
	<X>	<XX>	<XXX>
	<XX>	<XXX>	<XXXX>

r2= <Ye2>/<Yd2>

ccf2= $(r2)^{1/2}$

10.5.1.6. **Calculate for Standard Deviation (SD)**

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$ Eq.-10.5.1.6

where:

X_i – result at i^{th} measurement

$\bar{x}$ – mean

n – number of measurements

10.5.1.7. **Calculate for % relative standard deviation (%RSD)**

$$\%RSD = \frac{SD}{RRF_m} * 100\%$$ Eq.-10.5.1.7

where:

SD – standard deviation

RRFm – average response factor

10.5.1.8. **Calculate the Relative Retention Time (RRT)**

$$\text{RRT} = \frac{\text{Retention Time of the Analyte}}{\text{Retention Time of the Internal Standard}} \qquad \text{Eq.-10.5.1.8}$$

10.5.2. Calibration Check/Continuing Calibration

10.5.2.1. **Calculate Percent Difference (%D) when RRFm is used for quantitation**

$$\%D = \frac{\left[RRF_c - RRF_m\right]}{RRF_m} * 100\% \qquad \text{Eq.-10.5.2.1}$$

where:

RRFc – response factor from continuing calibration standard

RRFm – average response factor

10.5.2.2. **Calculate Percent Deviation ($\%D_t$) when applied calculation is other than ARF**

$$\%D_t = \frac{\left|T_t - T_f\right|}{T_t} * 100\% \qquad \text{Eq.-10.5.2.2}$$

where:

T_t – true value of standard in µg/L

T_f – found value of standard in µg/L

10.5.3. Calculation of Sample Concentration (Water and Soil/Sediment Samples). When a compound is identified, the quantitation of that compound shall be based on the integrated abundance from the EICP of the primary characteristic ion.

10.5.3.1. **Water Samples**

$$\text{Concentration (ug/L)} = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(V_t)} \qquad \text{Eq.-10.5.3.1}$$

where:

A_X – area of characteristic ion for the compound to be measured

I_S – amount of internal standard added

V_e – extract final volume from sample extraction, usually 1 mL

DF – dilution factor = $\frac{aliquot(\mu L) + solvent(\mu L)}{aliquot(\mu L)}$

A_{is} – area of characteristic ion for the internal standard

RRFm – average response factor

V_i – volume of extract injected in µL, usually 1 µL

V_t – volume of water extracted in mL, usually 1000 mL

10.5.3.2. **Soil/Sediment Samples (Dry weight basis)**

$$Concentration\left(\mu g / Kg\right) = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(W_s)(DW)} \qquad \text{Eq.-10.5.3.2}$$

where:

A_x – area of characteristic ion for the compound to be measured

I_s – amount of internal standard injected in ng

V_e – volume of extract in mL, usually 1 mL[4]

DF – dilution factor = $\frac{aliquot\left(\mu L\right) + solvent\left(\mu L\right)}{aliquot\left(\mu L\right)}$

A_{is} – area of characteristic ion for the internal standard

RRF_m – average response factor

V_i – volume of extract injected in µL, usually 1 µL

W_s – wet soil weight in kg

DW – % solid = $\frac{100 - \%moisture}{100}$

10.5.3.3. **Calculation for results subjected to cleanup shall have the following equations:**

$$Concentration(\mu g / Kg) = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(W_s)(DW)} \bullet \frac{V_{bg}}{V_{ig}} \qquad \text{Eq.-10.5.3.3.1}$$

$$Concentration(\mu g / L) = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(V_t)} \bullet \frac{V_{bg}}{V_{ig}} \qquad \text{Eq-10.5.3.3.2}$$

where:

A_X – area of characteristic ion for the compound to be measured

I_S – amount of internal standard injection in ng

V_e – volume of extract in mL

DF – dilution factor = $\frac{aliquot(\mu l) + solvent(\mu l)}{aliquot(\mu l)}$

A_{is} – area of characteristic ion for the compound to be measured

RRF_m – average response factor

V_i – volume of extract injected in µL, usually 1 µL

W_s – wet soil weight in Kg

[4] For extracts subjected to GPC V_i=0.5-mL

DW – %solid = $\frac{100 - \%moisture}{100}$

V_t – volume of water extracted in mL

V_{bg} – total volume of extract before GPC clean-up in mL

V_{ig} – injected volume of extracts to GPC in mL

10.5.4. Base all sample result calculations on the ICAL curve, e.g., area ratio of Ax/Ais versus concentration using inverse weighting factor fitted to the initial calibration is also used for determination of sample concentration.

10.5.5. Concentration of TIC is estimated by the same method as target compounds with the following assumptions:

10.5.5.1. The area "Ax" and "Ais" are derived from total ion chromatogram. "Ais" refers to the closest internal standard (IS) free of interference.

10.5.5.2. RRF of the TIC is 1.

10.5.6. Accuracy and Precision

10.5.6.1. **Percent Recovery**

$$\%\,\mathrm{Recovery} = \frac{\left(C_f - C\right)}{C_s} \times 100 \qquad \text{Eq.-10.5.6.1}$$

where:

C_f – concentration found

C – concentration of sample (use 0 for LCS)

C_s – concentration of spike

10.5.6.2. **Relative Percent Difference (%RPD)**

$$\%RPD = \frac{\left|C_1 - C_2\right|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \qquad \text{Eq.-10.5.6.2}$$

where:

C_1 – Measured concentration of the first sample aliquot

C_2 – Measured concentration of the second sample aliquot

10.5.7. DDT Degradation

$$\%B = \frac{A_{DDD} + A_{DDE}}{A_{DDT} + A_{DDD} + A_{DDE}}(100) \qquad \text{Eq.-10.5.7}$$

where:

$\%B$ – percent breakdown

ADDD – area of DDD

ADDE – area of DDE

ADDT – area of DDT

10.6. **Data Reduction**

10.6.1. Make a copy of the analytical run log and sample preparation log.

10.6.2. Highlight the data to be reported.

10.6.3. Print a copy of the raw data and the QC report.

10.6.4. Check that all positively identified analytes are within the calibration range.

10.6.5. Collate the reportable data separating the QC results from the sample results.

10.6.6. Keep all other data generated with the analytical folder marked with “For record only”.

10.7. **Report Generation**

10.7.1. Generate the method.txt file using WDB1C.exe

10.7.2. Generate the sample results using F1NV3C.exe or F1N3C4.exe

10.7.3. Generate the QC summary using QCV3CN.exe or QCV3CN4.exe

10.7.4. Generate the Instrument Performance Check (ICAL and DCC) using F5SVTEST.exe.

10.7.5. Generate the IS and RT Summary using F8SV.exe.

10.7.6. Generate Lab Chronicle using LABCHRN1.exe

10.7.7. Generate the Case Narrative using CN1.exe

10.7.8. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analyses.

10.7.8.1. Case Narrative

10.7.8.2. Lab Chronicle

10.7.8.3. Sample Results

10.7.8.4. Method Blank Results

10.7.8.5. LCS/LCSD Summary

10.7.8.6. MS/MSD Summary

10.7.8.7. Instrument Performance Check (ICAL)

10.7.8.8. ICAL Summary

10.7.8.9. ICV Summary

10.7.8.10. Instrument Performance Check (DCC)

10.7.8.11. IS and RT Summary

10.7.8.12. DCC Summary

10.7.8.13. Analytical Run Log

10.7.8.14. Sample Preparation Log

10.7.8.15. Non-Conformance Report (if any)

10.8. **Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

10.8.1.1. If any of the checkpoints below indicates a problem, re-analysis is required.

- ✓ Check internal standard area. They should be within -50 to +100% of the ICAL midpoint to be acceptable, otherwise follow PSR.
- ✓ Check retention time of each IS to the ICAL midpoint. They should be within ± 30 seconds to be acceptable, otherwise follow PSR.
- ✓ Check surrogate recoveries against Project Specific requirements (PSR). In the absence of PSR, default to in-house QC limits.
- ✓ Check concentrations of target analytes if calibration range is exceeded.

10.8.1.2. Review the attached log that they are properly filled.

10.8.1.3. Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.8.1.4. Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.2. Submit the analytical folder for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Perform instrument routine preventive maintenance and record on instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction that may affect data quality.

10.9.2. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality. The table below is a list of preventive maintenance activities that are essential to consider in performing this SOP.

Maintenance Activity	Description	Frequency
Autosampler	Inspect and clean syringe. Check autosampler response.	Daily prior to analysis
Vacuum System Verification	Verify pressure. Perform system tune check	Daily prior to analysis
Verification	Check instrument parameters to ensure normal operating conditions. Check instrument performance	Daily prior to analysis

	(e.g., Daily calibration check, instrument blank, DDT/Endrin breakdown).	
Source Cleaning	Remove and clean the Mass Spec ion source	Every 6 months or as necessary
Vacuum System Maintenance	Inspect vacuum pumps, and replace mechanical/diffusion pump oil	Every 6 months
Documentation	Record maintenance in instrument service logs	Daily prior to analysis to include services done by third party.

11.0 QUALITY CONTROL

11.1. Analytical Batch QC

11.1.1. Perform tune check to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to calibration and check for any contamination.

11.1.2. Perform Initial Calibration (ICAL) to establish a calibration curve for the quantification of the analytes of interest. Obtain secondary review before using the new ICAL.

11.1.3. Establish Retention Time Window position for each analyte every after ICAL for proper qualitative identification.

11.1.4. Perform Initial Calibration Verification (ICV) every after ICAL to verify accuracy of ICAL.

11.1.5. Perform Continuing Calibration Verification (CCV) every 12 hours to verify that the instrument response is reliable, and has not changed significantly from the current ICAL curve.

11.1.6. Evaluate Relative Retention Time for each analytes in every sample to be within ± 0.06 RRT units.

11.1.7. Verify Internal Standards (IS) for quantitatively accuracy and that its Retention Time is within ± 30 seconds from retention time of the midpoint standard in the ICAL and EICP area is within -50 to +100% of ICAL midpoint standard.

11.1.8. Evaluate Surrogate recovery to monitor instrument response on every sample.

11.2. Sample Preparation Batch QC

11.2.1. Analyze Method Blank (MB) to demonstrate that preparation of sample was free from contamination.

11.2.2. Analyze Lab Control Sample (LCS) to assess preparative batch accuracy and precision.

11.2.3. Analyze Matrix Spike (MS/MSD) to assess matrix interference.

11.2.4. Properly treat lab wares used in the sample preparation as specified in EMAX-QC07.

11.2.5. Solvents and reagents must undergo quality control check in the stationary laboratory prior to its use. Refer to EMAX-QC01 for details.

- Verify that the spike amount added is accurate by checking the record.

- If LCS is within acceptance criteria and the right amount of spike is added into the sample then it is indicative of matrix interference. Discuss the probable matrix interference in the case narrative.

11.3. **Method QC**

11.3.1. All analytes reported must have a valid DL, LOD and LOQ as described in EMAX-QA04.

11.3.2. All analysts conducting this analysis must demonstrate capability (IDOC/DOC) as described in EMAX-QA05.

11.4. Refer to Appendix 1 for all related Quality Control parameters, frequency and acceptance criteria.

12.0 CORRECTIVE ACTIONS

12.1. Corrective action for each Quality Control Procedures is summarized in Appendix 1.

12.2. **Sample Preparation QC**

12.2.1. If the instrument blank is non-compliant, consider the following suggestions to correct the problem:

12.2.1.1. Rule out instrument contamination by performing the instrument daily maintenance, such as changing septum, cleaning liner, cleaning or using new autosampler syringe.

12.2.1.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.

12.2.1.3. Rule out preparation contamination by preparing a new instrument blank.

12.2.1.4. If the problem persists, inform the supervisor for further advice.

12.2.2. If method blank is non-compliant, consider the following suggestions to correct the problem:

12.2.2.1. Rule out instrument contamination by checking instrument blank.

12.2.2.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01.

12.2.2.3. Rule out glassware contamination used for extraction as described in EMAX-QC07.

12.2.2.4. Re-extract MB and the associated samples with reagents free of contamination or with newly opened reagents.

12.2.2.5. If problem persists, inform the supervisor for further advice.

12.2.3. If LCS is non-compliant, consider the following suggestions to correct the problem:

12.2.3.1. If result is bias-high or bias-low, check the LCS Standard by analyzing at the spike level.

12.2.3.2. If LCS check is within 80-120% of expected value, check the calibration of the micropipette or syringe used for spiking. Re-extract and re-analyze the LCS and the associated samples.

12.2.3.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS standard, re-extract and re-analyze LCS and the associated samples.

12.2.3.4. If LCS is within acceptance then and the right amount of spike is added into sample then it is indicative of matrix interference. Discuss the probable matrix interference in Case Narrative.

12.2.4. If MS is non-compliant, consider the following suggestions to correct the problem:

12.2.4.1. Verify from the sample preparation log that the spike amount added is correct.

12.2.4.2. If LCS is within acceptance criteria and the right amount of spike is added into the sample then it is indicative of matrix interference. Discuss the probability of matrix interference in the case narrative.

12.3. **Analytical Batch QC**

12.3.1. Tune Check

12.3.1.1. If tune check is non-compliant, consider the following suggestion to correct the problem:

- Check the instrument settings and make sure that the instrument parameters are properly set-up.
- Check gas flow.
- Perform auto-tune or visual optimization.
- If the problem persists, inform the supervisor for further actions.

12.3.1.2. If instrument performance is non-compliant, consider the following suggestion to correct the problem:

- Excessive degradation of DDT and/or poor chromatography demonstrated by too much tailing are indications of dirty injection port. Clean or replace the injection port. If problem persist, cut off the first 6-12 inches of the capillary column.

12.3.2. Initial Calibration (ICAL)

12.3.2.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:

- If RSD% is out of acceptance criteria, review result and identify presence of an outlier.
- If one of the standard return a bias-low or bias-high on all of the analytes, then the point is considered an outlier, prepare a standard at the ICAL point and re-analyze.
- If the highest ICAL point appears to be saturated, drop the highest point.
- If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.

Note: The lowest calibration identifies the limit of quantitation (LOQ). Therefore, check that the LOQ is in conformance to the current projects where the ICAL will be used.

12.3.2.2. If instrumentation problem is suspected, consider the following suggestions to correct the problem:

- Check the connections and make sure they are air-tight and perform maintenance as needed.
- Check the gas flow.
- Re-tune the MS.
- Prepare a fresh standard and repeat calibration.
- Clean the MS source and repeat the calibration.

12.3.2.3. If the problem persists, inform the supervisor for further action.

12.3.3. Initial Calibration Verification (ICV)

12.3.3.1. If the ICV is non-compliant, consider the following suggestions to correct the problem:

- Re-analyze ICV to rule out poor injection.
- If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration.
- If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration.
- If problem persist, inform the supervisor for further action.

12.3.4. Daily Calibration Check (DCC)

12.3.4.1. If DCC is non-compliant consider the following suggestions to correct the problem:

- If majority of the analyte response are low and no evidence of leak in the system is apparent, it is indicative of a poor injection or leak in the vial. Re-analyze DCC.
- If problem persist, rule out standard degradation. Prepare a fresh standard and repeat DCC.

12.3.4.2. If Continuing Calibration is non-compliant consider the following suggestions to correct the problem:

- Change the liner.
- Clean the injection port.
- Prepare a new standard.
- Cut or replace column.
- Rule out leaks by checking all connections.
- If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL.

12.3.5. Instrument Blank: If instrument blank is non-compliant, consider the following suggestions to correct the problem:

- Rule out instrument contamination.

- Rule out reagent contamination by testing each reagent used as described in EMAX-QC01.
- Rule out vials and glassware contamination as described in EMAX-QC07.
- If the problem persists, inform the supervisor for further advice.

12.4. Discuss in the case narrative, water samples that have residual chlorine above acceptable limits.

12.5. Execute a Non-Conformance Report (NCR) when the following circumstances occur:

12.5.1. If corrective action needs the function of the department; e.g. if the sample needs to be re-extracted, refer to EMAX-QA08 for details of completing an NCR.

12.5.2. If corrective action needs the assistance of the project manager; e.g. if the sample is non-compliant to the technical holding time requirement, insufficient amount of sample, or other non-conforming issues.

12.6. For other problems encountered, inform the supervisor immediately for further instruction.

13.0 POLLUTION PREVENTION

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2. Prepare all standards in fume hoods.

14.0 WASTE MANAGEMENT

14.1. No samples shall be dumped on the laboratory sink.

14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.

14.3. Place all waste generated during analytical process in properly labeled satellite waste containers for proper collection.

14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. **Definition of Terms**

15.1.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.

15.1.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

15.1.2.1. **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.

15.1.2.2. **Analytical Batch** – is composed of prepared samples (extracts, digestates or concentrates) which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples

originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.3. Detection Limit (DL) – The lowest concentration or amount of the target analyte that can be identified, measured and reported with confidence that the analyte concentration is not false positive.

15.1.4. Limit of Detection (LOD) – An estimate of the minimum amount of substance that an analytical process can reliably detect.

15.1.5. Limit of Quantitation (LOQ) – The minimum levels, concentrations or quantities of target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

15.1.6. Safety Data Sheet (SDS) – is where the physical data, toxicology and safety precaution of a certain substance is listed.

15.1.7. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.8. Carry-over – are contaminants retained in the instruments/apparatus from a highly contaminated sample that is passed into the succeeding sample(s).

15.1.9. Calibration Check Compounds (CCC) – evaluate the integrity of the system. Variability of these compounds may indicate system leak or reactive sites in the column.

15.1.10. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.1.11. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.12. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.

15.1.13. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.

15.1.14. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.

15.1.15. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

15.1.16. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.17. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.18. Matrix – is a component or form of a sample.

15.1.19. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.

15.1.20. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

15.1.21. Response Factor – is the ratio of the peak area of the target compound in the sample or sample extract.

15.1.22. Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard used to evaluate analytical efficiency be measuring recovery. Compounds not expected to be expected to be detected in environmental media.

15.1.23. System Performance Check Compounds (SPCC) – are compounds that are used to check compound stability and to check for degradation cause by contaminated lines or active sites in the system.

15.2. **Application of QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing Semi Volatile analysis by GC/MS unless otherwise other directive is specified by the project requirements.

15.3. **Department of Defense (DoD) and Department of Energy Projects**

15.3.1. Samples from DoD and DoE sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, is applied.

16.0 REFERENCES

16.1. "Test Methods for Evaluation of Solid Wastes", EPA SW846, Method 8270C as updated

16.2. EMAX Quality Systems Manual, as updated

17.0 APPENDICES

17.1. **Tables**

17.1.1. Table 1 Intermediate Standard Preparation

A) Primary Source B) Secondary Source

17.1.2. Table 2A Working Standard Preparation

17.1.3. Table 2B Working Standard Preparation for Benzidine

17.1.4. Table 2C Working Standard Preparation for Additional Analytes (Appendix IX)

17.1.5. Table 3A Working Secondary Source Standard

17.1.6. Table 3B Working Secondary Source Standard for Benzidine

17.1.7. Table 3C Working Secondary Source Standard for Additional Analytes "Appendix IX"

17.1.8. Table 4 Working Internal Standard Preparation

17.1.9. Table 5 Working GC/MS Tuning (DFTPP) Standard

17.1.10. Table 6 Instrument Parameters

17.1.11. Table 7 DFTPP Key Ions and Ion Abundance Criteria for 8270C

17.1.12. Table 8 Typical Analyte List, Quantitation Ions, IS, Surrogates, Calibration Standards and Detection Limits

17.1.13. Table 9 Typical Analyte List, Quantitation Ions, IS, Surrogates, Calibration Standards and Detection Limits for Appendix IX

17.1.14. Table 10 Calibration Check Compounds (CCCs)

17.1.15. Table 11 System Performance Calibration Check Compounds (SPCCs)

17.2. **Figures**

17.2.1. Figure 1 Peak Evaluation Technique

17.2.2. Figure 2 Typical Chromatogram

17.2.3. Figure 2A Typical Chromatogram of Benzidine

17.2.4. Figure 2B Typical Chromatogram of Additional Analytes “Appendix IX”

17.2.5. Figure 3 Typical ICAL Summary

17.2.6. Figure 3A Typical ICAL Summary of Benzidine

17.2.7. Figure 3B Typical ICAL Summary of Appendix IX

17.2.8. Figure 3C Typical ICAL Summary of Additional Analytes Appendix IX

17.2.9. Figure 4 Typical DFTPP Tune Summary “8270C”

17.2.10. Figure 5 Typical Instrument Performance Check Summary “8270C”

17.2.11. Figure 6 Typical Continuing Calibration Summary

17.2.12. Figure 6A Typical Continuing Calibration Summary of Benzidine

17.2.13. Figure 6B Typical Continuing Calibration Summary of “Appendix IX”

17.2.14. Figure 6C Typical Continuing Calibration Summary of Additional Analytes “Appendix IX”

17.2.15. Figure 7A Typical Internal Standard and Retention Time Summary SV-1

17.2.16. Figure 7B Typical Internal Standard and Retention Time Summary SV-2

17.2.17. Figure 8 Typical Sample Result Summary

17.2.18. Figure 9 Typical LCS/LCSD Report Summary

17.2.19. Figure 10 Typical MS/MSD Report Summary

17.2.20. Figure 11 Typical Case Narrative

17.3. **Appendices**

17.3.1. Appendix 1 Summary of Quality Control Procedures

17.3.2. Appendix 2 Demonstration of Capability

17.3.3. Appendix 3 Demonstration of Capability for Appendix IX Compounds

17.4. **Forms**

17.4.1. 8270FS Sample Preparation Log

17.4.2. 8270FA Analytical Run Log

17.4.3. 8270FM Instrument Maintenance Log

Table 1: INTERMEDIATE STANDARD PREPARATION

A. Primary Source: Restek, CPI, AccuStandard or equivalent

Standard Name		Stock / Internal Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/mL)
				Aliquot (µL)	Dil. Solution	Final Vol. (mL)	
Standard List	8270 Mega Mix	1000	Restek	1000	MeCl2	5	200
	8270 Custom Std.	2000	CPI	500	MeCl2	5	200
	Benzidine Mix	2000	Restek	500	MeCl2	5	200
	Benzoic Mix	2000	Restek	500	MeCl2	5	200
	Acid Surrogate Mix	7500	Restek	133.3	MeCl2	5	200
	Base/Neutral Surrogate Mix	5000	Restek	200	MeCl2	5	200
Appendix IX	Appendix IX Mix # 1	2000	Restek	300	MeCl2	5	120
	Appendix IX Mix # 2	1000	Restek	600	MeCl2	5	120
	EPA 8270 Organophosphorous Pesticides Mix	2000	Restek	300	MeCl2	5	120
	Cust. 8270 Std.	2000	AccuStandard	300	MeCl2	5	120
	Acid Surrogate Mix	7500	Restek	80	MeCl2	5	120
	Base/Neutral Surrogate Mix	5000	Restek	120	MeCl2	5	120

B. Secondary Source: CPI, AccuStandard, Restek or equivalent

Standard Name		Stock / Internal Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/mL)
				Aliquot (µL)	Dil. Solution	Final Vol. (mL)	
Standard List	Custom Semi Volatile Standard	2000	CPI	NA	NA	NA	NA
	8270 LCS Solution	200	CPI				
	8270 Internal Standard	2000	AccuStandard				
	Benzidine	5000	Ultra				
Appendix IX	Appendix IX Mix # 1*	2000	Restek	300	MeCl2 / MeOH	5	120
	Appendix IX Mix # 2*	1000	Restek	600	MeCl2 / MeOH	5	120
	EPA 8270 Organophosphorous Pesticides Mix*	2000	Restek	300	MeCl2 / MeOH	5	120
	Cust. 8270 Std.	2000	CPI	300	MeCl2 / MeOH	5	120

*Different Lot

Table 1 (cont.): INTERMEDIATE STANDARD PREPARATION

a. Primary Source: Restek, SPEXertificate, AccuStandard, Ultra Scientific or equivalent

Standard Name		Stock / Internal Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/mL)
				Aliquot (µL)	Dil. Solution	Final Vol. (mL)	
Standard List	Benzidine	1000	Restek	NA	NA	NA	NA
Appendix IX Additional	Kepone	2000	SPEXertificate	750	MeCl2	5	300
	Famphur	2000	AccuStandard	750	MeCl2	5	300
	Hexacholoropene	5000	Ultra Scientific	300	MeCl2	5	300

b. Secondary Source: CPI, Supelco or equivalent

Standard Name		Stock / Internal Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/mL)
				Aliquot (µL)	Dil. Solution	Final Vol. (mL)	
Appendix IX Additional	Kepone	2000	CPI	750	MeCl2 / MeOH	5	300
	Famphur	2000	CPI	750	MeCl2 / MeOH	5	300
	Hexacholoropene	5000	Supelco	300	MeCl2 / MeOH	5	300

Table 2A: INITIAL CALIBRATION

WORKING STANDARD PREPARATION

Standard Name	Intermediate Standard µL of 200 mg/L	Internal Standard µL of 2000 mg/L	Dil. Solvent	Final Volume (µL)	Final Conc.
1	10	10	$MeCl_2$	500	4 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
2	25	10	$MeCl_2$	500	10 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
3	50	10	$MeCl_2$	500	20 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
4	62.5	10	$MeCl_2$	500	25 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
5	75	10	$MeCl_2$	500	30 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
6	100	10	$MeCl_2$	500	40 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
7	125	10	$MeCl_2$	500	50 µg/mL of Cal. Std. 40 µg/mL of Internal Std.

Table 2B: INITIAL CALIBRATION

WORKING STANDARD PREPARATION FOR BENZIDINE

Standard Name	Intermediate Standard µL of 1000 mg/L	Internal Standard µL of 2000 mg/L	Dil. Solvent	Final Volume (µL)	Final Conc.
1	2	10	$MeCl_2$	500	4 µg/mL of Benzidine Std. 40 µg/mL of Internal Std.
2	5	10	$MeCl_2$	500	10 µg/mL of Benzidine Std. 40 µg/mL of Internal Std.
3	12.5	10	$MeCl_2$	500	25 µg/mL of Benzidine Std. 40 µg/mL of Internal Std.
4	20	10	$MeCl_2$	500	40 µg/mL of Benzidine Std. 40 µg/mL of Internal Std.
5	25	10	$MeCl_2$	500	50 µg/mL of Benzidine Std. 40 µg/mL of Internal Std.
6	40	10	$MeCl_2$	500	80 µg/mL of Benzidine Std. 40 µg/mL of Internal Std.
7	50	10	$MeCl_2$	500	100 µg/mL of Benzidine Std. 40 µg/mL of Internal Std.

Table 2C: INITIAL CALIBRATION

WORKING STANDARD PREPARATION

FOR ADDITIONAL ANALYTES (APPENDIX IX)

Appendix IX Standard List

Standard Name	Intermediate Standard µL of 120 mg/L	Internal Standard µL of 2000 mg/L	Dil. Solvent	Final Volume (µL)	Final Conc.
1	16.7	10	$MeCl_2$	500	4 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
2	41.6	10	$MeCl_2$	500	10 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
3	83.3	10	$MeCl_2$	500	20 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
4	104.2	10	$MeCl_2$	500	25 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
5	125	10	$MeCl_2$	500	30 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
6	166.7	10	$MeCl_2$	500	40 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
7	208.3	10	$MeCl_2$	500	50 µg/mL of Cal. Std. 40 µg/mL of Internal Std.

Appendix IX Additional

Standard Name	Intermediate Standard µL of 300 mg/L	Internal Standard µL of 2000 mg/L	Dil. Solvent	Final Volume (µL)	Final Conc.
1	33.3	10	$MeCl_2$	500	20 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
2	66.6	10	$MeCl_2$	500	40 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
3	83.3	10	$MeCl_2$	500	50 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
4	133.3	10	$MeCl_2$	500	80 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
5	166.6	10	$MeCl_2$	500	100 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
6	200	10	$MeCl_2$	500	120 µg/mL of Cal. Std. 40 µg/mL of Internal Std.
7	233.3	10	$MeCl_2$	500	140 µg/mL of Cal. Std. 40 µg/mL of Internal Std.

Table 3A: WORKING SECONDARY SOURCE STANDARD

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (µL)	Dil. Solution	Final Vol. (µL)	
Custom Semi Volatile Standard	2000	6.25	$MeCl_2$	500	25
8270 LCS Solution	200	62.5	$MeCl_2$	500	25
8270 Internal Standard	2000	10	$MeCl_2$	500	40

Table 3B: WORKING SECONDARY SOURCE STANDARD FOR BENZIDINE

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (µL)	Dil. Solution	Final Vol. (µL)	
Benzidine	5000	2.5	$MeCl_2$	500	25
8270 Internal Standard	2000	10	$MeCl_2$	500	40

Table 3C: WORKING SECONDARY SOURCE STANDARD FOR ADDITIONAL ANALYTES APPENDIX IX

Appendix IX Standard List

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (µL)	Dil. Solution	Final Vol. (µL)	
Appendix IX Spike	120	104.2	$MeCl_2$	500	25
8270 Internal Standard	2000	10	$MeCl_2$	500	40

Appendix IX Additional

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (µL)	Dil. Solution	Final Vol. (µL)	
Appendix IX Add. Spike	300	83.3	$MeCl_2$	500	50
8270 Internal Standard	2000	10	$MeCl_2$	500	40

Table 4: WORKING INTERNAL STANDARD PREPARATION

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (mL)	Dil. Solution	Final Vol. (mL)	
Internal Standard Mix	4000	5	$MeCl_2$	10	2000

Table 5: WORKING GC/MS TUNING (DFTPP) STANDARD

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (µL)	Dil. Solution	Final Vol. (µL)	
GC/MS Tuning Mix	1000	25	$MeCl_2$	500	50

Table 6: INSTRUMENT PARAMETERS

	Inst. E4 / Inst. E7	Inst. E9 / Inst. F0
Carrier Gas	Helium at 100 psi at outlet	Helium at 100 psi at outlet
Column head pressure	15 - 35 psi at 40°C	15-35 psi at 45°C
Injection port temperature	280-300°C	200-250°C
Interface	Direct column interference at 280-300°C	Direct column interference at 280-300°C
Valve time	Split 0.2 minute	Split 0.2 minutes
Oven Temperature Program		
Initial Temperature	50°C/min; hold for 0min.	45°C/min; hold for 0.2min.
Rate	30°C/min to 100°C; hold for 0.0 min.; 20°C/min to 200°C; hold for 0.0 min.; 25°C/min to 320°C; hold for 2.53 min.	20°C/min to 320°C; hold for 4 min.
Run Time	14 minutes	18 minutes
Scan Parameters		
Scan start time	After solvent peak	After solvent peak
Mass range	40 to 550 AMU	40 to 550 AMU
Multiplier voltage	1000 - 3000	1000 – 3000
Number of sampling rate	1 - 2	1 – 2
Threshold	0 - 300	0 -300
Tuning File	DFTPP	DFTPP

Table 7: DFTTPP KEY IONS ABUNDANCE CRITERIA FOR 8270C

Mass	Ion Abundance Criteria
51	30 – 60% of mass 198
68	< 2.0% of mass 69
69	Present
70	< 2.0% of mass 69
127	40 – 60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 – 30% of mass 198
365	> 1% of mass 198
441	Present but < mass 443
442	> 40% of mass 198
443	17 – 23% of mass 442

Table 8: TYPICAL TARGET ANALYTE LIST

Analyte	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7	8			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
Acenaphthene	153	152	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Acenaphthylene	152	151	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Aniline	93	66	IS1	Sur1/2	4	10	20	25	40	50			25	40	5.3	10	20	µg/L	83	167	667	µg/Kg
Anthracene	178	152	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Azobenzene*	77	105	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	96	167	333	µg/Kg
Benzo(a)anthracene	228.1	113	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzo(a)pyrene	252.1	125	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzo(b)fluoranthene	252.1	124.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.6	5	10	µg/L	86	167	333	µg/Kg
Benzo(e)pyrene	252.1	125	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzo(g,h,i)perylene	276.1	137.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	87	167	333	µg/Kg
Benzo(k)fluoranthene	252.1	124.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzoic acid	105	77	IS2	Sur3	4	10	20	25	40	50	80	100	25	80	10	20	40	µg/L	333	667	1333	µg/Kg
Benzyl alcohol	108	79	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Biphenyl	154	153	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis (2-Chloroethoxy) methane	92.9	94.9	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis (2-Chloroethyl) ether	92.9	62.9	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis (2-chloroisopropyl) ether	45	76.9	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis(2-ethylhexyl)adipate	129	57	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	87	167	333	µg/Kg
bis(2-Ethylhexyl)phthalate	149	167	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	115	167	333	µg/Kg
4-Bromophenyl-phenylether	247.9	250	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	90	167	333	µg/Kg
Butylbenzylphthalate	149	91	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Carbazole	167	139	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	89	167	333	µg/Kg
4-Chloro-3-methylphenol	107	77	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Chloroaniline	127	129	IS2	Sur3	4	10	20	25	40	50			25	40	4.2	5	10	µg/L	83	167	333	µg/Kg
2-Chloronaphthalene	162	164	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Chlorophenol	127.9	64	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Chlorophenyl-phenylether	204	206	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Chrysene	228.1	226.1	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dibenzo(a,h)anthracene	278.1	139	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dibenzofuran	168	139	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,2-Dichlorobenzene	145.9	147.9	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,3-Dichlorobenzene	145.9	147.9	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,4-Dichlorobenzene	145.9	147.9	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
3,3'-Dichlorobenzidine	252	254	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	84	167	333	µg/Kg
2,4-Dichlorophenol	161.9	163.9	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Diethylphthalate	149	177	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,6-Dimethylnaphthalene	156	141	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,4-Dimethylphenol	107	122	IS2	Sur3	4	10	20	25	40	50			25	40	2.6	5	10	µg/L	83	167	333	µg/Kg

Table 8: TYPICAL TARGET ANALYTE LIST

Analyte	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7	8			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
3,4-Dimethylphenol	107	122	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dimethylphthalate	163	164	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Di-n-butylphthalate	149	150	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	97	167	333	µg/Kg
4,6-Dinitro-2-methylphenol	198	121	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,3-Dinitrobenzene	168	75	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,4-Dinitrophenol	184	106.9	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	86	167	333	µg/Kg
2,4-Dinitrotoluene	165	89	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,6-Dinitrotoluene	165	89	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Di-n-octylphthalate	149	150	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	97	167	333	µg/Kg
Fluoranthene	202	100.9	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	126	167	333	µg/Kg
Fluorene	166	165	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachlorobenzene	283.8	141.9	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachlorobutadiene	224.8	222.8	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachlorocyclopentadiene	236.8	234.8	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachloroethane	116.8	200.8	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Indeno(1,2,3-cd)pyrene	276.1	137.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Isophorone	82	138	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1-Methylnaphthalene	142	141	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Methylnaphthalene	142	141	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1-Methylphenanthrene	192.1	165	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Methylphenol	107	108	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Methylphenol	107	108	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Naphthalene	128	129	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Nitroaniline	65	92	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
3-Nitroaniline	138	92	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Nitroaniline	138	92	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	120	167	333	µg/Kg
Nitrobenzene	77	123	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Nitrophenol	139	65	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Nitrophenol	109	139	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	106	167	667	µg/Kg
N-nitrosodimethylamine	74	42	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-Nitroso-di-n-propylamine	70	42	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-Nitrosodiphenylamine	169	168	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	153	167	333	µg/Kg
Pentachlorophenol	265.8	164.9	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	667	µg/Kg
Perylene	252.1	125	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	86	167	333	µg/Kg
Phenanthrene	178	152	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Phenol	94	65	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Pyrene	202	100.9	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	160	167	333	µg/Kg
Pyridine	79	52	IS1	Sur1/2	4	10	20	25	40	50	80	100	25	80	11	20	40	µg/L	333	667	1333	µg/Kg
2,3,4,6-Tetrachlorophenol	231.8	229.8	IS3	Sur4	4	10	20	25	40	50			25	40	3.6	5	10	µg/L	100	167	333	µg/Kg

Table 8: TYPICAL TARGET ANALYTE LIST

Analyte	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7	8			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
1,2,4-Trichlorobenzene	179.9	181.9	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,3,4-Trichlorophenol	195.9	96.9	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,3,5-Trichlorophenol	195.9	96.9	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	165	167	333	µg/Kg
2,4,5-Trichlorophenol	195.9	197.9	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	91	167	333	µg/Kg
2,4,6-Trichlorophenol	195.9	197.9	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,3,5-Trimethylnaphthalene	171.1	155	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzidine	184	92	IS1/4	Sur5	4	10	25	40	50	80	100		25	80	10	20	50	µg/L	863	867	1333	µg/Kg
2-Fluorophenol (Sur1)	112	64	IS1		4	10	20	25	40	50			25	40	1.5	3	5	µg/L	61	100	167	µg/Kg
Phenol-d5 (Sur2)	99	71	IS1		4	10	20	25	40	50			25	40	1.5	3	5	µg/L	52	100	167	µg/Kg
Nitrobenzene-d5 (Sur3)	82	128	IS2		4	10	20	25	40	50			25	40	0.59	1	1.67	µg/L	17	33	56	µg/Kg
2-Fluorobiphenyl (Sur4)	172	171	IS3		4	10	20	25	40	50			25	40	0.5	1	1.67	µg/L	24	33	56	µg/Kg
2,4,6-Tribromophenol (Sur5)	329.8	331.8	IS4		4	10	20	25	40	50			25	40	1.5	3	5	µg/L	55	100	167	µg/Kg
Terphenyl-d14 (Sur6)	244.2	122	IS5		4	10	20	25	40	50			25	40	0.54	1	1.67	µg/L	24	33	56	µg/Kg
1,4-Dichlorobenzene-d4 (IS1)	151.9	115																				
Naphtalene-d8 (IS2)	136	68																				
Acenaphthene-d10 (IS3)	164.1	162.1																				
Phenanthrene-d10 (IS4)	188.1	94																				
Chrysene-d12 (IS5)	240.1	120																				
Perylene-d12 (IS6)	264.1	130																				

*Dissociated to 1,2-Diphenylhydrazine

Table 9: **TYPICAL TARGET ANALYTE LIST FOR APPENDIX IX**

Appendix IX Compound	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)							ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
a,a-dimethylphenethylamine	58	91	IS2	Sur3	4	10	20	25	30	40	50	25	40	13	20	40	µg/L	NA	NA	NA	µg/Kg
Acetophenone	77	120	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Acetylaminofluorene	181	223.1	IS6	Sur6	4	10	20	25	30	40	50	25	40	2.7	5	10	µg/L	83	167	333	µg/Kg
4-Aminobiphenyl	169	115	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	85	167	333	µg/Kg
Aramite	184.9	62.9	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Atrazine	200	215	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Biphenyl	154	76	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Chlorobenzilate	251	138.9	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1-Chloronaphthalene	162	127	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Diallate	86	234	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	100	167	333	µg/Kg
Dibenzo(a,j)acridine	279.1	139.3	IS6	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,6-Dichlorophenol	161.9	63	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dimethoate	86.9	124.9	IS4	Sur5	4	10	20	25	30	40	50	25	40	3	5	10	µg/L	89	167	333	µg/Kg
p-Dimethylaminoazobenzene	120	225.1	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
7,12-Dimethylben(a)anthracene	256.1	241.1	IS6	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
3,3-Dimethylbenzidine	212.1	196	IS5	Sur6	4	10	20	25	30	40	50	25	40	5	10	20	µg/L	130	167	667	µg/Kg
3,4-Dimethylphenol	107	122	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,3-Dinitrobenzene	167.9	75.9	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dinoseb	211	162.9	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,4-Dioxane	88	57.9	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Diphenyl ether	170	141	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Disulfoton	88	96.9	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Ethyl methacrylate	69	41	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Ethyl methanesulfonate	78.9	108.9	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Ethyl parathion	96.9	108.9	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachloropropene	212.8	140.8	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Isodrin	192.9	262.8	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Isosafrole	162	104	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Methapyrilene	58	96.9	IS5	Sur6	4	10	20	25	30	40	50	25	40	10	20	40	µg/L	NA	NA	NA	µg/Kg
3-Methylchlolanthrene	268.1	252.1	IS6	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Methyl methanesulfonate	79.9	78.9	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Methyl parathion	108.9	263	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,4-Naphthoquinone	158	130	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1-Naphthylamine	143	115	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Naphthylamine	143	115	IS3	Sur4	4	10	20	25	30	40	50	25	40	10	10	20	µg/L	330	333	667	µg/Kg
N-nitrosodiethylamine	102	56	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-nitrosomethylethylamine	88	42	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg

Table 9: **TYPICAL TARGET ANALYTE LIST FOR APPENDIX IX**

Appendix IX Compound	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)							ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
N-nitrosomorpholine	56	116	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-nitrosodi-n-butylamine	84	57	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-nitrosopiperdine	114	42	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-nitrosopyrrolidine	100	68	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
5-Nitro-o-toluidine	152	77	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.6	5	10	µg/L	83	167	333	µg/Kg
4-Nitroquinoline-N-oxide	89	190	IS5	Sur6	4	10	20	25	30	40	50	25	40	5	10	20	µg/L	83	167	333	µg/Kg
Pentachlorobenzene	249.8	107.9	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Pentachloroethane	166.8	116.8	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Pentachloronitrobenzene	236.8	294.8	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Phenacetin	108	179	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
p-phenylenediamine	80	107.9	IS2	Sur3	4	10	20	25	30	40	50	25	40	10	20	40	µg/L	83	167	667	µg/Kg
Phorate	75	121	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Picoline	93	65.9	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Pronamide	172.9	174.9	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Safrole	162	131	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Sulfotepp	322	201.9	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,2,4,5-Tetrachlorobenzene	215.8	178.9	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Thionazin	107	96.9	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
o-Toluidine	106	77	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
O,O,O-triethyl phosphorothioate	121	198	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,3,5-Trinitrobenzene	212.9	119.9	IS4	Sur5	4	10	20	25	30	40	50	25	40	5	10	20	µg/L	83	167	333	µg/Kg
Famphur	218	124.9	IS1-IS5	Sur6	20	40	50	80	100	120	140	25	40	10	20	50	µg/L	420	833	1667	µg/Kg
Kepone	271.8	273.8	IS1-IS5	Sur6	20	40	50	80	100	120	140	25	40	10	20	50	µg/L	420	833	1667	µg/Kg
Hexachlorophene	195.9	197.9	IS6	Sur6	20	40	50	80	100	120	140	25	40	10	20	80	µg/L	630	833	2670	µg/Kg
2-Fluorophenol (Sur1)	112	64	IS1		4	10	20	25	30	40	50	25	40	1.5	3	5	µg/L	61	100	167	µg/Kg
Phenol-d5 (Sur2)	99	71	IS1		4	10	20	25	30	40	50	25	40	1.5	3	5	µg/L	52	100	167	µg/Kg
Nitrobenzene-d5 (Sur3)	82	128	IS2		4	10	20	25	30	40	50	25	40	0.59	1	1.67	µg/L	17	33	56	µg/Kg
2-Fluorobiphenyl (Sur4)	172	171	IS3		4	10	20	25	30	40	50	25	40	0.5	1	1.67	µg/L	24	33	56	µg/Kg
2,4,6-Tribromophenol (Sur5)	329.8	331.8	IS4		4	10	20	25	30	40	50	25	40	1.5	3	5	µg/L	55	100	167	µg/Kg
Terphenyl-d14 (Sur6)	244.2	122	IS5		4	10	20	25	30	40	50	25	40	0.54	1	1.67	µg/L	24	33	56	µg/Kg
1,4-Dichlorobenzene-d4 (IS1)	151.9	115																			
Naphtalene-d8 (IS2)	136	68																			
Acenaphthene-d10 (IS3)	164.1	162.1																			
Phenanthrene-d10 (IS4)	188.1	94																			
Chrysene-d12 (IS5)	240.1	120																			
Perylene-d12 (IS6)	264.1	130																			

Table 10: CALIBRATION CHECK COMPOUNDS (CCCs)

Base/Neutral Fraction	Acid Fraction
Acenaphtene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadine	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Table 11: SYSTEM PERFORMANCE CALIBRATION CHECK COMPOUNDS (SPCCs)

Base/Neutral Compounds	Acid Compounds
N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

Figure 1: PEAK EVALUATION TECHNIQUE

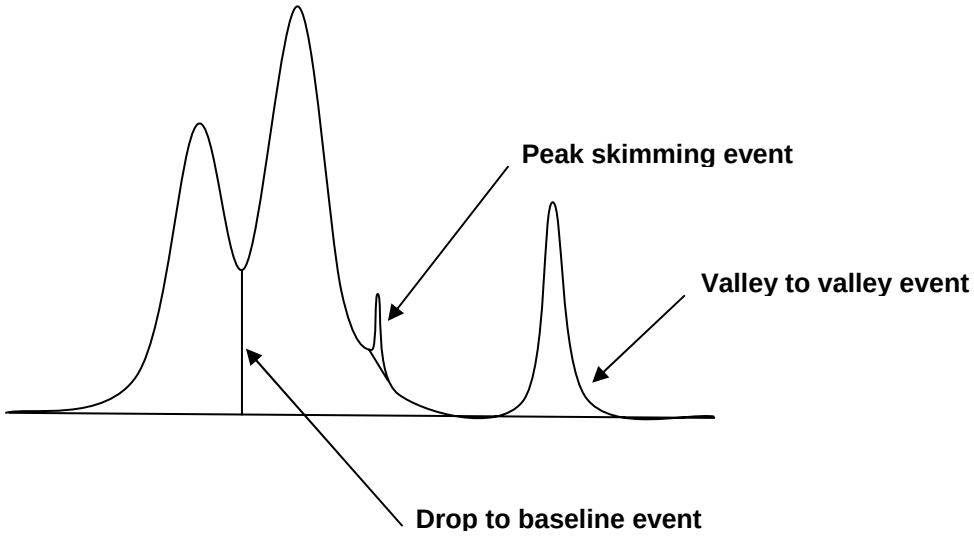
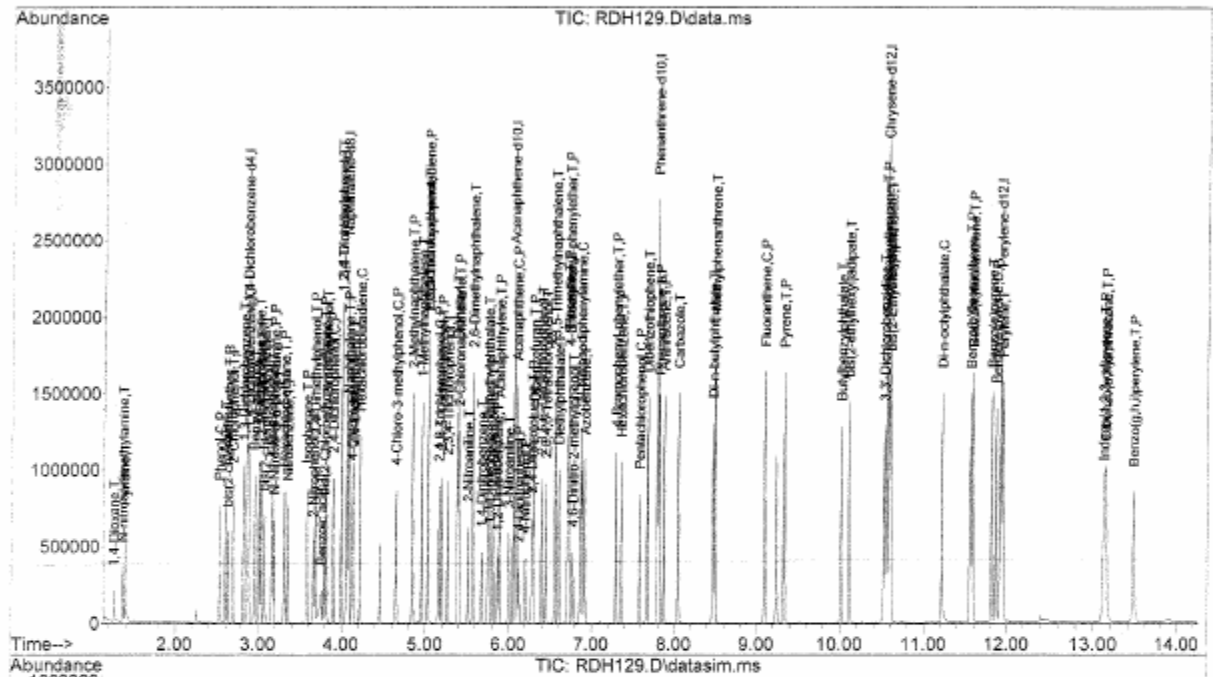


Figure 2: TYPICAL CHROMATOGRAM

```
                    Quantitation Report    (QT Reviewed)
Data File : D:\DATA\14D10\RDH129.D                      Vial: 16
Acq On    : 10 Apr 2014  20:01                      Operator: KV
Sample    : ISVE7D101                               Inst    : E7
Misc      : ICV                                     Multiplr: 1.00
Integrator: RTE
Quant Time: Apr 11 11:46:25 2014
Quant Results File: SVE7D10.RES
Quant Method : C:\msdchem\1\METHODS\SVE7D10.M
Quant Title  : SEMIVOLATILES
QLast Update : Thu Apr 10 18:33:28 2014
Response via : Initial Calibration
DataAcq Meth:SVE7D10S.M
```



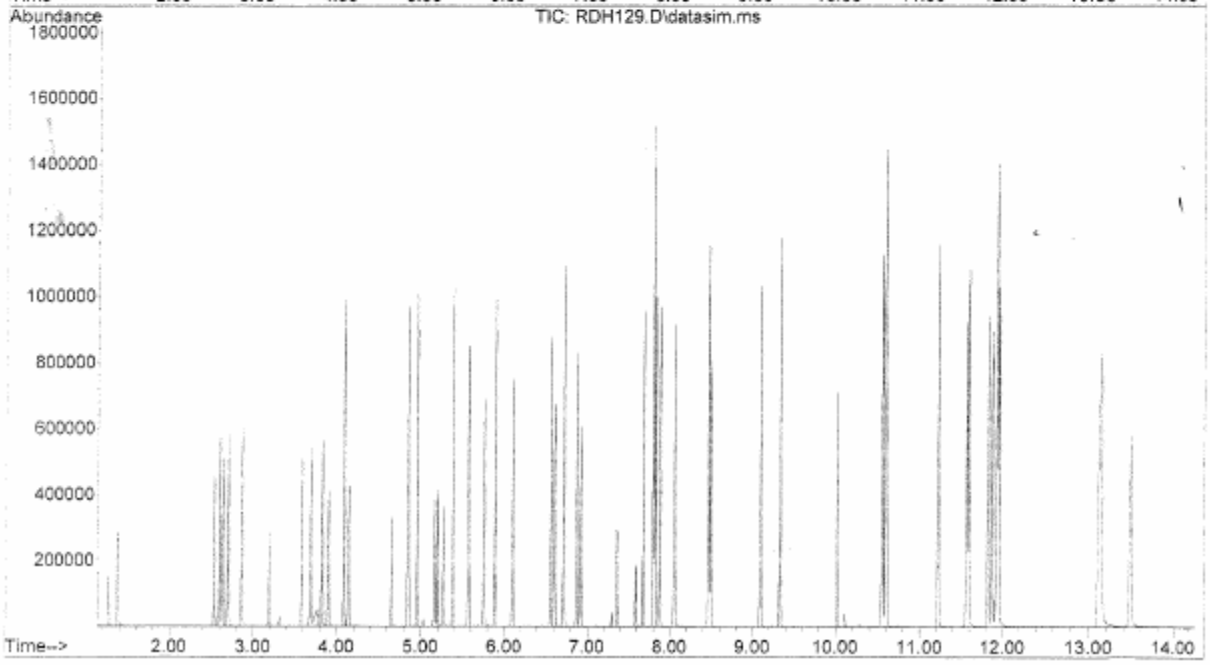


Figure 2A: TYPICAL CHROMATOGRAM FOR BENZIDINE

```
                    Quantitation Report     (QT Reviewed)
Data File : D:\DATA\14D10\RDH137.D                          Vial: 24
Acq On    : 10 Apr 2014  22:36                          Operator: KV
Sample    : ISVE7D10A1                                  Inst    : E7
Misc      : ICV                                         Multiplr: 1.00
Integrator: RTE
Quant Time: Apr 11 11:57:29 2014
Quant Results File: SVE7D10A.RES
Quant Method : C:\msdchem\1\METHODS\SVE7D10A.M
Quant Title  : Semivolatiles - Benzidine
QLast Update : Fri Apr 11 11:56:02 2014
Response via : Initial Calibration
DataAcq Meth:SVE7D10S.M
```

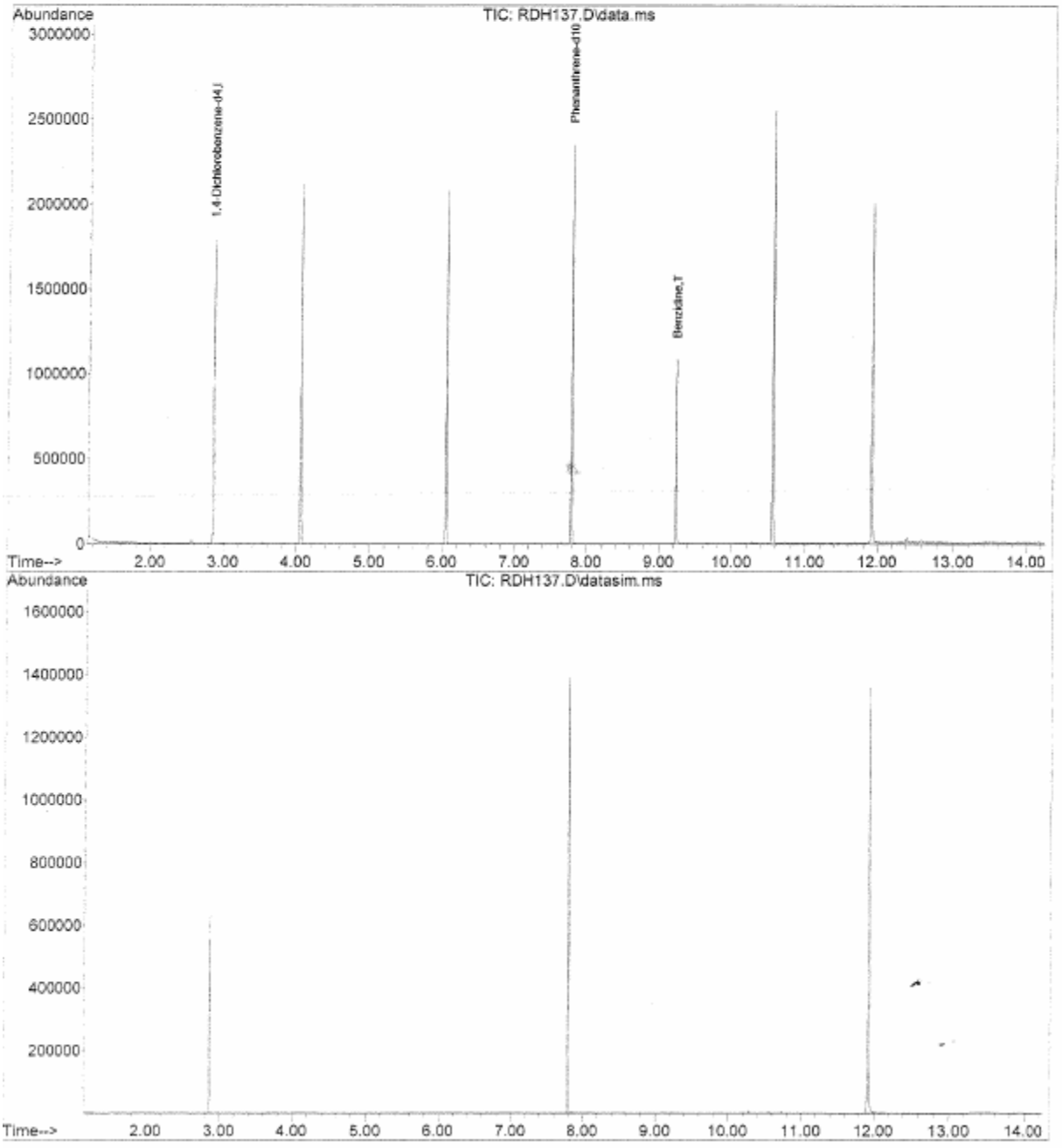


Figure 2B: TYPICAL CHROMATOGRAM FOR APPENDIX IX

Quantitation Report (QT Reviewed)

Data File : D:\DATA\14D10\RDH145.D Vial: 32
Acq On : 11 Apr 2014 1:11 Operator: KV
Sample : ISVE7D10B1 Inst : E7
Misc : 25PPM Multiplr: 1.00
Integrator: RTE
Quant Time: Apr 11 12:56:11 2014
Quant Results File: SVE7D10B.RES
Quant Method : C:\msdchem\1\METHODS\SVE7D10B.M
Quant Title : SEMIVOLATILES APP9
QLast Update : Fri Apr 11 12:49:28 2014
Response via : Initial Calibration
DataAcq Meth:SVE7D10S.M

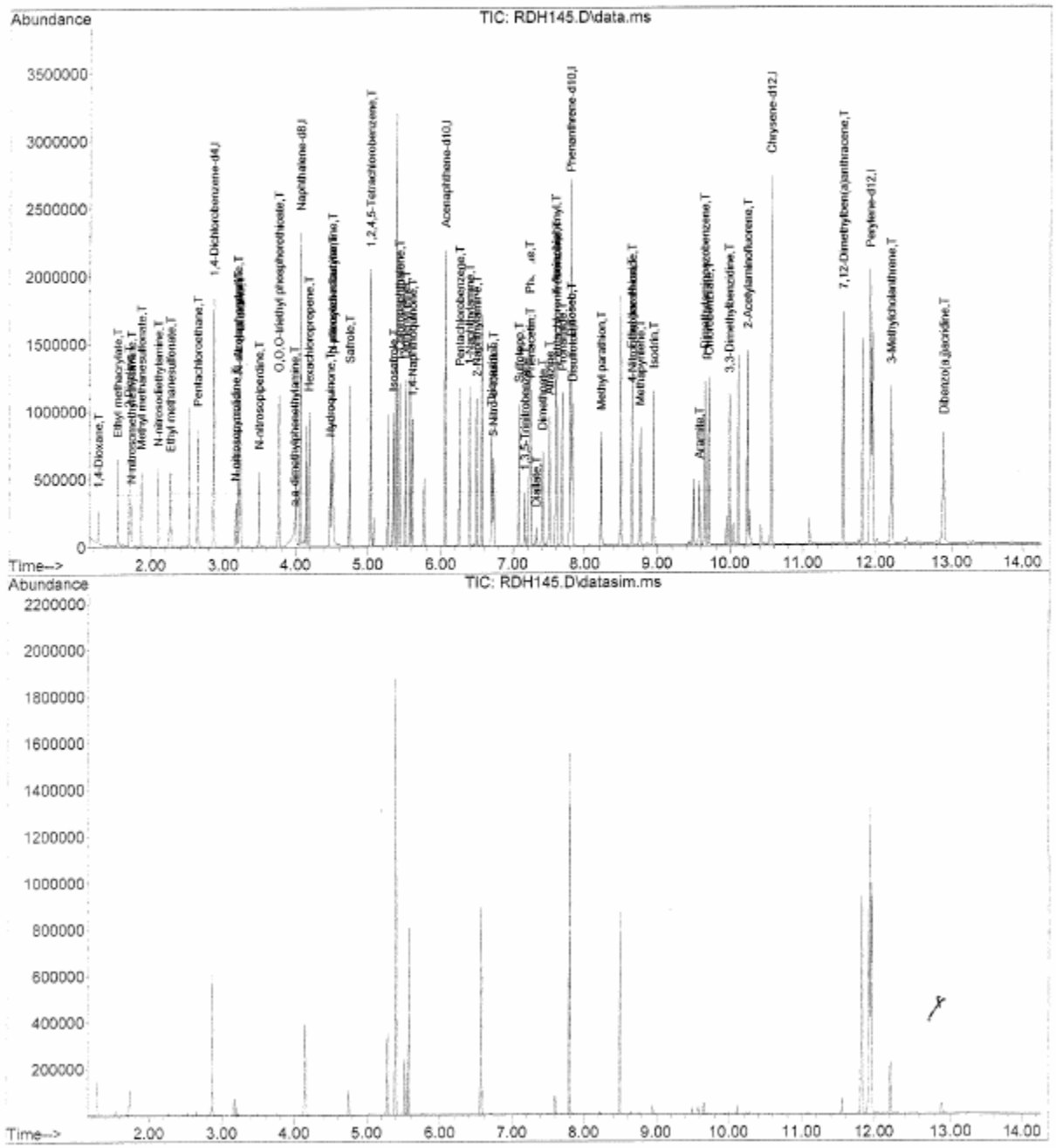


Figure 2B (cont.): TYPICAL CHROMATOGRAM FOR APPENDIX IX

```
                    Quantitation Report    (QT Reviewed)
Data File : D:\DATA\14D11\RDH155.D                        Vial: 10
Acq On    : 11 Apr 2014  17:10                          Operator: DJ
Sample    : ISVE7D11C1                                  Inst    : E7
Misc      : 50PPM                                       Multiplr: 1.00
Integrator: RTE
Quant Time: Apr 11 17:33:29 2014
Quant Results File: SVE7D11C.RES
Quant Method : C:\msdchem\1\METHODS\SVE7D11C.M
Quant Title  : SEMIVOLATILES APP9
QLast Update : Fri Apr 11 17:30:55 2014
Response via : Initial Calibration
DataAcq Meth:SVE7D10S.M
```

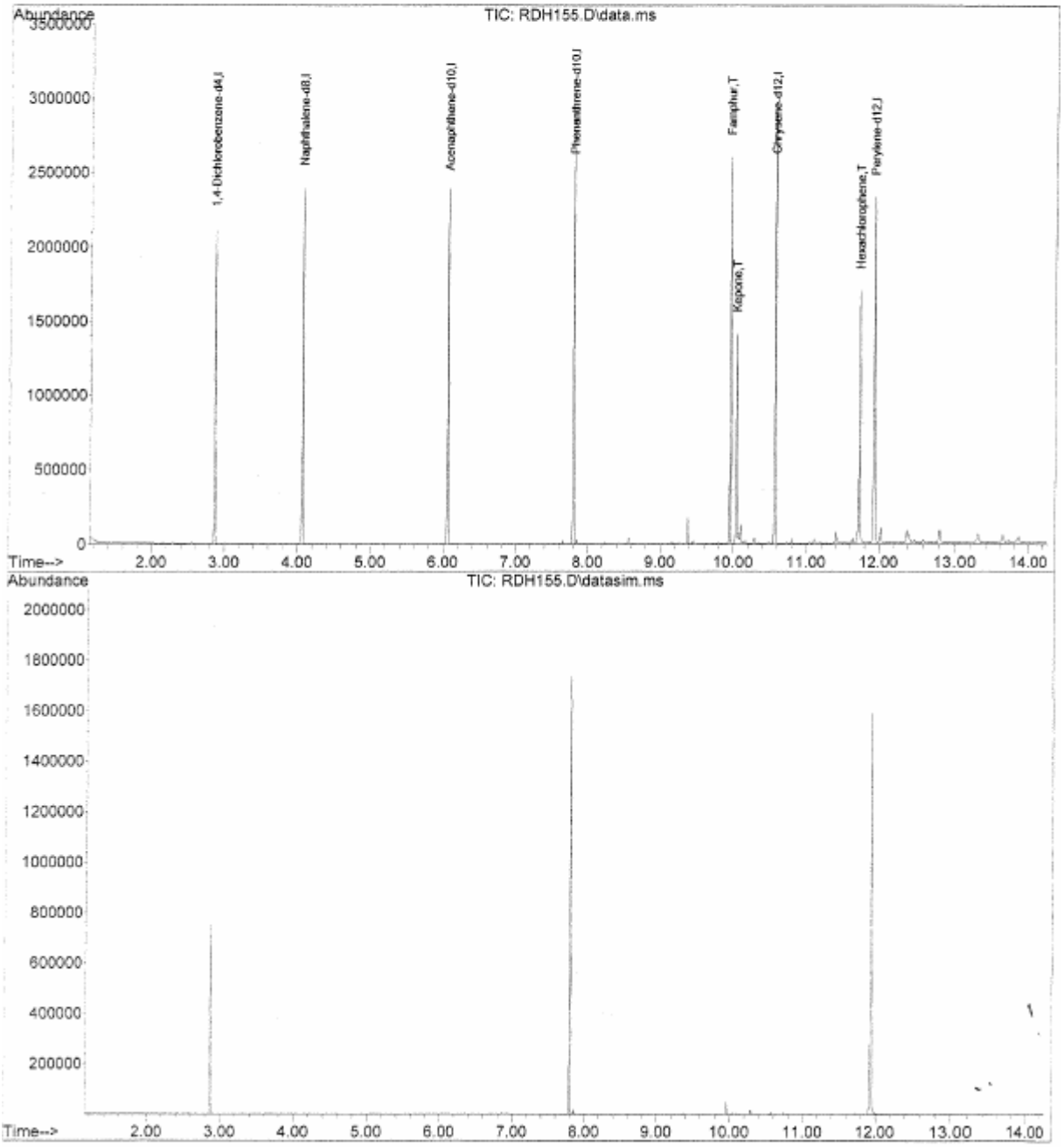


Figure 3: TYPICAL ICAL SUMMARY

INITIAL_CALIBRATION - RELATIVE_RESPONSE_FACTOR

Instrument ID :E4
Beginning DateTime :02/18/14 11:45
Spike Units :PPM
IC File :RBJ065

Column Spec :Rxi-5SilMS ID :0.18MM
Ending DateTime :02/18/14 14:01
HPChem Method :SVE4B18

IDX	Parameters	4 14:01 RBJ068	10 13:41 RBJ067	20 13:22 RBJ066	25 13:03 RBJ065	40 12:43 RBJ064	50 12:24 RBJ063	80 12:05 RBJ062	100 11:45 RBJ061	Av_RRF	%_RSD	Av_Rt_M
1	1,4-Dichlorobenzene-d4	1	1	1	1	1	1	1	1	1	0	3.0245
2	1,4-Dioxane	0.509	0.390	0.448	0.428	0.413	0.414	------	------	0.434	9.62	1.3713
3	N-nitrosodimethylamine	0.599	0.596	0.620	0.603	0.589	0.581	------	------	0.598	2.24	1.4806
4	Pyridine	1.163	1.093	1.184	1.121	1.110	1.102	1.158	1.125	1.132	2.90	1.5077
5	2-Fluorophenol	1.029	0.973	1.053	1.029	1.008	1.004	------	------	1.016	2.68	2.0861
6	Phenol-d5	1.212	1.238	1.333	1.311	1.294	1.275	------	------	1.277	3.55	2.6749
7	Phenol	1.312	1.303	1.394	1.360	1.341	1.342	------	------	1.342	2.46	2.6871
8	Aniline	1.681	1.642	1.768	1.712	1.718	1.699	------	------	1.704	2.47	2.7514
9	bis(2-chloroethyl)ether	0.986	0.951	1.015	0.998	0.951	0.972	------	------	0.979	2.64	2.7749
10	2-Chlorophenol	1.166	1.154	1.226	1.168	1.179	1.173	------	------	1.178	2.14	2.8513
11	1,3-Dichlorobenzene	1.413	1.394	1.460	1.416	1.364	1.379	------	------	1.404	2.40	2.9813
12	1,2-Dichlorobenzene-d4	0.890	0.888	0.916	0.897	0.883	0.875	------	------	0.892	1.58	3.1621
13	1,4-Dichlorobenzene	1.439	1.423	1.507	1.441	1.423	1.413	------	------	1.441	2.37	3.0389
14	Benzyl alcohol	0.703	0.707	0.774	0.763	0.753	0.749	------	------	0.742	3.99	3.1157
15	1,2-Dichlorobenzene	1.354	1.308	1.406	1.379	1.352	1.356	------	------	1.359	2.38	3.1749
16	2-Methylphenol	0.876	0.905	0.976	0.953	0.948	0.941	------	------	0.933	3.90	3.1957
17	bis(2-chloroisopropyl)ether	1.904	1.877	1.989	1.909	1.875	1.822	------	------	1.896	2.89	3.2341
18	4-Methylphenol	1.172	1.167	1.223	1.226	1.209	1.183	------	------	1.197	2.17	3.3364
19	N-Nitroso-di-n-propylamine	0.768	0.766	0.833	0.817	0.798	0.769	------	------	0.792	3.60	3.3563
20	Hexachloroethane	0.529	0.548	0.591	0.565	0.564	0.555	------	------	0.559	3.69	3.4872
21	Naphthalene-d8	1	1	1	1	1	1	1	1	1	0	4.2527
22	Nitrobenzene-d5	0.421	0.423	0.454	0.397	0.388	0.387	------	------	0.412	6.35	3.5128
23	Nitrobenzene	0.374	0.378	0.411	0.396	0.396	0.396	------	------	0.392	3.46	3.5303
24	Isophorone	0.608	0.615	0.656	0.707	0.707	0.717	------	------	0.668	7.34	3.7598
25	2-Nitrophenol	0.168	0.169	0.186	0.176	0.179	0.180	------	------	0.176	3.80	3.8445
26	2,4-Dimethylphenol	0.368	0.368	0.395	0.376	0.377	0.379	------	------	0.377	2.66	3.8637
27	Benzoic acid	0.121	0.195	0.238	0.234	0.240	0.274	0.253	0.253	0.226	21.27	3.9613
28	bis(2-Chloroethoxy)methane	0.350	0.359	0.378	0.357	0.367	0.368	------	------	0.363	2.77	3.9643
29	3,5-Dimethylphenol	0.360	0.423	0.448	0.427	0.437	0.437	------	------	0.422	7.45	4.0026
30	2,4-Dichlorophenol	0.308	0.309	0.338	0.329	0.324	0.327	------	------	0.322	3.68	4.0860
31	3,4-Dimethylphenol	0.438	0.444	0.484	0.465	0.471	0.480	------	------	0.464	4.03	4.1817
32	1,2,4-Trichlorobenzene	0.401	0.387	0.416	0.403	0.405	0.408	------	------	0.403	2.32	4.1873
33	Naphthalene	0.948	0.958	1.031	0.986	0.985	1.002	------	------	0.985	3.04	4.2742
34	4-Chloroaniline	0.388	0.396	0.439	0.413	0.415	0.419	------	------	0.412	4.36	4.3169
35	2,6-Dichlorophenol	0.306	0.299	0.328	0.316	0.318	0.322	------	------	0.315	3.37	4.3305
36	Hexachlorobutadiene	0.283	0.286	0.297	0.283	0.290	0.291	------	------	0.288	1.93	4.4118
37	Hydroquinone	------	------	------	------	------	------	------	------	0.000	0.00	0.0000
38	4-Chloro-3-methylphenol	0.261	0.283	0.307	0.301	0.299	0.299	------	------	0.292	5.80	4.8467
39	2-Methylnaphthalene	0.661	0.659	0.707	0.678	0.684	0.695	------	------	0.680	2.81	5.0423
40	1-Methylnaphthalene	0.614	0.607	0.648	0.632	0.630	0.636	------	------	0.628	2.38	5.1573
41	Acenaphthene-d10	1	1	1	1	1	1	1	1	1	0	6.2772
42	Hexachlorocyclopentadiene	0.518	0.512	0.573	0.529	0.553	0.569	------	------	0.542	4.83	5.2364
43	2,3,5-Trichlorophenol	0.312	0.332	0.371	0.392	0.370	0.421	------	------	0.366	10.81	5.2356
44	2,4,6-Trichlorophenol	0.412	0.424	0.463	0.434	0.446	0.449	------	------	0.438	4.19	5.3654
45	2,4,5-Trichlorophenol	0.418	0.430	0.471	0.452	0.469	0.468	------	------	0.451	4.99	5.4033
46	2-Fluorobiphenyl	1.449	1.468	1.566	1.696	1.526	1.795	------	------	1.583	8.58	5.4693
47	2,3,4-Trichlorophenol	0.422	0.420	0.447	0.429	0.460	0.447	------	------	0.437	3.69	5.4789
48	Biphenyl	1.390	1.403	1.543	1.433	1.464	1.497	------	------	1.455	4.02	5.5870
49	2-Chloronaphthalene	1.131	1.156	1.243	1.170	1.173	1.202	------	------	1.179	3.29	5.6102
50	2-Nitroaniline	0.310	0.332	0.366	0.341	0.345	0.352	------	------	0.341	5.56	5.7229
51	2,6-Dimethylnaphthalene	0.993	1.054	1.109	1.045	1.063	1.095	------	------	1.060	3.88	5.7741
52	1,4-Dinitrobenzene	0.132	0.151	0.173	0.165	0.166	0.167	------	------	0.159	9.39	5.8772
53	Dimethylphthalate	1.511	1.509	1.646	1.511	1.483	1.511	------	------	1.528	3.82	5.9517
54	1,3-Dinitrobenzene	0.168	0.180	0.197	0.186	0.188	0.194	------	------	0.185	5.64	5.9726
55	2,6-Dinitrotoluene	0.249	0.265	0.284	0.271	0.274	0.272	------	------	0.269	4.34	6.0144

Figure 3 (cont.): TYPICAL ICAL SUMMARY

56	1,2-Dinitrobenzene	0.102	0.112	0.120	0.112	0.112	0.116	------	------	0.113	5.35	6.0771
57	Acenaphthylene	1.692	1.729	1.881	1.770	1.788	1.812	------	------	1.779	3.70	6.1053
58	3-Nitroaniline	0.245	0.265	0.281	0.267	0.268	0.274	------	------	0.267	4.63	6.2152
59	Acenaphthene	1.156	1.143	1.234	1.155	1.184	1.195	------	------	1.178	2.85	6.3167
60	2,4-Dinitrophenol	0.130	0.156	0.186	0.175	0.189	0.192	------	------	0.172	14.10	6.3366
61	4-Nitrophenol	0.203	0.228	0.253	0.230	0.243	0.248	------	------	0.234	7.77	6.4165
62	2,4-Dinitrotoluene	0.327	0.363	0.393	0.364	0.363	0.370	------	------	0.363	5.78	6.5004
63	Dibenzofuran	1.588	1.632	1.740	1.637	1.672	1.678	------	------	1.658	3.11	6.5228
64	2,3,5,6-Tetrachlorophenol	0.287	0.307	0.329	0.306	0.323	0.326	------	------	0.313	5.12	6.6194
65	2,3,4,6-Tetrachlorophenol	0.345	0.374	0.405	0.370	0.422	0.441	------	------	0.393	9.19	6.6722
66	2,3,5-Trimethylnaphthalene	0.966	0.968	1.046	0.988	1.007	1.034	------	------	1.002	3.35	6.7808
67	Diethylphthalate	1.323	1.323	1.422	1.337	1.356	1.380	------	------	1.357	2.84	6.8074
68	Fluorene	1.302	1.310	1.422	1.355	1.396	1.439	------	------	1.370	4.20	6.9356
69	4-Chlorophenyl-phenylether	0.714	0.720	0.778	0.732	0.746	0.772	------	------	0.744	3.61	6.9379
70	4-Nitroaniline	0.244	0.262	0.290	0.262	0.273	0.276	------	------	0.268	5.87	6.9535
71	Phenanthrene-d10	1	1	1	1	1	1	1	1	1	0	8.0159
72	4,6-Dinitro-2-methylphenol	0.097	0.123	0.140	0.131	0.135	0.132	------	------	0.126	12.28	6.9926
73	N-Nitrosodiphenylamine	0.608	0.578	0.546	0.523	0.524	0.568	------	------	0.558	5.95	7.0767
74	Azobenzene	0.598	0.577	0.628	0.603	0.595	0.593	------	------	0.599	2.79	7.1258
75	2,4,6-Tribromophenol	0.092	0.093	0.105	0.097	0.098	0.099	------	------	0.097	4.74	7.2191
76	4-Bromophenyl-phenylether	0.207	0.203	0.215	0.204	0.206	0.203	------	------	0.207	2.23	7.5094
77	Hexachlorobenzene	0.228	0.213	0.228	0.222	0.218	0.214	------	------	0.221	3.11	7.5889
78	Pentachlorophenol	0.124	0.140	0.151	0.143	0.145	0.146	------	------	0.142	6.58	7.8061
79	Dibenzothiophene	0.884	0.882	0.945	0.987	0.887	1.005	------	------	0.932	5.95	7.8997
80	Phenanthrene	0.976	0.966	1.021	0.981	0.970	0.972	------	------	0.981	2.07	8.0419
81	Dinoseb	------	------	------	------	------	------	------	------	0.000	0.00	0.0000
82	Anthracene	0.981	0.979	1.040	0.993	0.995	0.993	------	------	0.997	2.21	8.0975
83	Carbazole	0.836	0.846	0.895	0.833	0.839	0.841	------	------	0.848	2.75	8.2730
84	Di-n-butylphthalate	1.012	1.030	1.122	1.050	1.059	1.068	------	------	1.057	3.57	8.6639
85	1-Methylphenanthrene	0.714	0.715	0.777	0.727	0.726	0.731	------	------	0.732	3.17	8.7171
86	Fluoranthene	1.111	1.163	1.244	1.173	1.182	1.202	------	------	1.179	3.72	9.3297
87	Chrysene-d12	1	1	1	1	1	1	1	1	1	0	10.8160
88	Benzidine	------	------	------	------	------	------	------	------	0.000	0.00	0.0000
89	Pyrene	1.059	1.039	1.100	1.051	1.041	1.028	------	------	1.053	2.40	9.5665
90	Terphenyl-d14	0.719	0.701	0.750	0.703	0.705	0.692	------	------	0.712	2.91	9.7277
91	Butylbenzylphthalate	0.361	0.377	0.422	0.392	0.392	0.391	------	------	0.389	5.21	10.2291
92	bis(2-ethylhexyl)adipate	0.282	0.290	0.317	0.299	0.301	0.292	------	------	0.297	4.08	10.3082
93	3,3'-Dichlorobenzidine	0.323	0.348	0.362	0.346	0.347	0.347	------	------	0.346	3.58	10.7722
94	Benzo(a)anthracene	1.035	1.013	1.072	1.012	1.024	1.021	------	------	1.029	2.18	10.8022
95	Chrysene	0.931	0.928	0.965	0.918	0.918	0.908	------	------	0.928	2.14	10.8419
96	bis(2-Ethylhexyl)phthalate	0.602	0.616	0.669	0.639	0.632	0.630	------	------	0.631	3.57	10.8127
97	Perylene-d12	1	1	1	1	1	1	1	1	1	0	12.2554
98	Di-n-octylphthalate	0.969	1.019	1.179	1.084	1.117	1.135	------	------	1.084	7.19	11.4319
99	Benzo(b)fluoranthene	0.990	1.002	1.130	1.005	1.068	1.105	------	------	1.050	5.64	11.8473
100	Benzo(k)fluoranthene	0.966	0.989	1.067	1.056	1.010	1.014	------	------	1.017	3.81	11.8754
101	Benzo(e)pyrene	0.933	0.917	1.033	0.953	0.965	0.983	------	------	0.964	4.26	12.1343
102	Benzo(a)pyrene	0.923	0.946	1.040	0.973	0.990	1.005	------	------	0.980	4.25	12.1910
103	Perylene	0.995	0.986	1.050	0.980	0.984	1.001	------	------	0.999	2.60	12.2849
104	Indeno(1,2,3-cd)pyrene	1.069	1.070	1.202	1.112	1.146	1.172	------	------	1.129	4.83	13.5858
105	Dibenzo(a,h)anthracene	0.858	0.897	0.995	0.925	0.956	0.981	------	------	0.935	5.58	13.5980
106	Benzo(g,h,i)perylene	0.898	0.908	0.989	0.941	0.930	0.950	------	------	0.936	3.48	13.9846

Ave_%RSD : 4.5 Max_%RSD : 21.3

Use Least Square Linear Regression with weighting factor of inverse concentration
Resp_Ratio = x0 + x1 * Amt_Ratio

IDX	Parameter	x0	x1	CCF
27	Benzoic acid	-0.01462	0.26295	0.9988

Figure 3A: TYPICAL ICAL SUMMARY OF BENZIDINE

INITIAL_CALIBRATION - RELATIVE_RESPONSE_FACTOR

Instrument ID :E4
Beginning DateTime :02/18/14 09:10
Spike Units :PPM
IC File :RBJ057

Column Spec :Rxi-5SilMS ID :0.18MM
Ending DateTime :02/18/14 11:07
HPChem Method :SVE4B18A

IDX	Parameters	4 11:07 RBJ059	10 10:47 RBJ058	25 10:28 RBJ057	40 10:09 RBJ056	50 09:49 RBJ055	80 09:30 RBJ054	100 09:10 RBJ053	AV_RRF	%_RSD	AV_Rt_M
1	1,4-Dichlorobenzene-d4	1	1	1	1	1	1	1	1	0	3.0246
2	Benzaldehyde	------	------	------	------	------	------	------	0.000	0.00	0.0000
3	Caprolactam	------	------	------	------	------	------	------	0.000	0.00	0.0000
4	Phenanthrene-d10	1	1	1	1	1	1	1	1	0	8.0143
5	Benzidine	0.585	0.692	0.774	0.775	0.778	0.785	0.820	0.744	10.77	9.4674

Ave_%RSD : 10.8 Max_%RSD : 10.8

Figure 3B: TYPICAL ICAL SUMMARY OF APPENDIX IX

INITIAL_CALIBRATION - RELATIVE_RESPONSE_FACTOR

Instrument ID :E4
Beginning DateTime :02/18/14 16:27
Spike Units :PPM
IC File :RBJ078

Column Spec :Rxi-5SilMS ID :0.18MM
Ending DateTime :02/18/14 18:43
HPChem Method :SVE4B18B

IDX	Parameters	4 18:43 RBJ081	10 18:24 RBJ080	20 18:05 RBJ079	25 17:26 RBJ078	30 17:07 RBJ077	40 16:47 RBJ076	50 16:27 RBJ075	Av_RRF	%_RSD	Av_Rt_M
1	1,4-Dichlorobenzene-d4	1	1	1	1	1	1	1	1	0	3.0246
2	1,4-Dioxane	0.465	0.420	0.478	0.471	0.479	0.469	0.476	0.465	4.39	1.3713
3	Ethyl methacrylate	0.887	0.901	0.938	0.908	0.915	0.907	0.926	0.912	1.82	1.6489
4	2-Picoline	1.242	1.210	1.266	1.222	1.242	1.242	1.239	1.238	1.43	1.8165
5	N-nitrosomethylethylamine	0.480	0.475	0.469	0.460	0.473	0.459	0.458	0.468	1.92	1.8507
6	Methyl methanesulfonate	0.794	0.912	0.941	0.908	0.926	0.894	0.899	0.896	5.33	1.9942
7	2-Fluorophenol	1.113	1.068	1.111	1.072	1.088	1.092	1.094	1.091	1.57	2.0894
8	N-nitrosodiethylamine	0.433	0.459	0.484	0.473	0.484	0.479	0.486	0.471	4.09	2.2339
9	Ethyl methanesulfonate	0.937	0.889	0.937	0.904	0.908	0.921	0.913	0.916	1.92	2.4017
10	Phenol-d5	1.416	1.333	1.398	1.399	1.414	1.404	1.413	1.397	2.08	2.6748
11	Benzaldehyde	------	------	------	------	------	------	------	0.000	0.00	0.0000
12	Pentachloroethane	0.595	0.635	0.648	0.616	0.633	0.623	0.633	0.626	2.73	2.7992
13	1,2-Dichlorobenzene-d4	0.998	0.990	1.035	0.983	1.005	1.019	1.030	1.009	1.97	3.1638
14	N-nitrosopyrrolidine	0.392	0.420	0.480	0.456	0.469	0.469	0.454	0.449	6.99	3.3351
15	Naphthalene-d8	1	1	1	1	1	1	1	1	0	4.2541
16	Acetophenone	0.446	0.435	0.448	0.441	0.448	0.444	0.451	0.445	1.22	3.3645
17	N-nitrosomorpholine	0.237	0.236	0.248	0.246	0.248	0.247	0.247	0.244	2.18	3.3652
18	o-toluidine	0.537	0.608	0.547	0.616	0.552	0.633	0.637	0.590	7.34	3.3994
19	Nitrobenzene-d5	0.462	0.453	0.468	0.467	0.480	0.477	0.472	0.468	1.98	3.5123
20	N-nitrosopiperdine	0.158	0.147	0.156	0.152	0.157	0.157	0.155	0.155	2.38	3.6706
21	O,O,O-triethyl phosphorothioate	0.153	0.151	0.157	0.156	0.157	0.161	0.164	0.157	2.77	3.9336
22	a,a-dimethylphenethylamine	1.090	1.043	1.050	1.030	1.006	0.982	0.914	1.016	5.58	4.1700
23	3,4-Dimethylphenol	------	------	------	------	------	------	------	0.000	0.00	0.0000
24	2,6-Dichlorophenol	------	------	------	------	------	------	------	0.000	0.00	0.0000
25	Hexachloropropene	0.355	0.356	0.377	0.374	0.381	0.380	0.383	0.372	3.17	4.3755
26	Hydroquinone	------	------	------	------	------	------	------	0.000	0.00	0.0000
27	N-nitrosodi-n-butylamine	0.237	0.228	0.303	0.292	0.285	0.289	0.285	0.274	10.65	4.6892
28	p-phenylenediamine	0.279	0.338	0.300	0.290	0.290	0.267	0.256	0.289	9.17	4.6988
29	epsilon-Caprolactam	------	------	------	------	------	------	------	0.000	0.00	0.0000
30	Safrole	0.298	0.292	0.304	0.301	0.302	0.306	0.356	0.309	6.95	4.9365
31	1-Methylnaphthalene	------	------	------	------	------	------	------	0.000	0.00	0.0000
32	1,2,4,5-Tetrachlorobenzene	0.476	0.459	0.485	0.482	0.489	0.492	0.491	0.482	2.41	5.2417
33	Acenaphthene-d10	1	1	1	1	1	1	1	1	0	6.2771
34	2-Fluorobiphenyl	1.709	1.687	1.768	1.689	1.994	1.998	2.024	1.838	8.63	5.4710
35	Isosafrole	0.530	0.491	0.517	0.498	0.509	0.518	0.519	0.512	2.61	5.5453
36	Biphenyl	------	------	------	------	------	------	------	0.000	0.00	0.0000
37	1-Chloronaphthalene	1.168	1.140	1.184	1.159	1.184	1.192	1.203	1.176	1.81	5.6397
38	Diphenyl ether	0.832	0.954	0.866	0.847	0.843	0.852	0.865	0.866	4.74	5.7124
39	1,4-Naphthoquinone	0.402	0.429	0.403	0.373	0.347	0.322	0.316	0.370	11.79	5.8083
40	1,3-Dinitrobenzene	------	------	------	------	------	------	------	0.000	0.00	0.0000
41	Pentachlorobenzene	0.647	0.630	0.635	0.638	0.652	0.654	0.657	0.645	1.61	6.4818
42	1-Naphthylamine	1.047	1.127	1.104	1.066	1.099	1.104	1.110	1.094	2.52	6.6099
43	2,3,4,6-Tetrachlorophenol	------	------	------	------	------	------	------	0.000	0.00	0.0000
44	2-Naphthylamine	1.248	1.277	1.200	1.153	1.174	1.187	1.198	1.205	3.56	6.7064
45	Thionazin	0.177	0.185	0.183	0.181	0.184	0.184	0.183	0.183	1.38	6.8995
46	5-Nitro-o-toluidine	0.279	0.339	0.320	0.317	0.326	0.320	0.327	0.318	5.89	6.9438
47	Phenanthrene-d10	1	1	1	1	1	1	1	1	0	8.0190
48	2,4,6-Tribromophenol	0.092	0.095	0.100	0.099	0.104	0.100	0.101	0.099	3.94	7.2173
49	Sulfotepp	0.077	0.076	0.084	0.084	0.084	0.085	0.084	0.082	4.54	7.2756
50	1,3,5-Trinitrobenzene	0.049	0.055	0.060	0.061	0.066	0.061	0.063	0.059	9.19	7.3744

Figure 3B (cont.): TYPICAL ICAL SUMMARY OF APPENDIX IX

51	Phorate	0.325	0.315	0.351	0.345	0.348	0.343	0.348	0.339	4.02	7.4303
52	Phenacetin	0.298	0.303	0.299	0.297	0.318	0.296	0.306	0.302	2.52	7.4439
53	Diallate	0.037	0.027	0.029	0.027	0.027	0.026	0.026	0.028	13.80	7.5241
54	Dimethoate	0.182	0.181	0.174	0.172	0.175	0.164	0.165	0.173	4.10	7.6166
55	Atrazine	0.208	0.194	0.187	0.176	0.164	0.155	0.148	0.176	12.33	7.7029
56	Pentachloronitrobenzene	0.126	0.116	0.123	0.121	0.120	0.119	0.119	0.121	2.70	7.8304
57	4-Aminobiphenyl	0.681	0.658	0.642	0.632	0.639	0.608	0.613	0.639	3.96	7.8061
58	Pronamide	0.319	0.328	0.338	0.333	0.364	0.330	0.333	0.335	4.19	7.8851
59	Dinoseb	0.157	0.179	0.198	0.202	0.206	0.201	0.206	0.193	9.49	8.0217
60	Disulfoton	0.377	0.317	0.305	0.304	0.300	0.292	0.292	0.312	9.55	8.0231
61	Chrysene-d12	1	1	1	1	1	1	1	1	0	10.8113
62	Methyl parathion	0.153	0.160	0.175	0.170	0.166	0.167	0.160	0.164	4.35	8.4320
63	Ethyl parathion	0.105	0.115	0.128	0.126	0.124	0.131	0.125	0.122	7.52	8.8524
64	4-Nitroquinoline-N-oxide	0.044	0.048	0.057	0.057	0.058	0.059	0.057	0.054	10.89	8.8759
65	Methapyrilene	0.318	0.317	0.296	0.277	0.265	0.253	0.239	0.281	11.07	8.9719
66	Isodrin	0.122	0.110	0.126	0.124	0.116	0.128	0.120	0.121	5.04	9.1741
67	Terphenyl-d14	0.754	0.750	0.786	0.795	0.798	0.820	0.818	0.789	3.53	9.7271
68	Aramite	0.049	0.055	0.063	0.061	0.061	0.060	0.059	0.058	8.11	9.7813
69	p-Dimethylaminoazobenzene	0.200	0.220	0.234	0.226	0.231	0.236	0.233	0.226	5.52	9.8725
70	Chlorobenzilate	0.273	0.274	0.294	0.282	0.284	0.297	0.291	0.285	3.32	9.9216
71	Famphur	------	------	------	------	------	------	------	0.000	0.00	0.0000
72	3,3-Dimethylbenzidine	0.241	0.226	0.224	0.215	0.211	0.205	0.201	0.218	6.31	10.2143
73	Kepone	------	------	------	------	------	------	------	0.000	0.00	0.0000
74	Perylene-d12	1	1	1	1	1	1	1	1	0	12.2513
75	2-Acetylaminofluorene	0.386	0.434	0.462	0.472	0.472	0.467	0.468	0.452	7.07	10.4753
76	7,12-Dimethylben(a)anthracene	0.446	0.461	0.491	0.493	0.492	0.486	0.492	0.480	3.93	11.8314
77	3-Methylcholanthrene	0.477	0.500	0.516	0.524	0.522	0.507	0.525	0.510	3.38	12.5691
78	Dibenzo(a,j)acridine	0.765	0.794	0.836	0.844	0.845	0.838	0.855	0.825	3.99	13.3122

Ave_%RSD : 5 Max_%RSD : 13.8

Figure 3C: TYPICAL ICAL SUMMARY OF ADDITIONAL ANALYTES APPENDIX IX

INITIAL_CALIBRATION - RELATIVE_RESPONSE_FACTOR

Instrument ID :E4
Beginning DateTime :02/18/14 19:31
Spike Units :PPM
IC File :RBJ088

Column Spec :Rxi-5SilMS ID :0.18MM
Ending DateTime :02/18/14 21:28
HPChem Method :SVE4B18C

IDX	Parameters	20 21:28 RBJ090	40 21:08 RBJ089	50 20:49 RBJ088	80 20:30 RBJ087	100 20:10 RBJ086	120 19:51 RBJ085	140 19:31 RBJ084	Av_RRF	%_RSD	Av_Rt_M
1	1,4-Dichlorobenzene-d4	1	1	1	1	1	1	1	1	0	3.0246
2	Naphthalene-d8	1	1	1	1	1	1	1	1	0	4.2506
3	Acenaphthene-d10	1	1	1	1	1	1	1	1	0	6.2736
4	Phenanthrene-d10	1	1	1	1	1	1	1	1	0	8.0130
5	Chrysene-d12	1	1	1	1	1	1	1	1	0	10.8083
6	Famphur	0.496	0.484	0.498	0.522	0.523	0.531	0.526	0.511	3.55	10.1741
7	Kepone	0.096	0.094	0.101	0.105	0.104	0.104	0.106	0.101	4.57	10.2749
8	Perylene-d12	1	1	1	1	1	1	1	1	0	12.2447
9	Hexachlorophene	0.042	0.060	0.069	0.084	0.083	0.085	0.088	0.073	23.28	12.0069

Ave_%RSD : 10.5 Max_%RSD : 23.3

Use Least Square Linear Regression with weighting factor of inverse concentration
Resp_Ratio = xo + x1 * Amt_Ratio

IDX	Parameter	x0	x1	CCF
9	Hexachlorophene	-0.02952	0.09531	0.9991

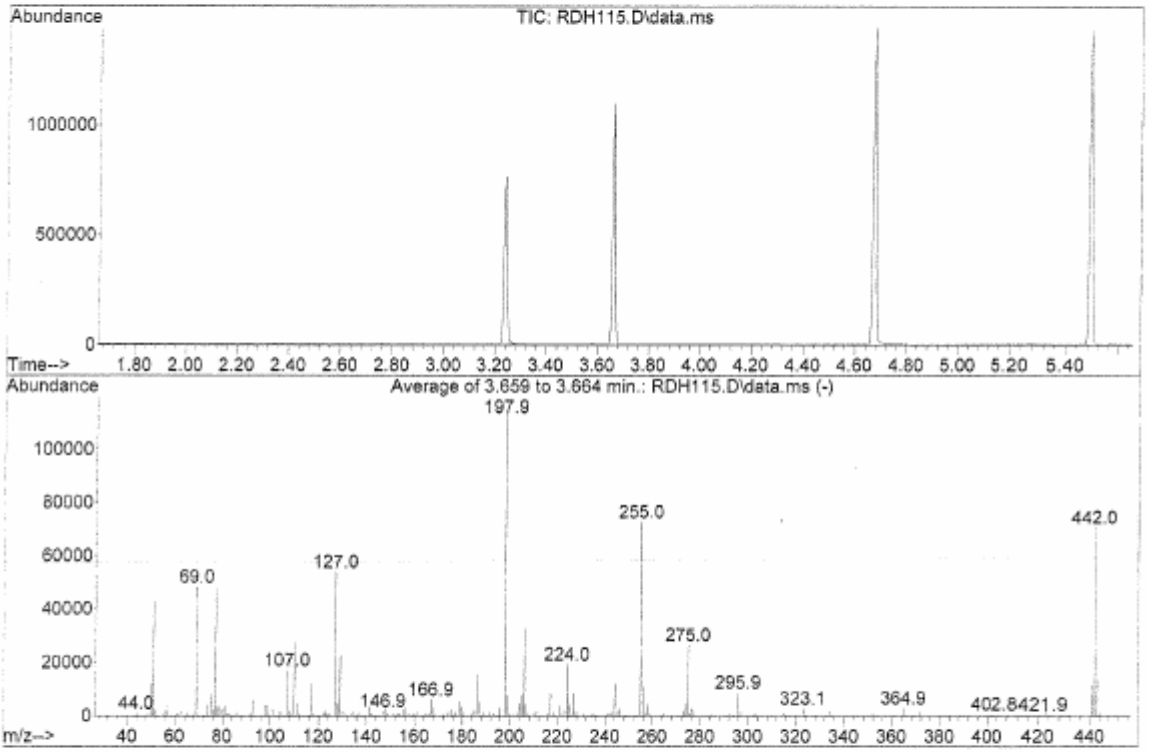
Figure 4: TYPICAL DFTPP TUNE SUMMARY FOR 8270C

DFTPP

Data Path : D:\DATA\14D10\
Data File : RDH115.D
Acq On : 10 Apr 2014 15:20
Operator : KV
Sample : DFTE7D1001
Misc :
ALS Vial : 2 Sample Multiplier: 1

Integration File: rteint.p

Method : C:\msdchem\1\METHODS\DFTPP.M
Title : DFTPP
Last Update : Thu Apr 10 18:47:54 2014



AutoFind: Scans 736, 737, 738; Background Corrected with Scan 728

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	30	60	37.6	42960	PASS
68	69	0.00	2	0.9	440	PASS
69	198	0.00	100	42.3	48224	PASS
70	69	0.00	2	0.0	0	PASS
127	198	40	60	47.1	53784	PASS
197	198	0.00	1	0.0	0	PASS
198	198	100	100	100.0	114120	PASS
199	198	5	9	6.7	7602	PASS
275	198	10	30	23.1	26336	PASS
365	198	1	100	2.5	2845	PASS
441	443	0.01	100	84.1	11129	PASS
442	198	40	100	61.7	70408	PASS
443	442	17	23	18.8	13237	PASS

Figure 5: TYPICAL INSTRUMENT PERFORMANCE CHECK

5B
SEMIVOLATILE ORGANIC INSTRUMENT PERFORMANCE CHECK
DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

Lab Name: EMAX Inc
Lab Code: EMXT
Lab File ID: RDH115
Instrument ID: TOE7

Project: CLEAN PROJECT
SDG No.: 14D050
DFTPP Injection Date: 04/10/14
DFTPP Injection Time: 15:20

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
51	30.0 - 60.0% of mass 198	37.65
68	Less than 2% of mass 69	0.39(0.9)1
69	Relative abundance of mass 198	42.26
70	Less than 2.0% of mass 69	0.00(0.0)1
127	40.0 - 60.0% of mass 198	47.13
197	Less than 1.0% of mass 198	0.00
198	Base Peak, 100% relative abundance	100.00
199	5.0 - 9.0% of mass 198	6.66
275	10.0 - 30.0% of mass 198	23.08
365	Greater than 1.00% of mass 198	2.49
441	Present, but less than mass 443	9.75(84.1)3
442	Greater than 40.0% of mass 198	61.70
443	17.0 - 23.0% of mass 442	11.60(18.8)2

1-Value is % mass 69
2-Value is % mass 442
3-Value is % mass 443

THIS CHECK APPLIES TO THE FOLLOWING SAMPLES, MS, MSD,BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
1	SSTD100	SVE7D101	RDH116	04/10/14	15:51
2	SSTD080	SVE7D102	RDH117	04/10/14	16:10
3	SSTD050	SVE7D103	RDH118	04/10/14	16:29
4	SSTD040	SVE7D104	RDH119	04/10/14	16:48
5	SSTD025	SVE7D105	RDH120	04/10/14	17:08
6	SSTD020	SVE7D106	RDH121	04/10/14	17:27
7	SSTD010	SVE7D107	RDH122	04/10/14	17:46
8	SSTD004	SVE7D108	RDH123	04/10/14	18:06
9	SSTD025	ISVE7D101	RDH129	04/10/14	20:01

page 1 of 1

FORM V SV OLM02.0

Figure 6: TYPICAL CONTINUING CALIBRATION SUMMARY

CONTINUE_CALIBRATION - CALIBRATION VERIFICATION

Instrument ID :E7
IC_Beginning DateTime :04/10/14 15:51
Spike Amount :25 PPM
CC/CV File :RDH129
IC File :RDH120

Column Spec :ZB-SemiVoa ID :0.18MM
IC_Ending DateTime :04/10/14 18:06
HPChem Method :SVE7D10
Date_Time :04/10/14 20:01

M IDX	Parameters	CC_Con	CC%_D	CC_Resp	CCRRF	AvRRF	CC_Rtm	AvRtm	%_RSD	Co_X0	Co_X1	Co_X2	Co_Cor
1	1,4-Dichlorobenzene-d4	40.000	0	236184	1	1	2.866	2.865	0				
2	1,4-Dioxane	24.932	-0.3	69957	0.474	0.475	1.262	1.265	6.36				
3	N-nitrosodimethylamine	22.338	-10.6	87392	0.592	0.663	1.372	1.371	1.92				
4	Pyridine	24.262	-3.0	183749	1.245	1.283	1.396	1.397	2.73				
5	2-Fluorophenol												
6	Phenol-d5												
7	Phenol	22.423	-10.3	197093	1.335	1.489	2.536	2.535	2.55				
8	Aniline	24.673	-1.3	277116	1.877	1.902	2.599	2.598	2.64				
9	bis(2-chloroethyl)ether	23.556	-5.8	148463	1.006	1.067	2.630	2.630	3.66				
10	2-Chlorophenol	22.391	-10.4	169755	1.150	1.284	2.693	2.693	3.29				
11	1,3-Dichlorobenzene	23.135	-7.5	199038	1.348	1.457	2.823	2.822	1.68				
12	1,2-Dichlorobenzene-d4												
13	1,4-Dichlorobenzene	22.931	-8.3	201819	1.367	1.491	2.880	2.879	1.29				
14	Benzyl alcohol	22.739	-9.0	110549	0.749	0.823	2.952	2.956	3.27				
15	1,2-Dichlorobenzene	22.985	-8.1	189990	1.287	1.400	3.010	3.009	2.35				
16	2-Methylphenol	23.651	-5.4	140091	0.949	1.003	3.034	3.032	3.37				
17	bis(2-chloroisopropyl)ether	23.562	-5.8	192513	1.304	1.384	3.077	3.076	3.68				
18	4-Methylphenol	23.276	-6.9	177716	1.204	1.293	3.168	3.168	3.19				
19	N-Nitroso-di-n-propylamine	22.403	-10.4	103672	0.702	0.784	3.192	3.192	3.80				
20	Hexachloroethane	22.794	-8.8	75921	0.514	0.564	3.312	3.313	2.70				
21	Naphthalene-d8	40.000	0	892727	1	1	4.066	4.064	0				
22	Nitrobenzene-d5												
23	Nitrobenzene	22.194	-11.2	183217	0.328	0.370	3.355	3.358	2.02				
24	Isophorone	25.643	2.6	376664	0.675	0.658	3.585	3.587	8.28				
25	2-Nitrophenol	22.051	-11.8	87468	0.157	0.178	3.667	3.664	6.11				
26	2,4-Dimethylphenol	22.620	-9.5	180337	0.323	0.357	3.686	3.687	4.76				
27	Benzoic acid	20.404	-18.4	111108	0.199	0.244	3.767	3.779	10.58				
28	bis(2-Chloroethoxy)methane	23.447	-6.2	190911	0.342	0.365	3.796	3.794	2.74				
29	3,5-Dimethylphenol	21.933	-12.3	180596	0.324	0.369	3.825	3.825	10.56				
30	2,4-Dichlorophenol	22.510	-10.0	168452	0.302	0.335	3.902	3.901	1.95				
31	1,2,4-Trichlorobenzene	22.451	-10.2	185770	0.333	0.371	4.003	4.001	1.93				
32	3,4-Dimethylphenol	23.048	-7.8	224613	0.403	0.437	3.998	4.000	3.88				
33	Naphthalene	22.617	-9.5	514465	0.922	1.019	4.085	4.088	1.20				
34	4-Chloroaniline	23.283	-6.9	216083	0.387	0.416	4.128	4.130	2.49				
35	2,6-Dichlorophenol	23.965	-4.1	151484	0.271	0.283	4.143	4.142	3.59				
36	Hexachlorobutadiene	22.924	-8.3	129760	0.233	0.254	4.220	4.221	2.29				
37	Hydroquinone												
38	4-Chloro-3-methylphenol	21.618	-13.5	134354	0.241	0.278	4.654	4.653	2.25				
39	2-Methylnaphthalene	22.657	-9.4	348859	0.625	0.690	4.846	4.846	1.32				
40	1-Methylnaphthalene	23.384	-6.5	329637	0.591	0.632	4.956	4.958	1.95				
41	Acenaphthene-d10	40.000	0	490143	1	1	6.062	6.061	0				
42	Hexachlorocyclopentadiene	20.470	-18.1	131441	0.429	0.524	5.033	5.032	5.14				
43	2,3,5-Trichlorophenol	28.830	15.3	119762	0.391	0.339	5.033	5.030	1.69				
44	2,4,6-Trichlorophenol	22.427	-10.3	116051	0.379	0.422	5.163	5.160	2.21				
45	2,4,5-Trichlorophenol	24.747	-1.0	130233	0.425	0.429	5.196	5.196	4.44				
46	2-Fluorobiphenyl												
47	2,3,4-Trichlorophenol	24.201	-3.2	109305	0.357	0.369	5.269	5.271	2.79				
48	Biphenyl	24.460	-2.2	445998	1.456	1.488	5.384	5.385	1.35				
49	2-Chloronaphthalene	23.038	-7.8	332776	1.086	1.179	5.404	5.403	1.66				
50	2-Nitroaniline	23.173	-7.3	88791	0.290	0.313	5.514	5.515	4.97				
51	2,6-Dimethylnaphthalene	24.654	-1.4	333443	1.088	1.104	5.571	5.569	1.78				
52	1,4-Dinitrobenzene	24.237	-3.1	50453	0.165	0.170	5.677	5.679	4.29				
53	Dimethylphthalate	23.149	-7.4	452676	1.478	1.596	5.754	5.754	1.32				
54	1,3-Dinitrobenzene	21.607	-13.6	52316	0.171	0.198	5.768	5.767	2.00				
55	2,6-Dinitrotoluene	23.249	-7.0	83399	0.272	0.293	5.812	5.811	1.95				

Figure 6 (cont.): TYPICAL CONTINUING CALIBRATION SUMMARY

56	1,2-Dinitrobenzene	24.096	-3.6	33719	0.110	0.114	5.865	5.866	6.89				
57	Acenaphthylene	23.349	-6.6	541096	1.766	1.891	5.889	5.890	0.80				
58	3-Nitroaniline	23.121	-7.5	85538	0.279	0.302	6.000	6.002	2.88				
59	Acenaphthene	22.946	-8.2	349336	1.140	1.242	6.100	6.100	1.87				
60	2,4-Dinitrophenol	22.266	-10.9	43948	0.143	0.161	6.124	6.125	7.03				
61	4-Nitrophenol	22.782	-8.9	68322	0.223	0.245	6.201	6.199	4.70				
62	2,4-Dinitrotoluene	23.512	-6.0	111877	0.365	0.388	6.287	6.287	2.60				
63	Dibenzofuran	22.654	-9.4	458083	1.495	1.650	6.306	6.306	1.34				
64	2,3,5,6-Tetrachlorophenol	23.149	-7.4	83137	0.271	0.293	6.397	6.398	5.24				
65	2,3,4,6-Tetrachlorophenol	20.679	-17.3	98188	0.321	0.387	6.450	6.449	5.82				
66	2,3,5-Trimethylnaphthalene	25.063	0.3	300611	0.981	0.979	6.564	6.563	2.80				
67	Diethylphthalate	23.039	-7.8	402694	1.315	1.426	6.606	6.608	1.28				
68	Fluorene	22.257	-11.0	366505	1.196	1.344	6.719	6.718	1.79				
69	4-Chlorophenyl-phenylether	22.927	-8.3	191484	0.625	0.682	6.733	6.731	0.94				
70	4-Nitroaniline	24.201	-3.2	89054	0.291	0.300	6.733	6.732	2.83				
71	Phenanthrene-d10	40.000	0	919199	1	1	7.796	7.797	0				
72	4,6-Dinitro-2-methylphenol	24.229	-3.1	64140	0.112	0.115	6.775	6.775	14.46				
73	N-Nitrosodiphenylamine	23.461	-6.2	307643	0.535	0.571	6.869	6.870	2.15				
74	Azobenzene	21.761	-13.0	337764	0.588	0.675	6.917	6.918	2.94				
75	2,4,6-Tribromophenol												
76	4-Bromophenyl-phenylether	24.111	-3.6	114341	0.199	0.206	7.301	7.302	1.74				
77	Hexachlorobenzene	22.913	-8.3	118046	0.205	0.224	7.362	7.361	1.26				
78	Pentachlorophenol	23.935	-4.3	81305	0.142	0.148	7.584	7.582	5.21				
79	Dibenzothiophene	23.518	-5.9	511803	0.891	0.947	7.679	7.679	2.00				
80	Phenanthrene	22.694	-9.2	524956	0.914	1.007	7.825	7.822	1.25				
81	Dinoseb												
82	Anthracene	22.872	-8.5	541793	0.943	1.031	7.876	7.877	2.39				
83	Carbazole	23.627	-5.5	511559	0.890	0.942	8.055	8.055	1.57				
84	Di-n-butylphthalate	23.206	-7.2	652551	1.136	1.224	8.470	8.467	4.52				
85	1-Methylphenanthrene	24.442	-2.2	424846	0.740	0.756	8.494	8.493	2.43				
86	Fluoranthene	21.256	-15.0	599853	1.044	1.228	9.098	9.097	7.92				
87	Chrysene-d12	40.000	0	962830	1	1	10.567	10.565	0				
88	Benzidine												
89	Pyrene	22.417	-10.3	635848	1.057	1.178	9.329	9.330	2.15				
90	Terphenyl-d14												
91	Butylbenzylphthalate	24.144	-3.4	287923	0.478	0.495	10.016	10.013	5.78				
92	bis(2-ethylhexyl)adipate	23.931	-4.3	228198	0.379	0.396	10.108	10.109	5.76				
93	3,3'-Dichlorobenzidine	23.151	-7.4	215515	0.358	0.387	10.528	10.528	1.43				
94	Benzo(a)anthracene	22.565	-9.7	612810	1.018	1.128	10.552	10.553	1.48				
95	Chrysene	22.740	-9.0	556264	0.924	1.016	10.591	10.589	2.32				
96	bis(2-Ethylhexyl)phthalate	23.271	-6.9	431382	0.717	0.770	10.601	10.602	4.61				
97	Perylene-d12	40.000	0	949618	1	1	11.926	11.927	0				
98	Di-n-octylphthalate	23.347	-6.6	735924	1.240	1.328	11.217	11.218	6.06				
99	Benzo(b)fluoranthene	22.146	-11.4	572045	0.964	1.088	11.563	11.561	4.73				
100	Benzo(k)fluoranthene	23.377	-6.5	584658	0.985	1.053	11.587	11.588	4.31				
101	Benzo(e)pyrene	24.842	-0.6	591183	0.996	1.002	11.819	11.818	3.35				
102	Benzo(a)pyrene	22.403	-10.4	538072	0.907	1.012	11.868	11.870	4.47				
103	Perylene	22.655	-9.4	557932	0.940	1.037	11.950	11.952	3.09				
104	Indeno(1,2,3-cd)pyrene	23.046	-7.8	631945	1.065	1.155	13.116	13.120	2.77				
105	Dibenzo(a,h)anthracene	23.005	-8.0	513056	0.864	0.939	13.136	13.140	3.75				
106	Benzo(g,h,i)perylene	22.976	-8.1	532510	0.897	0.976	13.470	13.473	2.54				

Figure 6A: TYPICAL CONTINUING CALIBRATION SUMMARY OF BENZIDINE

CONTINUE_CALIBRATION - CALIBRATION VERIFICATION

Instrument ID :E7
IC_Beginning DateTime :04/10/14 20:20
Spike Amount :25 PPM
CC/CV File :RDH137
IC File :RDH134

Column Spec :ZB-SemiVoa ID :0.18MM
IC_Ending DateTime :04/10/14 22:16
HPChem Method :SVE7D10A
Date_Time :04/10/14 22:36

M IDX	Parameters	CC_Con	CC%_D	CC_Resp	CCRRF	AvRRF	CC_Rtm	AvRtm	%_RSD	Co_X0	Co_X1	Co_X2	Co_Cor
1	1,4-Dichlorobenzene-d4	40.000	0	346380	1	1	2.861	2.862	0				
2	Benzaldehyde												
3	Caprolactam												
4	Phenanthrene-d10	40.000	0	834502	1	1	7.797	7.797	0				
5	Benzidine	23.091	-7.6	372153	0.714	0.773	9.238	9.242	5.16				

Figure 6B: TYPICAL CONTINUING CALIBRATION SUMMARY OF ANALYTES APPENDIX IX

CONTINUE_CALIBRATION - CALIBRATION VERIFICATION

Instrument ID :E7
IC_Beginning DateTime :04/10/14 22:55
Spike Amount :25 PPM
CC/CV File :RDH145
IC File :RDH141

Column Spec :ZB-SemiVoa ID :0.18MM
IC_Ending DateTime :04/11/14 00:51
HPChem Method :SVE7D10B
Date_Time :04/11/14 01:11

M IDX	Parameters	CC_Con	CC%_D	CC_Resp	CCRRF	AvRRF	CC_Rtm	AvRtm	%_RSD	Co_X0	Co_X1	Co_X2	Co_Cor
1	1,4-Dichlorobenzene-d4	40.000	0	230153	1	1	2.866	2.865	0				
2	1,4-Dioxane	25.971	3.9	72862	0.507	0.488	1.266	1.267	11.36				
3	Ethyl methacrylate	25.653	2.6	122878	0.854	0.832	1.539	1.539	3.19				
4	2-Picoline	25.947	3.8	184288	1.281	1.234	1.697	1.697	1.44				
5	N-nitrosomethylethylamine	26.794	7.2	69565	0.484	0.451	1.731	1.732	1.64				
6	Methyl methanesulfonate	26.173	4.7	133985	0.931	0.890	1.870	1.871	3.09				
7	2-Fluorophenol												
8	N-nitrosodiethylamine	26.054	4.2	69732	0.485	0.465	2.100	2.101	3.89				
9	Ethyl methanesulfonate	26.340	5.4	125629	0.873	0.829	2.267	2.266	2.88				
10	Phenol-d5												
11	Benzaldehyde												
12	Pentachloroethane	25.700	2.8	84159	0.585	0.569	2.646	2.646	1.77				
13	1,2-Dichlorobenzene-d4												
14	N-nitrosopyrrolidine	26.733	6.9	66848	0.465	0.435	3.168	3.168	3.44				
15	Naphthalene-d8	40.000	0	852034	1	1	4.066	4.065	0				
16	Acetophenone	26.641	6.6	203560	0.382	0.359	3.197	3.197	1.85				
17	N-nitrosomorpholine	25.817	3.3	90254	0.169	0.164	3.202	3.202	3.62				
18	o-toluidine	25.462	1.8	277841	0.522	0.512	3.230	3.230	8.37				
19	Nitrobenzene-d5												
20	N-nitrosopiperdine	26.895	7.6	83284	0.156	0.145	3.494	3.496	2.60				
21	O,O,O-triethyl phosphorothioate	26.547	6.2	75709	0.142	0.134	3.767	3.766	1.18				
22	a,a-dimethylphenethylamine	23.912	-4.4	266734	0.501	0.524	3.984	3.978	4.66				
23	3,4-Dimethylphenol												
24	2,6-Dichlorophenol												
25	Hexachloropropene	26.085	4.3	163140	0.306	0.294	4.182	4.184	2.25				
26	Hydroquinone	27.037	8.1	132905	0.250	0.231	4.476	4.477	7.00				
27	N-nitrosodi-n-butylamine	26.689	6.8	117036	0.220	0.206	4.505	4.506	7.81				
28	p-phenylenediamine	27.088	8.4	74061	0.139	0.128	4.505	4.506	12.87				
29	epsilon-Caprolactam												
30	Safrole	26.054	4.2	137077	0.257	0.247	4.745	4.747	1.91				
31	1-Methylnaphthalene												
32	1,2,4,5-Tetrachlorobenzene	25.935	3.7	201858	0.379	0.365	5.037	5.036	1.74				
33	Acenaphthene-d10	40.000	0	472216	1	1	6.062	6.058	0				
34	2-Fluorobiphenyl												
35	Isosafrole	25.201	0.8	165442	0.561	0.556	5.346	5.347	2.12				
36	Biphenyl												
37	1-Chloronaphthalene	26.557	6.2	331463	1.123	1.057	5.432	5.431	1.95				
38	Diphenyl ether	26.307	5.2	230815	0.782	0.743	5.514	5.513	1.66				
39	1,4-Naphthoquinone	27.435	9.7	135307	0.458	0.418	5.605	5.604	7.33				
40	1,3-Dinitrobenzene												

Figure 6B (cont.): TYPICAL CONTINUING CALIBRATION SUMMARY OF ANALYTES APPENDIX IX

41	Pentachlorobenzene	26.133	4.5	165684	0.561	0.537	6.258	6.256	1.70				
42	1-Naphthylamine	26.797	7.2	340129	1.152	1.075	6.393	6.392	1.82				
43	2,3,4,6-Tetrachlorophenol												
44	2-Naphthylamine	25.795	3.2	362974	1.230	1.192	6.489	6.488	4.73				
45	Thionazin	26.482	5.9	53904	0.183	0.172	6.700	6.699	4.45				
46	5-Nitro-o-toluidine	26.914	7.7	98851	0.335	0.311	6.723	6.725	2.86				
47	Phenanthrene-d10	40.000	0	897257	1	1	7.796	7.799	0				
48	2,4,6-Tribromophenol												
49	Sulfotepp	26.272	5.1	39561	0.071	0.067	7.083	7.084	5.08				
50	1,3,5-Trinitrobenzene	26.235	4.9	31338	0.056	0.053	7.163	7.166	12.28				
51	Phorate	27.525	10.1	233279	0.416	0.378	7.230	7.230	1.57				
52	Phenacetin	26.331	5.3	201737	0.360	0.342	7.239	7.240	2.59				
53	Diallate	26.990	8.0	17045	0.030	0.028	7.320	7.322	3.49				
54	Dimethoate	26.661	6.6	105997	0.189	0.177	7.409	7.411	5.69				
55	Atrazine	26.216	4.9	104341	0.186	0.177	7.504	7.503	7.44				
56	Pentachloronitrobenzene	26.988	8.0	66443	0.118	0.110	7.603	7.604	3.80				
57	4-Aminobiphenyl	25.698	2.8	383831	0.684	0.666	7.594	7.594	1.11				
58	Pronamide	25.511	2.0	193073	0.344	0.337	7.683	7.681	1.71				
59	Dinoseb	27.411	9.6	90062	0.161	0.146	7.806	7.806	8.34				
60	Disulfoton	26.641	6.6	152036	0.271	0.254	7.825	7.824	4.54				
61	Chrysene-d12	40.000	0	912248	1	1	10.562	10.562	0				
62	Methyl parathion	27.196	8.8	112386	0.197	0.181	8.224	8.224	4.04				
63	Ethyl parathion	27.100	8.4	78996	0.139	0.128	8.649	8.647	6.80				
64	4-Nitroquinoline-N-oxide	27.734	10.9	54674	0.096	0.086	8.653	8.653	6.78				
65	Methapyrilene	27.737	10.9	185847	0.326	0.294	8.765	8.763	6.69				
66	Isodrin	26.753	7.0	73147	0.128	0.120	8.948	8.947	4.26				
67	Terphenyl-d14												
68	Aramite	26.566	6.3	31743	0.056	0.052	9.576	9.576	5.90				
69	p-Dimethylaminoazobenzene	26.486	5.9	149654	0.262	0.248	9.649	9.650	3.61				
70	Chlorobenzilate	27.780	11.1	163928	0.288	0.259	9.703	9.704	5.49				
71	3,3-Dimethylbenzidine	26.573	6.3	328992	0.577	0.543	9.986	9.988	7.37				
72	Perylene-d12	40.000	0	911888	1	1	11.926	11.926	0				
73	2-Acetylaminofluorene	27.124	8.5	273538	0.480	0.442	10.240	10.240	6.11				
74	7,12-Dimethylben(a)anthracene	26.412	5.6	262003	0.460	0.435	11.553	11.553	4.04				
75	3-Methylcholanthrene	25.078	0.3	274943	0.482	0.481	12.217	12.219	3.80				
76	Dibenzo(a,j)acridine	26.759	7.0	450787	0.791	0.739	12.888	12.886	5.22				

Figure 6C: TYPICAL CONTINUING CALIBRATION SUMMARY OF ADDITIONAL ANALYTES APPENDIX IX

CONTINUE_CALIBRATION - CALIBRATION VERIFICATION

Instrument ID :E7
IC_Beginning DateTime :04/11/14 14:56
Spike Amount :50 PPM
CC/CV File :RDH155
IC File :RDH152

Column Spec :ZB-SemiVoa ID :0.18MM
IC_Ending DateTime :04/11/14 16:51
HPChem Method :SVE7D11C
Date_Time :04/11/14 17:10

M IDX	Parameters	CC_Con	CC%_D	CC_Resp	CCRRF	AvRRF	CC_Rtm	AvRtm	%_RSD	Co_X0	Co_X1	Co_X2	Co_Cor
1	1,4-Dichlorobenzene-d4	40.000	0	268288	1	1	2.861	2.862	0				
2	Naphthalene-d8	40.000	0	966151	1	1	4.061	4.063	0				
3	Acenaphthene-d10	40.000	0	521650	1	1	6.062	6.059	0				
4	Phenanthrene-d10	40.000	0	1036706	1	1	7.797	7.796	0				
5	Chrysene-d12	40.000	0	1071961	1	1	10.562	10.567	0				
6	Famphur	51.359	2.7	731295	0.546	0.531	9.952	9.959	2.38				
7	Kepone	48.840	-2.3	136412	0.102	0.104	10.040	10.040	1.74				
8	Perylene-d12	40.000	0	1015463	1	1	11.926	11.933	0				
9	Hexachlorophene	55.518	11.0	158347	0.125	0.111	11.727	11.731	17.61	-0.0344	0.1372		0.9982

Figure 7A: TYPICAL INTERNAL STANDARD AREA AND RT SUMMARY SV-1

8B

SEMIVOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name: EMAX Inc
Lab Code: EMXT
Lab File ID: RDH120
Instrument ID: TOE7

Project:ICAL
SDG No.:CAL\8270\2014
Date Analyzed: 04/10/14
Time Analyzed: 17:08

		IS1(DCB)		IS2(NPT)		IS3(ANT)	
		AREA #	RT #	AREA #	RT #	AREA #	RT #
	12 HOUR STD	245336	2.87	927578	4.07	503408	6.06
	UPPER LIMIT	490672	3.37	1855156	4.57	1006816	6.56
	LOWER LIMIT	122668	2.37	463789	3.57	251704	5.56
	SAMPLE ID						
1	SVE7D101	239309	2.86	866575	4.06	473493	6.06
2	SVE7D102	230716	2.87	847950	4.06	448973	6.06
3	SVE7D103	237631	2.87	896721	4.07	495041	6.07
4	SVE7D104	243979	2.87	900688	4.07	497255	6.06
5	SVE7D106	243684	2.87	899798	4.07	491550	6.06
6	SVE7D107	249787	2.87	909145	4.07	501058	6.06
7	SVE7D108	241130	2.87	887123	4.06	477950	6.06
8	ISVE7D101	236184	2.87	892727	4.07	490143	6.06

IS1 (DCB) = 1,4-Dichlorobenzene-d4
IS2 (NPT) = Naphthalene-d8
IS3 (ANT) = Acenaphthene-d10

AREA UPPER LIMIT = +100% of internal satndard area
AREA LOWER LIMIT = - 50% of internal standard area
RT UPPER LIMIT = +0.50 minutes of internal standard RT
RT LOWER LIMIT = -0.50 minutes of internal standard RT

Column used to flag internal standard area values with an asterisk
* Values outside of QC limits.

page 1 of 1

FORM VIII SV-1 OLM02.0

Figure 7B: TYPICAL INTERNAL STANDARD AREA AND RT SUMMARY SV-2

8C

SEMIVOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name: EMAX Inc
Lab Code: EMXT
Lab File ID: RDH120
Instrument ID: TOE7

Project:ICAL
SDG No.:CAL\8270\2014
Date Analyzed: 04/10/14
Time Analyzed: 17:08

	IS4(PHN) AREA #	RT #	IS5(CRY) AREA #	RT #	IS6(PRY) AREA #	RT #
12 HOUR STD	939603	7.80	976667	10.57	953304	11.93
UPPER LIMIT	1879206	8.30	1953334	11.07	1906608	12.43
LOWER LIMIT	469802	7.30	488334	10.07	476652	11.43
SAMPLE ID						
1 SVE7D101	913375	7.80	935897	10.56	923517	11.93
2 SVE7D102	873339	7.80	901672	10.56	894094	11.93
3 SVE7D103	928269	7.80	971213	10.57	940714	11.93
4 SVE7D104	936666	7.80	985427	10.57	952812	11.93
5 SVE7D106	931801	7.80	980634	10.57	946988	11.93
6 SVE7D107	958223	7.80	983938	10.57	969359	11.93
7 SVE7D108	926152	7.80	941776	10.56	937143	11.92
8 ISVE7D101	919199	7.80	962830	10.57	949618	11.93

IS4 (PHN) = Phenanthrene-d10
IS5 (CRY) = Chrysene-d12
IS6 (PRY) = Perylene-d12

AREA UPPER LIMIT = +100% of internal satndard area
AREA LOWER LIMIT = - 50% of internal standard area
RT UPPER LIMIT = +0.50 minutes of internal standard RT
RT LOWER LIMIT = -0.50 minutes of internal standard RT

Column used to flag internal standard area values with an asterisk
* Values outside of QC limits.

page 1 of 1

FORM VIII SV-2 OLM02.0

Figure 8: TYPICAL SAMPLE RESULT

METHOD SW3550B/8270C
SEMI VOLATILE ORGANICS BY GC/MS

Client : XYZ, INC.	Date Collected: 06/09/14
Project : CLEAN PROJECT	Date Received: 06/10/14
Batch No. : 14F050	Date Extracted: 06/19/14 12:14
Sample ID: 507-DR-SO-011	Date Analyzed: 06/19/14 21:17
Lab Samp ID: F050-12	Dilution Factor: 1
Lab File ID: RFH297	Matrix : SOIL
Ext Btch ID: SVF031S	% Moisture : 21.5
Calib. Ref.: RDH120	Instrument ID : T-OE7

PARAMETERS	RESULTS (ug/kg)	LOQ (ug/kg)	LOD (ug/kg)
1,2,4-TRICHLOROBENZENE	ND	420	210
1,2-DICHLOROBENZENE	ND	420	210
1,2-DIPHENYLHYDRAZINE	ND	420	210
1,3-DICHLOROBENZENE	ND	420	210
1,4-DICHLOROBENZENE	ND	420	210
2,4,5-TRICHLOROPHENOL	ND	420	210
2,4,6-TRICHLOROPHENOL	ND	420	210
2,4-DICHLOROPHENOL	ND	420	210
2,4-DIMETHYLPHENOL	ND	420	210
2,4-DINITROPHENOL	ND	850	210
2,4-DINITROTOLUENE	ND	420	210
2,6-DINITROTOLUENE	ND	420	210
2-CHLORONAPHTHALENE	ND	420	210
2-CHLOROPHENOL	ND	420	210
2-METHYLNAPHTHALENE	ND	420	210
2-METHYLPHENOL	ND	420	210
2-NITROANILINE	ND	420	210
2-NITROPHENOL	ND	420	210
3,3'-DICHLOROBENZIDINE	ND	420	210
4-METHYLPHENOL (1)	ND	420	210
3-NITROANILINE	ND	420	210
4,6-DINITRO-2-METHYLPHENOL	ND	850	210
4-BROMOPHENYL-PHENYL ETHER	ND	420	210
4-CHLORO-3-METHYLPHENOL	ND	420	210
4-CHLOROANILINE	ND	420	210
4-CHLOROPHENYL-PHENYL ETHER	ND	420	210
4-NITROANILINE	ND	420	210
4-NITROPHENOL	ND	850	210
ACENAPHTHENE	ND	420	210
ACENAPHTHYLENE	ND	420	210
ANTHRACENE	ND	420	210
BENZO(A)ANTHRACENE	ND	420	210
BENZO(A)PYRENE	ND	420	210
BENZO(B)FLUORANTHENE	ND	420	210
BENZO(G,H,I)PERYLENE	ND	420	210
BENZO(K)FLUORANTHENE	ND	420	210
BENZOIC ACID	ND	1700	850
BENZYL ALCOHOL	ND	420	210
2,2'-OXYBIS(1-CHLOROPROPANE)	ND	420	210
BIS(2-CHLOROETHOXY)METHANE	ND	420	210
BIS(2-CHLOROETHYL)ETHER	ND	420	210
BIS(2-ETHYLHEXYL)PHTHALATE	ND	420	210
BUTYLBENZYLPHTHALATE	ND	420	210
CARBAZOLE	ND	420	210
CHRYSENE	ND	420	210
DIBENZO(A,H)ANTHRACENE	ND	420	210
DIBENZOFURAN	ND	420	210
DIETHYLPHTHALATE	ND	420	210
DIMETHYLPHTHALATE	ND	420	210
DI-N-BUTYLPHTHALATE	ND	420	210
DI-N-OCTYLPHTHALATE	ND	420	210
FLUORANTHENE	ND	420	210
FLUORENE	ND	420	210
HEXACHLOROBENZENE	ND	420	210
HEXACHLOROBUTADIENE	ND	420	210
HEXACHLOROETHANE	ND	420	210

Figure 8 (cont.): TYPICAL SAMPLE RESULT

INDENO(1,2,3-CD)PYRENE	ND	420		210
ISOPHORONE	ND	420		210
NAPHTHALENE	ND	420		210
NITROBENZENE	ND	420		210
N-NITROSODIMETHYLAMINE	ND	420		210
N-NITROSO-DI-N-PROPYLAMINE	ND	420		210
N-NITROSODIPHENYLAMINE (2)	ND	420		210
PENTACHLOROPHENOL	ND	850		210
PHENANTHRENE	ND	420		210
PHENOL	ND	420		210
PYRENE	ND	420		210
BENZIDINE	ND	2500		1100
2,6-DICHLOROPHENOL	ND	420		210

SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
2,4,6-TRIBROMOPHENOL	1950	2548	76.6	35-125
2-FLUOROBIPHENYL	520	849.3	61.2	45-105
2-FLUOROPHENOL	1380	2548	54.1	35-105
NITROBENZENE-D5	455	849.3	53.6	35-100
PHENOL-D5	1460	2548	57.1	40-100
TERPHENYL-D14	610	849.3	71.8	30-125

(1): Cannot be separated from 3-Methylphenol
(2): Cannot be separated from Diphenylamine

Figure 9: TYPICAL LCS/LCSD SUMMARY

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN PROJECT
BATCH NO.: 14F050
METHOD: SW3550B/8270C

MATRIX: SOIL % MOISTURE: NA
DILUTION FACTOR: 1 1 1
SAMPLE ID: MBLK1S
LAB SAMP ID: SVF031SB SVF031SL SVF031SC
LAB FILE ID: RFH277 RFH278 RFH279
DATE EXTRACTED: 06/19/1412:14 06/19/1412:14 06/19/1412:14 DATE COLLECTED: NA
DATE ANALYZED: 06/19/1414:50 06/19/1415:09 06/19/1415:28 DATE RECEIVED: 06/19/14
PREP. BATCH: SVF031S SVF031S SVF031S
CALIB. REF: RDH120 RDH120 RDH120

ACCESSION:

PARAMETER	BLNK RSLT (ug/kg)	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	SPIKE AMT (ug/kg)	BSD RSLT (ug/kg)	BSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
1,2,4-Trichlorobenzene	ND	1330	884	66	1330	869	65	2	45-110	50
1,2-Dichlorobenzene	ND	1330	971	73	1330	974	73	0	45-100	50
1,2-Diphenylhydrazine	ND	1330	1070	80	1330	1010	76	6	30-130	50
1,3-Dichlorobenzene	ND	1330	960	72	1330	943	71	2	40-100	50
1,4-Dichlorobenzene	ND	1330	980	74	1330	965	72	2	35-105	50
2,4,5-Trichlorophenol	ND	1330	981	74	1330	939	70	4	50-110	50
2,4,6-Trichlorophenol	ND	1330	1130	85	1330	1080	81	5	45-110	50
2,4-Dichlorophenol	ND	1330	1110	83	1330	1060	80	4	45-110	50
2,4-Dimethylphenol	ND	1330	1120	84	1330	1080	81	4	30-105	50
2,4-Dinitrophenol	ND	1330	936	70	1330	916	69	2	15-130	50
2,4-Dinitrotoluene	ND	1330	1080	81	1330	1060	80	1	50-115	50
2,6-Dinitrotoluene	ND	1330	1040	78	1330	1030	77	2	50-110	50
2-Chloronaphthalene	ND	1330	1030	77	1330	994	75	4	45-105	50
2-Chlorophenol	ND	1330	1210	91	1330	1170	88	4	45-105	50
2-Methylnaphthalene	ND	1330	942	71	1330	932	70	1	45-105	50
2-Methylphenol	ND	1330	973	73	1330	956	72	2	40-105	50
2-Nitroaniline	ND	1330	1050	78	1330	1020	76	3	45-120	50
2-Nitrophenol	ND	1330	1280	96	1330	1110	84	14	40-110	50
3,3'-Dichlorobenzidine	ND	1330	1130	84	1330	1060	79	6	10-130	50
4-Methylphenol	ND	1330	1040	78	1330	985	74	5	40-105	50
3-Nitroaniline	ND	1330	1060	80	1330	1010	76	5	25-110	50
4,6-Dinitro-2-Methylphenol	ND	1330	1330	100	1330	1280	96	4	30-135	50
4-Bromophenyl-phenyl ether	ND	1330	1170	88	1330	1110	83	6	45-115	50
4-Chloro-3-Methylphenol	ND	1330	1220	91	1330	1150	86	5	45-115	50
4-Chloroaniline	ND	1330	958	72	1330	936	70	2	10-100	50
4-Chlorophenyl-phenyl ether	ND	1330	1050	79	1330	1010	76	4	45-110	50
4-Nitroaniline	ND	1330	952	71	1330	925	69	3	35-115	50
4-Nitrophenol	ND	1330	865	65	1330	837	63	3	15-140	50
Acenaphthene	ND	1330	1050	79	1330	1030	77	2	45-110	50
Acenaphthylene	ND	1330	1050	79	1330	1030	77	2	45-105	50
Anthracene	ND	1330	1120	84	1330	1060	80	5	55-105	50
Benzo(a)anthracene	ND	1330	1100	82	1330	1060	79	4	50-110	50
Benzo(a)pyrene	ND	1330	1130	84	1330	1080	81	4	50-110	50
Benzo(b)fluoranthene	ND	1330	1080	81	1330	1080	81	1	45-115	50
Benzo(g,h,i)perylene	ND	1330	1130	84	1330	1080	81	4	40-125	50
Benzo(k)fluoranthene	ND	1330	1150	86	1330	1050	79	9	45-125	50
Benzoic Acid	ND	2670	1720	64	2670	1730	65	1	0-110	50
Benzyl Alcohol	ND	1330	963	72	1330	930	70	4	20-125	50
2,2'-oxybis(1-chloropropane)	ND	1330	1120	84	1330	1100	82	2	20-115	50
bis(2-Chloroethoxy)methane	ND	1330	1050	79	1330	1010	76	3	45-110	50
bis(2-Chloroethyl)ether	ND	1330	1020	77	1330	978	73	4	40-105	50
bis(2-Ethylhexyl)phthalate	ND	1330	1200	90	1330	1130	85	6	45-125	50
Butylbenzylphthalate	ND	1330	1220	91	1330	1160	87	5	50-125	50
Carbazole	ND	1330	1040	78	1330	1000	75	4	45-115	50
Chrysene	ND	1330	1090	82	1330	1050	79	4	55-110	50
Dibenzo(a,h)anthracene	ND	1330	1190	89	1330	1150	86	3	40-125	50
Dibenzofuran	ND	1330	1100	82	1330	1060	79	4	50-105	50
Diethylphthalate	ND	1330	1110	83	1330	1050	79	6	50-115	50
Dimethylphthalate	ND	1330	940	71	1330	899	67	4	50-110	50
Di-n-butylphthalate	ND	1330	1290	97	1330	1290	97	0	55-110	50
Di-n-octylphthalate	ND	1330	1150	86	1330	1100	83	4	40-130	50
Fluoranthene	ND	1330	1010	76	1330	966	72	5	55-115	50
Fluorene	ND	1330	1050	79	1330	1010	75	4	50-110	50

Figure 9: TYPICAL LCS/LCSD SUMMARY

Hexachlorobenzene	ND	1330	1220	91	1330	1200	90	2	45-120	50
Hexachlorobutadiene	ND	1330	829	62	1330	817	61	2	40-115	50
Hexachloroethane	ND	1330	967	73	1330	966	72	0	35-110	50
Indeno(1,2,3-cd)pyrene	ND	1330	1120	84	1330	1090	82	3	40-120	50
Isophorone	ND	1330	1120	84	1330	1090	82	3	45-110	50
Naphthalene	ND	1330	932	70	1330	907	68	3	40-105	50
Nitrobenzene	ND	1330	964	72	1330	951	71	1	40-115	50
N-Nitrosodimethylamine	ND	1330	1050	78	1330	1040	78	0	20-115	50
n-Nitroso-di-n-propylamine	ND	1330	1070	80	1330	1050	79	2	40-115	50
n-Nitrosodiphenylamine	ND	1330	1220	91	1330	1160	87	5	50-115	50
Pentachlorophenol	ND	1330	1010	75	1330	975	73	3	25-120	50
Phenanthrene	ND	1330	1150	86	1330	1080	81	6	50-110	50
Phenol	ND	1330	1120	84	1330	1070	80	4	40-100	50
Pyrene	ND	1330	1120	84	1330	1050	79	6	45-125	50
Benzidine	ND	2670	1450J	54	2670	1390J	52	4	30-150	50
2,6-Dichlorophenol	ND	1330	1020	77	1330	1010	76	1	10-150	50

SURROGATE PARAMETER	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	SPIKE AMT (ug/kg)	BSD RSLT (ug/kg)	BSD % REC	QC LIMIT (%)
2,4,6-Tribromophenol	2000	2110	105	2000	2030	101	35-125
2-Fluorobiphenyl	667	543	81	667	545	82	45-105
2-Fluorophenol	2000	1640	82	2000	1620	81	35-105
Nitrobenzene-d5	667	532	80	667	534	80	35-100
Phenol-d5	2000	1740	87	2000	1690	84	40-100
Terphenyl-d14	667	684	103	667	669	100	30-125

Figure 10: TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN PROJECT
BATCH NO.: 14F050
METHOD: SW3550B/8270C

MATRIX: SOIL % MOISTURE: 21.5

DILUTION FACTOR:	1	1	1		
SAMPLE ID:	507-DR-SO-011				
LAB SAMP ID:	F050-12	F050-12M	F050-12S		
LAB FILE ID:	RFH297	RFH282	RFH283		
DATE EXTRACTED:	06/19/1412:14	06/19/1412:14	06/19/1412:14	DATE COLLECTED:	06/09/14
DATE ANALYZED:	06/19/1421:17	06/19/1416:26	06/19/1416:46	DATE RECEIVED:	06/10/14
PREP. BATCH:	SVF031S	SVF031S	SVF031S		
CALIB. REF:	RDH120	RDH120	RDH120		

ACCESSION:

PARAMETER	SMPL RSLT (ug/kg)	SPIKE AMT (ug/kg)	MS RSLT (ug/kg)	MS % REC	SPIKE AMT (ug/kg)	MSD RSLT (ug/kg)	MSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
1,2,4-Trichlorobenzene	ND	1700	1120	66	1700	1130	67	1	45-110	50
1,2-Dichlorobenzene	ND	1700	1170	69	1700	1190	70	1	45-100	50
1,2-Diphenylhydrazine	ND	1700	1370	81	1700	1420	84	4	30-130	50
1,3-Dichlorobenzene	ND	1700	1160	69	1700	1180	69	1	40-100	50
1,4-Dichlorobenzene	ND	1700	1180	69	1700	1170	69	1	35-105	50
2,4,5-Trichlorophenol	ND	1700	1340	79	1700	1400	82	4	50-110	50
2,4,6-Trichlorophenol	ND	1700	1510	89	1700	1560	92	3	45-110	50
2,4-Dichlorophenol	ND	1700	1370	81	1700	1430	84	4	45-110	50
2,4-Dimethylphenol	ND	1700	1370	81	1700	1410	83	3	30-105	50
2,4-Dinitrophenol	ND	1700	1330	79	1700	1310	77	2	15-130	50
2,4-Dinitrotoluene	ND	1700	1450	85	1700	1480	87	2	50-115	50
2,6-Dinitrotoluene	ND	1700	1370	80	1700	1430	84	5	50-110	50
2-Chloronaphthalene	ND	1700	1350	79	1700	1400	82	4	45-105	50
2-Chlorophenol	ND	1700	1300	77	1700	1330	78	2	45-105	50
2-Methylnaphthalene	ND	1700	1180	70	1700	1220	72	3	45-105	50
2-Methylphenol	ND	1700	1180	69	1700	1190	70	1	40-105	50
2-Nitroaniline	ND	1700	1410	83	1700	1440	85	2	45-120	50
2-Nitrophenol	ND	1700	1390	82	1700	1440	85	4	40-110	50
3,3'-Dichlorobenzidine	ND	1700	1470	86	1700	1570	92	7	10-130	50
4-Methylphenol	ND	1700	1380	81	1700	1300	76	6	40-105	50
3-Nitroaniline	ND	1700	1400	82	1700	1450	86	4	25-110	50
4,6-Dinitro-2-Methylphenol	ND	1700	1770	104	1700	1850	109	5	30-135	50
4-Bromophenyl-phenyl ether	ND	1700	1500	88	1700	1590	93	6	45-115	50
4-Chloro-3-Methylphenol	ND	1700	1520	89	1700	1540	91	1	45-115	50
4-Chloroaniline	ND	1700	1160	68	1700	1210	71	4	10-100	50
4-Chlorophenyl-phenyl ether	ND	1700	1330	78	1700	1370	81	3	45-110	50
4-Nitroaniline	ND	1700	1190	70	1700	1270	75	6	35-115	50
4-Nitrophenol	ND	1700	1180	69	1700	1220	72	3	15-140	50
Acenaphthene	ND	1700	1370	81	1700	1410	83	3	45-110	50
Acenaphthylene	ND	1700	1370	80	1700	1430	84	4	45-105	50
Anthracene	ND	1700	1390	82	1700	1470	87	6	55-105	50
Benzo(a)anthracene	ND	1700	1360	80	1700	1420	84	5	50-110	50
Benzo(a)pyrene	ND	1700	1390	82	1700	1480	87	7	50-110	50
Benzo(b)fluoranthene	ND	1700	1410	83	1700	1560	92	10	45-115	50
Benzo(g,h,i)perylene	ND	1700	1380	81	1700	1430	84	4	40-125	50
Benzo(k)fluoranthene	ND	1700	1390	82	1700	1380	81	1	45-125	50
Benzoic Acid	ND	3400	1530J	45	3400	1870	55	20	0-110	50
Benzyl Alcohol	ND	1700	1180	69	1700	1180	69	0	20-125	50
2,2'-oxybis(1-chloropropane)	ND	1700	1350	79	1700	1360	80	1	20-115	50
bis(2-Chloroethoxy)methane	ND	1700	1260	74	1700	1300	77	4	45-110	50
bis(2-Chloroethyl)ether	ND	1700	1190	70	1700	1230	73	3	40-105	50
bis(2-Ethylhexyl)phthalate	ND	1700	1400	82	1700	1520	89	8	45-125	50
Butylbenzylphthalate	ND	1700	1400	82	1700	1510	89	8	50-125	50
Carbazole	ND	1700	1340	79	1700	1450	85	8	45-115	50
Chrysene	ND	1700	1340	79	1700	1430	84	6	55-110	50
Dibenzo(a,h)anthracene	ND	1700	1420	84	1700	1510	89	6	40-125	50
Dibenzofuran	ND	1700	1420	84	1700	1470	87	3	50-105	50
Diethylphthalate	ND	1700	1390	82	1700	1450	85	4	50-115	50
Dimethylphthalate	ND	1700	1210	71	1700	1240	73	3	50-110	50
Di-n-butylphthalate	ND	1700	1470	87	1700	1620	96	10	55-110	50
Di-n-octylphthalate	ND	1700	1400	83	1700	1470	86	5	40-130	50
Fluoranthene	ND	1700	1380	81	1700	1470	87	6	55-115	50
Fluorene	ND	1700	1340	79	1700	1390	82	3	50-110	50

Figure 10 (cont.): TYPICAL MS/MSD SUMMARY

Hexachlorobenzene	ND	1700	1500	89	1700	1600	94	6	45-120	50
Hexachlorobutadiene	ND	1700	1050	62	1700	1050	62	1	40-115	50
Hexachloroethane	ND	1700	1390	82	1700	1180	70	16	35-110	50
Indeno(1,2,3-cd)pyrene	ND	1700	1370	81	1700	1460	86	6	40-120	50
Isophorone	ND	1700	1350	80	1700	1250	74	8	45-110	50
Naphthalene	ND	1700	1160	68	1700	1170	69	1	40-105	50
Nitrobenzene	ND	1700	1190	70	1700	1200	71	1	40-115	50
N-Nitrosodimethylamine	ND	1700	1270	75	1700	1250	74	1	20-115	50
n-Nitroso-di-n-propylamine	ND	1700	1290	76	1700	1320	78	2	40-115	50
n-Nitrosodiphenylamine	ND	1700	1660	97	1700	1720	101	4	50-115	50
Pentachlorophenol	ND	1700	1360	80	1700	1470	87	8	25-120	50
Phenanthrene	ND	1700	1430	84	1700	1530	90	7	50-110	50
Phenol	ND	1700	1360	80	1700	1370	81	1	40-100	50
Pyrene	ND	1700	1240	73	1700	1330	78	7	45-125	50
Benzidine	ND	3400	1260J	37	3400	1210J	36	4	30-150	50
2,6-Dichlorophenol	ND	1700	1250	74	1700	1310	77	4	10-150	50

SURROGATE PARAMETER	SPIKE AMT (ug/kg)	MS RSLT (ug/kg)	MS % REC	SPIKE AMT (ug/kg)	MSD RSLT (ug/kg)	MSD % REC	QC LIMIT (%)
2,4,6-Tribromophenol	2550	2730	107	2550	2930	115	35-125
2-Fluorobiphenyl	849	724	85	849	752	89	45-105
2-Fluorophenol	2550	1930	76	2550	1940	76	35-105
Nitrobenzene-d5	849	659	78	849	668	79	35-100
Phenol-d5	2550	2070	81	2550	2100	82	40-100
Terphenyl-d14	849	831	98	849	808	95	30-125

Figure 11: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN PROJECT

SDG : 14F050

METHOD SW3550B/8270C
SEMI VOLATILE ORGANICS BY GC/MS

A total of nineteen (19) soil samples were received on 06/10/14 for Semivolatile Organics by GC/MS analysis, Method SW3550B/8270C in accordance with Project QAPP, 2/2013.

Holding Time
Samples were analyzed within the prescribed holding time.

Instrument Performance and Calibration
Instrument tune check was performed prior to calibration. Instrument mass ratios as well as DDT breakdown were evaluated. Results were within acceptance criteria. Tailing factor for Benzidine and Pentachlorophenol were also verified and results were within the method limits. Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using secondary source (ICV). Continuing calibration (CCV) was carried on at a frequency required by the project. All project calibration requirements were satisfied. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank
Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Results were compliant to project requirement.

Lab Control Sample
Two (2) sets of LCS/LCD were analyzed with the samples in this SDG.
Percent recoveries for SVF031SL/C were all within QC limits.
Percent recoveries for A9F031SL/C were all within QC limits.

Matrix QC Sample
A set of MS/MSD was analyzed with the samples in this SDG.
Percent recoveries for F050-12M/S were within project QC limits.

Surrogate
Surrogates were added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis
Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter. Sample F050-14 was initially analyzed at dilution due to dark-colored appearance in extract.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
DFTPP Tune Check	Prior to calibration (ICAL, ICV or CCV)	Refer to Table 7.	Re-tune instrument and verify.		
Breakdown Check	Prior to calibration (ICAL, ICV or CCV)	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, Benzidine tailing ≤3, Pentachlorophenol tailing <5.	Clean the injection port and repeat breakdown check. If problem persists, cut or replace column.		
Multi point Initial Calibration (ICAL) minimum of 5 points	Initially; as needed.	SPCCs (Table 11): Average RF ≥ 0.05 CCCs (Table 10): R5D ≤ 30% 1.) If RRF is applied, then R5D ≤ 15% 2.) If 1st order is applied, then r≥ 0.995 with min. 5 pt ICAL 3.) if 2nd order is applied, then COD ≥0.99 with min 6 pt ICAL	Check for outliers. Otherwise, optimize the instrument then repeat initial calibration.		
Initial Calibration Verification (ICV)	After initial calibration	All analytes within ± 20% of expected value. * Exclusions Analytes within 35% of expected value. Height of valley between benzo(b)fluoranthene & benzo(k)fluoranthene is less than 25% of the sum of the two peak heights.	Verify second source standard. Prepare fresh standard and re-run ICV. If that fails, optimize instrument and repeat ICAL.		
Evaluation of Relative Retention Times (RRT)	Each sample	Within ± 0.06 RRT units of the RRT	Correct the problem then re-analyze all samples analyzed since the last retention time check.		
Continuing Calibration Verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time	5PCCs: Min. RF same as ICAL CCCs: %Diff ≤20% (when using RFs) or drift (when using least squares regression or non-linear calibration) Height of valley between benzo(b)fluoranthene & benzo(k)fluoranthene is less than 25% of the sum of the two peak heights.	Correct the problem , then repeat initial calibration.		
Internal Standard (IS)	Every sample, spiked sample, standard, and method blank	Retention time ±30 seconds from retention time of the mid-point standard in the ICAL.; EICP area within -50% to +100% of ICAL mid-point standard	Inspect mass spectrometer and GC for malfunction; mandatory re-analysis of samples analyzed while system was malfunctioning.		
Method Blank (MB)	One per preparation batch	In the absence of P5R apply No analytes detected > ½ LOQ	Rule-out instrument contamination by re-analyzing the MB. If problem persists, refer to P5R. In the absence of P5R, report NDs and results > 10X of the MB concentration. Otherwise, cure contamination source, re-prep and re-analyze method blank and all associated samples.		
LCS	One LCS per preparation batch	In the absence of P5R default to EMAX QC Limits	Re-prep and re-analyze the LCS and all associated samples.		
MS/MSD	One MS/MSD per every 20 project samples per matrix	In the absence of P5R default to EMAX QC Limits	Check is sample was properly spiked. If indicative of matrix interference, discuss in the case narrative, otherwise re-prep and re-analyze the sample.		
Surrogate spike	Every sample, MB, LCS, MS/MSD, DCC	In the absence of P5R, at least 2 out of 3 Acids and 2 out of 3 BN surrogates are within EMAX QC Limits.	Check is sample was properly spiked. If indicative of matrix interference, discuss in the case narrative, otherwise re-prep and re-analyze the sample.		
Comments: For flagging criteria refer to P5R. Otherwise, if MB is non-compliant, apply "B" to specific analyte(s) on all associated samples, apply "J" to all values between LOD and LOQ. *Exception Analytes – Analytes known to have erratic chromatographic behavior: Benzidine, 4,6-dinitro-2methylphenol, 4-chloroaniline, benzyl alcohol, n-Nitrosodimethylamine, 4-nitrophenol, 2-nitroaniline, pyridine, benzoic acid and 3-nitroaniline.				Reviewed By: Date:	

Revision approved by: 10/26/17
Caspar Pang, Laboratory Director (Sign/Date)

10/26/17
Kenette Pimentel, QA Manager (Sign/Date)

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
METHOD: EPA 8270

Sample Prep SOP: EMAX-3550 Rev. 4
Analytical SOP: EMAX-8270 Rev. 5
Conc Unit: µg/Kg
Sample Amt(g): 15
Extract Volume (mL): 2

Instrument ID: E7
Extraction date: 3/22 & 3/24/14
Extracted by: J. Villena
Analysis date: 3/24/2014
Analyzed by: D. Jun

PARAMETER	SVC041SL	SVC041SC	SVC042SL	SVC042SC	TV	Ave. Conc.	Ave. %Rec	SD	QC Criteria	COMMENTS
	RCH420	RCH421	RCH422	RCH423						
Acenaphthene	2218	2120	2086	1995	2667	2105	79	92.0	50 - 130	PASSED
Acenaphthylene	2284	2197	2166	2127	2667	2194	82	66.8	50 - 130	PASSED
Aniline	2315	2090	2115	2086	2667	2152	81	109.5	40 - 130	PASSED
Anthracene	2242	2117	2117	2034	2667	2128	80	86.0	50 - 130	PASSED
Azobenzene	2095	1960	1906	1873	2667	1958	73	97.9	30 - 160	PASSED
Benzidine	1865	1743	1869	1716	2667	1798	67	80.4	20 - 130	PASSED
Benzo(a)anthracene	2620	2534	2556	2506	2667	2554	96	48.7	60 - 130	PASSED
benzo(a)pyrene	2478	2411	2481	2392	2667	2440	92	45.8	50 - 130	PASSED
Benzo(b)fluoranthene	2660	2560	2457	2403	2667	2520	94	113.8	60 - 130	PASSED
Benzo(g,h,i)perylene	2505	2378	2659	2356	2667	2475	93	139.3	50 - 130	PASSED
Benzo(k)fluoranthene	2327	2322	2510	2366	2667	2381	89	88.0	60 - 130	PASSED
Benzoic Acid	1116	1090	1330	1206	2667	1185	44	108.3	20 - 130	PASSED
Benzyl Alcohol	2144	1857	1920	1977	2667	1975	74	123.3	50 - 130	PASSED
bis(2-chloroethoxy)methane	2354	2119	2139	1997	2667	2152	81	148.4	50 - 130	PASSED
bis(2-chloroethyl)ether	2160	2073	2073	2011	2667	2079	78	61.4	50 - 130	PASSED
bis(2-chloroisopropyl)ether	2962	2690	2732	2679	2667	2766	104	132.9	30 - 130	PASSED
bis(2-Ethylhexyl)phthalate	2547	2411	2442	2391	2667	2448	92	69.5	60 - 130	PASSED
4-Bromophenyl-phenylether	2274	2232	2179	2101	2667	2197	82	74.5	50 - 130	PASSED
Butylbenzylphthalate	2715	2587	2617	2524	2667	2611	98	79.4	60 - 130	PASSED
Carbazole	2472	2345	2381	2296	2667	2373	89	74.4	50 - 130	PASSED
4-Chloro-3-methylphenol	2623	2485	2427	2239	2667	2443	92	159.5	50 - 130	PASSED
4-Chloroaniline	2273	2046	2002	1981	2667	2076	78	134.3	40 - 130	PASSED
2-Chloronaphthalene	2181	2071	2016	1972	2667	2060	77	90.5	50 - 130	PASSED
2-Chlorophenol	2202	1982	1992	2007	2667	2046	77	104.4	40 - 130	PASSED
4-Chlorophenyl-phenylether	2357	2305	2263	2185	2667	2278	85	72.7	50 - 130	PASSED
Chrysene	2707	2564	2572	2496	2667	2585	97	88.1	60 - 130	PASSED
Dibenzo(a,h)anthracene	2578	2466	2529	2447	2667	2505	94	60.1	60 - 130	PASSED
Dibenzofuran	2256	2126	2080	2037	2667	2125	80	95.1	50 - 130	PASSED
1,2-Dichlorobenzene	2165	1986	2030	2062	2667	2061	77	76.1	50 - 130	PASSED
1,3-Dichlorobenzene	2224	2011	2130	2076	2667	2110	79	90.4	50 - 130	PASSED
1,4-Dichlorobenzene	2198	1983	2104	2025	2667	2077	78	94.5	40 - 130	PASSED
3,3'-Dichlorobenzidine	2972	2745	2853	2746	2667	2829	106	107.8	60 - 130	PASSED
2,4-Dichlorophenol	2219	2044	2038	1929	2667	2058	77	120.1	40 - 130	PASSED
2,6-Dichlorophenol	2343	2146	2232	2037	2667	2190	82	129.7	30 - 160	PASSED
Diethylphthalate	2338	2210	2217	2111	2667	2219	83	92.6	60 - 130	PASSED
2,4-Dimethylphenol	2259	2028	2021	1972	2667	2070	78	128.7	40 - 130	PASSED
Dimethylphthalate	2804	2661	2565	2458	2667	2622	98	147.0	60 - 130	PASSED
Di-n-butylphthalate	2317	2189	2268	2143	2667	2229	84	78.0	60 - 130	PASSED
4,6-Dinitro-2-methylphenol	2332	2126	2224	2260	2667	2236	84	85.7	50 - 140	PASSED
2,4-Dinitrophenol	2325	2218	2275	2177	2667	2249	84	64.6	20 - 130	PASSED

Appendix 2 (cont.): DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
METHOD: EPA 8270

Sample Prep SOP: EMAX-3550 Rev. 4
Analytical SOP: EMAX-8270 Rev. 5
Conc Unit: µg/Kg
Sample Amt(g): 15
Extract Volume (mL): 2

Instrument ID: E7
Extraction date: 3/22 & 3/24/14
Extracted by: J. Villena
Analysis date: 3/24/2014
Analyzed by: D. Jun

PARAMETER	SVC041SL	SVC041SC	SVC042SL	SVC042SC	TV	Ave. Conc.	Ave. %Rec	SD	QC Criteria	COMMENTS
	RCH420	RCH421	RCH422	RCH423						
2,4-Dinitrotoluene	2590	2285	2381	2342	2667	2400	90	132.7	60 - 130	PASSED
2,6-Dinitrotoluene	2386	2378	2246	2237	2667	2312	87	81.4	60 - 130	PASSED
Di-n-octylphthalate	2775	2698	2799	2631	2667	2726	102	76.5	60 - 130	PASSED
Fluoranthene	2577	2483	2519	2332	2667	2478	93	104.4	60 - 130	PASSED
Fluorene	2203	2125	2036	2023	2667	2097	79	84.1	50 - 130	PASSED
Hexachlorobenzene	2174	1969	2021	1918	2667	2020	76	110.5	50 - 130	PASSED
Hexachlorobutadiene	2263	2073	2072	2043	2667	2113	79	101.5	40 - 130	PASSED
Hexachlorocyclopentadiene	1704	1651	1573	1575	2667	1626	61	63.8	20 - 130	PASSED
Hexachloroethane	2251	2071	2102	2082	2667	2127	80	84.0	40 - 130	PASSED
Indeno(1,2,3-cd)pyrene	2498	2420	2495	2372	2667	2446	92	61.1	60 - 130	PASSED
Isophorone	2087	1949	1960	1865	2667	1966	74	91.5	50 - 130	PASSED
1-Methylnaphthalene	2389	2178	2176	2101	2667	2211	83	123.9	50 - 130	PASSED
2-Methylnaphthalene	2689	2520	2428	2406	2667	2511	94	128.8	50 - 130	PASSED
2-Methylphenol	2222	1996	2076	1972	2667	2066	77	113.0	50 - 130	PASSED
4-Methylphenol	2298	2289	2267	2066	2667	2230	84	109.9	50 - 130	PASSED
Naphthalene	2208	2000	2030	1977	2667	2054	77	105.0	50 - 130	PASSED
2-Nitroaniline	2616	2515	2414	2705	2667	2563	96	126.0	50 - 130	PASSED
3-Nitroaniline	2503	2434	2364	2253	2667	2388	90	106.6	50 - 130	PASSED
4-Nitroaniline	2657	2497	2560	2487	2667	2550	96	78.3	50 - 130	PASSED
Nitrobenzene	2210	2044	2027	1995	2667	2069	78	96.3	50 - 130	PASSED
2-Nitrophenol	2631	2520	2405	2372	2667	2482	93	117.9	40 - 130	PASSED
4-Nitrophenol	2333	2258	2316	2303	2667	2303	86	32.0	40 - 130	PASSED
n-Nitrosodimethylamine	1914	1721	1784	1684	2667	1776	67	101.1	40 - 130	PASSED
n-Nitroso-di-n-propylamine	2214	1938	1972	1933	2667	2014	76	134.2	50 - 130	PASSED
n-Nitrosodiphenylamine	2324	2202	2208	2221	2667	2239	84	57.4	40 - 130	PASSED
Pentachlorophenol	2233	2091	2121	2013	2667	2114	79	91.5	30 - 130	PASSED
Phenanthrene	2216	2105	2104	2030	2667	2114	79	76.7	50 - 130	PASSED
Phenol	2125	1980	2038	1906	2667	2012	75	92.5	50 - 130	PASSED
Pyrene	2549	2398	2434	2326	2667	2427	91	93.1	50 - 130	PASSED
Pyridine	1679	1552	1668	1591	2667	1623	61	61.2	30 - 130	PASSED
2,3,4,6-Tetrachlorophenol	2374	2214	2222	2137	2667	2237	84	99.4	30 - 160	PASSED
1,2,4-Trichlorobenzene	2199	1997	2070	1968	2667	2059	77	102.8	40 - 130	PASSED
2,4,5-Trichlorophenol	2330	2223	2257	2070	2667	2220	83	109.8	50 - 130	PASSED
2,4,6-Trichlorophenol	2170	2040	1988	1995	2667	2048	77	84.3	50 - 130	PASSED
2-Fluorophenol	3555	3234	3374	3295	4000	3364	84	139.2	40 - 130	PASSED
Phenol-d5	3636	3338	3424	3264	4000	3415	85	161.0	50 - 130	PASSED
2-Fluorobiphenyl	1122	1065	1093	1052	1333	1083	81	31.3	40 - 130	PASSED
Nitrobenzene-d5	1136	1009	1066	974	1333	1046	78	70.7	40 - 130	PASSED
2,4,6-Tribromophenol	3419	3256	3355	3093	4000	3281	82	142.0	50 - 130	PASSED
Terphenyl-d14	1489	1427	1478	1453	1333	1462	110	27.6	60 - 130	PASSED

Appendix 3: DEMONSTRATION OF CAPABILITY FOR APPENDIX IX COMPOUNDS

DEMONSTRATION OF CAPABILITY
METHOD: SW 3550C / SW 8270C/D
APPENDIX IX COMPOUNDS

Sample Prep SOP: EMAX-3550
Analytical SOP: EMAX-8270
Conc Unit: µg/Kg
Sample Amt(gm): 30
Extract Volume (mL): 2

Instrument ID: E4
Extraction date: 6/24/2011
Extracted by: J. Villena
Analysis date: 6/27 & 6/28/11
Analyzed by: D. Cheung

PARAMETER	RFJ303	RFJ304	RFJ343	RFJ344	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	A9F044SL	A9F044SC	A9F045SL	A9F045SC							
Acetophenone	1225	1231	1632	1204	1600	1323	83	206.5	16	30 - 150	PASSED
2-acetylaminofluorene	1433	1485	1884	1669	1600	1618	101	204.7	13	30 - 150	PASSED
4-Aminobiphenyl	1502	1589	1895	1650	1600	1659	104	168.6	10	30 - 150	PASSED
Aramite	1526	1586	2057	1770	1600	1735	108	238.9	14	30 - 150	PASSED
Atrazine	1508	1500	1858	1643	1600	1627	102	167.2	10	30 - 150	PASSED
Biphenyl	1235	1256	1600	1247	1600	1334	83	177.5	13	30 - 130	PASSED
Chlorobenzilate	1390	1454	1825	1541	1600	1553	97	192.1	12	30 - 150	PASSED
1-Chloronaphthalene	1266	1295	1616	1277	1600	1363	85	168.7	12	30 - 150	PASSED
Diallate	1272	1303	1638	1490	1600	1426	89	171.3	12	30 - 150	PASSED
Dibenzo[a,j]acridine	1272	1319	1660	1385	1600	1409	88	173.5	12	30 - 150	PASSED
2,6-Dichlorophenol	1228	1225	1633	1248	1600	1334	83	200.0	15	30 - 150	PASSED
Dimethoate	1563	1616	2017	1741	1600	1734	108	202.6	12	30 - 150	PASSED
p-Dimethylaminoazobenze	1374	1394	1776	1544	1600	1522	95	185.8	12	30 - 150	PASSED
7,12-Dimethylben(a)anthracene	1383	1428	1764	1547	1600	1530	96	170.5	11	30 - 150	PASSED
3,3-Dimethylbenzidine	1383	1427	988	1516	1600	1329	83	233.7	18	10 - 150	PASSED
3,4-Dimethylphenol	1172	1194	1610	1291	1600	1317	82	202.2	15	30 - 150	PASSED
Dinoseb	1324	1419	1836	1556	1600	1534	96	222.8	15	30 - 150	PASSED
1,4-Dioxane	1097	1122	1376	1041	1600	1159	72	148.7	13	30 - 150	PASSED
Diphenyl ether	1219	1237	1565	1243	1600	1316	82	166.2	13	30 - 150	PASSED
Disulfoton	1356	1414	1782	1554	1600	1526	95	189.7	12	30 - 150	PASSED
Ethyl methacrylate	1121	1170	1440	1082	1600	1203	75	162.0	13	30 - 150	PASSED
Ethyl methanesulfonate	1217	1252	1589	1202	1600	1315	82	184.0	14	30 - 150	PASSED
Ethyl parathion	1364	1445	1880	1525	1600	1554	97	227.6	15	30 - 150	PASSED
Hexachloropropene	1226	1261	1578	1201	1600	1317	82	176.0	13	30 - 150	PASSED
Isodrin	1308	1276	1642	1449	1600	1419	89	166.5	12	30 - 150	PASSED
Isosafrole	1427	1445	1867	1473	1600	1553	97	210.5	14	30 - 150	PASSED
3-Methylchlolanthrene	1407	1445	1811	1579	1600	1561	98	182.6	12	30 - 150	PASSED
Methyl methanesulfonate	1219	1216	1423	1154	1600	1253	78	117.3	9	30 - 150	PASSED
Methyl parathion	1418	1509	1923	1667	1600	1630	102	221.1	14	30 - 150	PASSED
1,4-Naphthoquinone	1337	1376	1702	1517	1600	1483	93	165.3	11	10 - 150	PASSED
1-Naphthylamine	1285	1288	1660	1396	1600	1407	88	176.1	13	30 - 150	PASSED
2-Naphthylamine	1425	1418	1806	1586	1600	1559	97	182.3	12	30 - 150	PASSED
N-nitrosodiethylamine	1207	1253	1604	1202	1600	1317	82	192.9	15	30 - 150	PASSED
N-Nitrosomethylethylamine	1187	1252	1577	1171	1600	1297	81	190.0	15	30 - 150	PASSED
N-Nitrosomorpholine	1206	1249	1627	1210	1600	1323	83	203.4	15	30 - 150	PASSED
N-Nitrosodi-n-butylamine	1263	1295	1764	1399	1600	1430	89	230.0	16	30 - 150	PASSED
N-Nitrosopiperdine	1231	1247	1647	1266	1600	1348	84	200.2	15	30 - 150	PASSED
N-Nitrosopyrrolidine	1194	1242	1678	1267	1600	1345	84	224.0	17	30 - 150	PASSED
5-Nitro-o-toluidine	1425	1465	1829	1506	1600	1556	97	185.0	12	30 - 150	PASSED
4-Nitroquinoline-N-oxide	1402	1493	1737	1567	1600	1550	97	142.1	9	10 - 150	PASSED
Pentachlorobenzene	1257	1306	1656	1355	1600	1393	87	179.7	13	30 - 150	PASSED

Appendix 3 (cont.): DEMONSTRATION OF CAPABILITY FOR APPENDIX IX COMPOUNDS

DEMONSTRATION OF CAPABILITY
METHOD: SW 3550C / SW 8270C/D
APPENDIX IX COMPOUNDS

Sample Prep SOP: EMAX-3550
Analytical SOP: EMAX-8270
Conc Unit: µg/Kg
Sample Amt(gm): 30
Extract Volume (mL): 2

Instrument ID: E4
Extraction date: 6/24/2011
Extracted by: J. Villena
Analysis date: 6/27 & 6/28/11
Analyzed by: D. Cheung

PARAMETER	RFJ303	RFJ304	RFJ343	RFJ344	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	A9F0445L	A9F0445C	A9F0455L	A9F0455C							
Pentachloroethane	1203	1195	1487	1106	1600	1248	78	165.2	13	30 - 150	PASSED
Pentachloronitrobenzene	1354	1424	1721	1553	1600	1513	95	161.4	11	30 - 150	PASSED
Phenacetin	1476	1527	1853	1585	1600	1610	101	167.8	10	30 - 150	PASSED
p-phenylenediamine	826	886	1211	844	1600	942	59	180.9	19	10 - 150	PASSED
Phorate	1340	1377	1752	1524	1600	1498	94	186.8	12	30 - 150	PASSED
2-Picoline	1036	1063	1338	956	1600	1098	69	165.9	15	10 - 150	PASSED
pronamide	1456	1506	1877	1625	1600	1616	101	187.7	12	30 - 150	PASSED
Safrole	1221	1263	1667	1277	1600	1357	85	208.0	15	30 - 150	PASSED
Sulfotepp	1317	1376	1723	1534	1600	1487	93	181.8	12	30 - 150	PASSED
1,2,4,5-Tetrachlorobenzene	1233	1269	1624	1246	1600	1343	84	187.8	14	30 - 150	PASSED
Thionazin	1364	1365	1809	1540	1600	1520	95	210.1	14	30 - 150	PASSED
o-toluidine	1387	1401	1769	1345	1600	1475	92	196.9	13	30 - 150	PASSED
O,O,O-triethyl phosphorothi	1218	1230	1612	1237	1600	1324	83	192.2	15	30 - 150	PASSED
1,3,5-Trinitrobenzene	1432	1566	1952	1464	1600	1604	100	239.1	15	10 - 150	PASSED
2-Fluorophenol	1602	1611	1767	1545	2000	1632	82	95.0	6	40 - 130	PASSED
Pheno-d5	1584	1616	1876	1623	2000	1675	84	135.3	8	50 - 130	PASSED
Nitrobenzene-d5	522	519	605	531	667	544	82	40.5	7	40 - 130	PASSED
2-Fluorobiphenyl	527	534	617	545	667	556	83	41.2	7	50 - 130	PASSED
2,4,6-Tribromophenol	1788	1860	2086	2059	2000	1948	97	147.2	8	50 - 130	PASSED
Terphenyl-d14	658	678	764	742	667	710	106	50.3	7	50 - 130	PASSED

8270FS: **SAMPLE PREPARATION LOG (Page 1)**

EMAX LABORATORIES, INC.

EXTRACTION LOG
for
SEMIVOLATILES

Page 1

SOP	Rev. #
☐ EMAX-3520	
☐ EMAX-3540	
☐ EMAX-3546	
☐ EMAX-3550	
☐ EMAX-3580	
☐ EMAX-625	

Note: For samples and relevant QCs/Standards extracted, refer to attached extraction sequence.

Comments:

Book #:	ESV-081
Preparation Batch:	
Matrix:	
Micropipette ID:	
Micropipette ID:	

Standards	ID	Amount Added (ml)
Surrogate		
LCS/MS		
LCS/MS		
LCS/MS		
LCS/MS		

Reagent	Lot# / ID
CH_2Cl_2	
Na_2SO_4	
H_2SO_4	
NaOH	
Silica Sand	
Silica Gel	
Reagent Water	
Residual Chlorine Strip	
pH Strip	

Lab Sample ID	Sonicator #	Cell #	Concentrator #

TUNING	
Sonicator #	Reading

Concentrator	Water Bath Temperature Setting (°C)	Thermometer Reading (°C)
1		
2		
3		
4		
5		
6		
8		

Thermometer ID = SVOC-T1

Prepared By:	Witnessed By:
Standard Added By:	Checked By:
Extract Received By:	Location:
Disposed By:	Disposed On:

8270FS: SAMPLE PREPARATION LOG (Page 2)

PrepBatchID	LabSampleID	Aliquot	Unit	DateTime	Ve(ml)	ExpAmt	ExpVe(ml)	PrepFctr	Comments

Ve=extract volume PrepFctr=(ExpAmt/Aliquot)(Ve/ExpVe)

☐ Extraction Started @ ☐ pHAdj(≤2) @ Prepared By:

☐ Extraction Ended @ ☐ pHAdj(≥11) @ Checked By:

Comments: Date

8270FA: **ANALYTICAL RUN LOG**

EMAX LABORATORIES, INC.

Page 1

ANALYSIS LOG FOR SEMIVOLATILES

SOP ☐ EMAX-8270 Rev. No._ ☐ EMAX-8270D Rev. No._ ☐ EMAX-8270SIM Rev. No._ ☐ EMAX-CLPSVOA ☐ EMAX-M8270SIM Rev. No._ ☐ EMAX-625 Rev. No._ ☐ Book #: AF0-002

Method File: **Tune File:** **Start Date/Time:** **End Date/Time:**

ANALYTICAL BATCH : ______

Preparative Batch	Data File Name	Run ID	DF	Matrix S	Matrix W	Notes

	Instrument No:	F0
INITIAL CALIBRATION REFERENCE		
Date		
ICAL ID		
Standards		
Name	ID	Conc. (mg/L)
DFTPP		
INT. STD.		
ICV		
DCC		
BENZIDINE		
APP 9		
APP 9 ADD		
Solvent	ID	
CH_2Cl_2		
DATA FILE		
Electronic Data Archival		
Location		Date
HPCHEM_SVOA/TOF0		

Micropipette ID: ☐ PO97A-02 ☐ PO97A-03 ☐ PO00-01

Comments: ______

Analyzed By: ______

Date Disposed: ______

Disposed By: ______

This page is checked during data review.

8270FM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

INSTRUMENT MAINTENANCE LOG

Instrument No: F0 Month: Year: 201_ Book #: MF0-003

Date							
Column Head Pressure: 20-40 (psi)							
Run Instrument Blank							
Column Temperature							
Autosampler							
Calibration curve updated							
Verify computer date & clock							
Check printer paper							
Change Septa							
Clean Linear							
Analyst Initial							
TroubleShooting							
Problems Encountered							
Return to Control (Analyst Initial)							

Code	Problems Encountered	Code	Corrective Action

STANDARD OPERATING PROCEDURES

pH, SOLID AND WASTE SAMPLES

SOP No.:	EMAX-9045	Revision No.	4	Date:	22-Jun-17
Prepared By:	Tyler Kosaka			Date:	06-20-17
Approved By:	Kenette Pimentel QA Manager			Date:	06-20-17
Approved By:	Caspar Pang Laboratory Director			Date:	06 2017

Control Number: 9045-04-

1.0 SCOPE AND APPLICATION

1.1. This procedure describes the process of pH determination of soils and waste samples.

1.2. This method is an adaptation of USEPA SW846 Method 9045D.

2.0 SUMMARY OF METHOD

2.1. The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured.

2.2. **Interferences**

2.2.1. Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of > 10, the measured pH may be incorrectly low. Using a low sodium error electrode can minimize this error. Strong acid solutions, with a true pH of < 1, may give incorrectly high pH measurements.

2.2.2. Temperature fluctuations will cause measurement errors.

2.2.3. Errors will occur when the electrodes become coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can undergo one of the following procedures:

2.2.3.1. Clean with an ultrasonic bath.

2.2.3.2. Wash with detergent and rinse several times with water. Place in 1:10 HCl so that the lower third of the electrode is submerged and then thoroughly rinse with water.

2.2.3.3. Clean electrode as per manufacturer's instructions.

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL), Limit of Detection (LOD)**

3.2. DL and LOD are not applicable for this method.

3.3. **Limit of Quantitation (LOQ)**

3.3.1. Refer to EMAX-QA04 for generation, validation and verification of LOQ.

3.3.2. Established LOQ for this method is 1 pH unit.

4.0 DYNAMIC RANGE

4.1. Range: 1 to 14 pH unit

5.0 SAMPLE PRESERVATION AND HOLDING TIME

5.1. Samples should be analyzed as soon as possible

6.0 ASSOCIATED SOPs

6.1.	EMAX-DM01	Data Flow & Review
6.2.	EMAX-QA04	Detection Limit (DL)
6.3.	EMAX-QA05	Training
6.4.	EMAX-QA08	Corrective Action
6.5.	EMAX-QC01	Quality Control for Chemicals
6.6.	EMAX-QC04	Balance Calibration
6.7.	EMAX-QC05	Calibration of Thermometers
6.8.	EMAX-SM03	Waste Disposal
6.9.	EMAX-SM04	Analytical and QC Sample Labeling

7.0 SAFETY

7.1. Read all SDS of chemicals and reagents listed in this SOP.

7.2. Treat reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure.

7.3. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS AND REAGENTS

8.1. **Instruments and Supplies**

8.1.1. pH Meter – Orion 420A or equivalent

8.1.2. Combination electrode

8.1.3. Stirrer – magnetic

8.1.4. 45-ml hinged lid sample container

8.2. **Chemicals and Reagents**

8.2.1. Reagent water: Type II

8.2.2. pH Electrode Storage Solution

9.0 STANDARDS

9.1. **Primary Standard Buffer: Calibration Buffer Standard**

9.1.1. PH 1.0 - BDH or equivalent

9.1.2. pH 4.0 - BDH or equivalent

9.1.3. pH 7.0 - BDH or equivalent

9.1.4. pH 10.0 - BDH or equivalent

9.1.5. pH 12.45 - BDH or equivalent

9.2. **Secondary Standard Buffer: Calibration Check Standard (second source)**

9.2.1. pH 8.0 - RICCA or equivalent

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Soil Samples

10.1.1.1. To 20 g of soil in a 50 mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 min. Additional dilutions are allowed if working with hygroscopic soils and salts or other problematic matrices.

10.1.1.2. Let the soil suspension stand for about 1 hr to allow most of the suspended clay to settle out from the suspension or filter or centrifuge off the aqueous phase for pH measurement.

10.1.1.3. Place the samples (aqueous phase) in water bath at 25 ± 0.4^{o}C to equilibrate.

10.1.2. Waste Samples

10.1.2.1. To 20 g of waste sample in a 50 ml beaker, add 20 ml of reagent water, cover, and continuously stir the suspension for 5 min. Additional dilutions are allowed if working with hygroscopic wastes and salts or other problematic matrices.

10.1.2.2. Let the waste suspension stand for about 15 min. to allow most of the suspended waste to settle out from the suspension or filter or centrifude off aqueous phase for pH measurement.

NOTE: If the waste is hygroscopic and absorbs all the reagent water, begin the experiment again using 20 g of waste and 40 mL of reagent water.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned if it becomes coated with an oily material.

10.1.2.3. Place the samples (aqueous phase) in water bath at 25 ± 0.4^{o}C to equilibrate.

10.2. **Instrument Parameters**

10.2.1. Refer to instrument operating manual.

10.3. **Calibration**

10.3.1. Initial Calibration

10.3.1.1. Transfer about 10 mL of pH 4 buffer solution into a clean properly labeled 20 mL vial. Similarly, transfer pH 7 and pH 10 buffer solutions.

10.3.1.2. Rinse the pH meter electrode thoroughly with reagent water and shake-off the excess water from the electrode.

10.3.1.3. Calibrate the pH meter according to the instrument-operating manual.

10.3.1.4. Record the calibration readings in the analysis run log.

10.3.1.5. Rinse the pH meter electrode thoroughly with reagent water and shake-off the excess water from the electrode.

NOTE: Different pH points can be used in the calibration depending on the pH of the samples. When a sample is found to be pH < 4.0, include ph 1.0 in the calibration. Likewise, if samples is ph > 10.0, include pH 12.45 in the calibration.

10.3.2. Calibration Check Standard (Second Source)

10.3.2.1. Take the pH measurement of a second source standard as described in 10.4.2. Wait until the reading stabilizes. Record the reading.

10.3.2.2. Check Appendix 1 for acceptance criteria.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. Calibration Standards

10.4.1.2. Calibration Check Standard

10.4.1.3. Samples to a maximum of 20

10.4.1.4. Duplicate sample

10.4.2. pH Measurements

10.4.2.1. Rinse the pH meter electrode thoroughly with reagent water and shake-off the excess water from the electrode.

10.4.2.2. Rinse the electrode with the sample to be analyzed.

10.4.2.3. Place a clean magnetic stir bar and turn on the stirrer at a medium speed.

10.4.2.4. Immerse the pH electrode into sample just deep enough into clear supernatant solution. Allow the electrode to stabilize for at least 1 minute or until the readout is stable.

10.4.2.5. Record the pH reading and the sample temperature in °C. Report the final pH reading to the nearest 0.01.

10.5. **Calculations**

10.5.1. Calculate Difference of Duplicate Samples

Difference = $|pH_1 - pH_2|$ Eq.-10.5.1

Where:

pH_1 = pH reading of first measurement

pH_2 = pH reading of second measurement

10.6. **Data Reduction**

10.6.1. Print a copy of the run log.

10.6.2. Highlight the data to be reported.

10.6.3. Keep all other data generated with the analytical folder marked with "For record only".

10.7. **Report Generation**

10.7.1. Generate the method.txt file, sample results and QC summaries using PH.exe.

10.7.2. Generate the case narrative using CN00.exe.

10.7.3. Arrange the analysis package in sequence as detailed below.

10.7.3.1. Case Narrative

10.7.3.2. Sample Results

10.7.3.3. Sample Duplicate Result Summary

10.7.3.4. Run Log

10.7.3.5. Non-Conformance Report (if any)

10.8. **Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

10.8.1.1. Review the generated reports against the run log. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.8.1.2. Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.2. Submit the analytical folder for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Rinse the pH electrode every after use and let it rest on the electrode hanger.

10.9.2. Instruments should receive routine preventive maintenance, which is reported in instrument-specific maintenance logs. Routine maintenance insures that all equipment are operating under optimum conditions, thus reducing the possibility of instrument malfunction, consequently affecting sample results.

11.0 QUALITY CONTROL

11.1. Acceptance criteria for each quality control procedure are summarized in Appendix 1.

11.2. **Analytical Batch QC**

11.2.1. Always use fresh buffer solutions for calibration.

11.2.2. Check standard is analyzed after the initial calibration, after every 20 field samples and at the end of the analysis sequence.

11.2.3. Rinse electrode thoroughly between measurements.

11.2.4. Reagents are subjected to QC check prior to its use. Refer to EMAX-QC01.

11.3. **Preparation Batch QC**

11.3.1. The maximum number of original field samples in a prep batch is 20 unless otherwise specified by the project.

11.3.2. Properly treat all lab wares used in the sample preparation as specified in EMAX-QC07.

11.4. **Method QC**

11.4.1. All analysts conducting this analysis must demonstrate capability as described in EMAX-QA05.

12.0 CORRECTIVE ACTIONS

12.1. Corrective action for each quality control procedure is summarized in Appendix 1

12.2. **Analytical Batch QC**

12.2.1. When calibration is non-compliant, consider the following suggestions to correct the problem:

- Check the electrode is clean and properly installed, verify the pH meter parameter setup and make sure that the electrode is clean.
- Rule out calibration buffer degradation/contamination by using a new calibration buffer.
- If problem persist, replace the electrode fill solution [refer to manufacturer's manual] and repeat the calibration process. Otherwise, inform the Supervisor for further guidance.

12.3. **Preparation Batch QC**

12.3.1. For insufficient amount of sample(s), inform the Supervisor immediately for further action.

12.4. A Non-Conformance Report (NCR) is required when the following circumstances occur:

- Anomalies other than specified in Appendix 1, is observed.
- Sample is out of technical holding time.

12.4.1. Refer to EMAX-QA08 for NCR details.

13.0 POLLUTION PREVENTION

13.1. Observe all necessary precautions to avoid spillage of sample and buffer solutions that may go to the wastewater drains.

14.0 WASTE MANAGEMENT

14.1. No samples shall be dumped on the laboratory sink.

14.2. Separate and properly identify all unused expired analytical standards for proper disposal.

14.3. Place all waste generated during analytical process in properly labeled satellite waste containers for proper collection.

14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. **Definition of Terms**

15.1.1. pH – is the intensity factor of hydrogen ions activity in a solution.

15.1.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

15.1.2.1. **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank and a sample duplicate.

15.1.2.2. **Analytical Batch** – is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.3. Limit of Quantitation (LOQ) – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

15.1.4. Safety Data Sheet (SDS) – is a written information concerning a chemical physical properties, toxicity, health hazards, fire hazard and reactivity data including storage, spill and handling precautions.

15.1.5. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.6. Calibration Check Standard - is a standard purchased from a different vendor at a different concentration from the calibration standards to verify the instrument calibration.

15.1.7. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

15.1.8. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.9. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.10. Matrix – is a component or form of a sample.

15.1.11. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.

15.2. **Application of EMAX QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing pH measurement. In instances where there is a project or program QAPP, the requirements given in the project shall take precedence over this SOP.

15.3. **Department of Defense (DoD) and Department of Energy (DoE) Projects**

15.3.1. Samples from DoD and DoE sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, shall be applied.

16.0 REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Method, SW-846 Method 9045D, Rev. 4, Nov. 2004

16.2. EMAX Quality Systems Manual, us updated

17.0 APPENDICES

17.1. **Figures**

17.1.1. Figure 1 Typical Sample Result Summary

17.1.2. Figure 2 Typical Sample Duplicate Result Summary

17.1.3. Figure 3 Typical Case Narrative

17.2. **Appendices**

17.2.1. Appendix 1 Summary of Quality Control Procedures

17.2.2. Appendix 2 Demonstration of Capability

17.3. **Forms**

17.3.1. 9045FA Analytical Run Log

Figure 1: **TYPICAL SAMPLE RESULT**

METHOD SW9045C
PH

Client : XYZ, INC. Matrix : SOLID
Project : CLEAN PROJECT InstrumentID : 53
Batch No. : 17A000

CLIENT SAMPLE ID	EMAX SAMPLE ID	RESULTS (pH Unit)	PREP FACTOR	MOIST (%)	LOQ (pH Unit)	LOD (pH Unit)	ANALYSIS DATETIME	PREPARATION DATETIME	DATA FILE ID	CAL REF	PREP BATCH	COLLECTION DATETIME	RECEIVED DATETIME
XXYYZZ	A000-01	6.45	1	NA	0.1	0.1	01/12/1718:08	01/12/1717:22	17PHA00501	17PHA005	PHA005S	01/10/1708:30	01/12/17
XXYYZZDUP	A000-01D	6.46	1	NA	0.1	0.1	01/12/1718:09	01/12/1717:22	17PHA00502	17PHA005	PHA005S	01/10/1708:30	01/12/17

Figure 2: TYPICAL SAMPLE DUPLICATE RESULT

EMAX QUALITY CONTROL DATA
SAMPLE DUPLICATE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN PROJECT
BATCH NO. : 17A000
METHOD : SW9045C

MATRIX :	SOLID	
DILUTION FACTOR:	1	1
SAMPLE ID :	XXYYZZ	XXYYZZDUP
LAB SAMPLE ID :	A000-01	A000-01D
LAB FILE ID :	17PHA00501	17PHA00502
DATE PREPARED :	01/12/1717:22	01/12/1717:22
DATE ANALYZED :	01/12/1718:08	01/12/1718:09
PREP BATCH :	PHA005S	PHA005S
CALIBRATION REF:	17PHA005	17PHA005

ACCESSION:

PARAMETER	PARENT RESULT (pH Unit)	DUP RESULT (pH Unit)	DIFFERENCE (pH Unit)	MAX DIFF. (+/- pH Unit)
PH	6.45	6.46	.01	0.1

Figure 3: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project: CLEAN PROJECT

SDG : 17A000

METHOD SW9045C
PH

One (1) solid sample was received on 01/12/17 to be analyzed for pH in accordance with Method SW9045C and project specific requirements.

Holding Time
The sample was analyzed within the prescribed holding time.

Calibration
pH meter was calibrated per instrument operating manual instruction. Calibration was verified using a secondary source (ICV). Continuing calibration (CCV) verifications were carried out on a frequency specified by the project. All calibration requirements were within acceptance criteria.

Matrix QC Sample
Sample duplicate was analyzed and RPD was within expected value.

Sample Analysis
The sample was analyzed according to prescribed analytical procedures. Results were evaluated in accordance to project requirements. For this SDG, all quality control requirements were met.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION		1ST Rvw	2ND Rvw
3 Pt. Initial Calibration	Daily	Buffers within ± 0.05 pH unit	Correct the problem and Re-calibrate before sample analysis.			
Calibration Check Standard	After initial calibration and every after 20 field samples	Buffers within ± 0.05 pH unit	Re-analyze. If still outside QC limits, re-calibrate and re-analyze			
Duplicate Sample	One per analytical batch (≤ 20 field samples)	Results within ± 0.1 pH unit	Re-analyze the sample and its duplicate once. If it still fails QC limits, re-calibrate using a fresh buffer solution and re-analyze.			
Comments:				Reviewed By:		
				Date:		

Appendix 2: DEMONSTRATION OF CAPABILITY

SOP: EMAX-9045 Rev. 4

Conc Unit: pH units

Sample Amount (ml): 20

Analysis date: 5/26/2017

Analyzed by: TKosak

PARAMETER	17PHE01201 PHE012WL	17PHE01202 PHE012WC	17PHE01203 PHE012WX	17PHE01204 PHE012WY	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria (pH units)	COMMENTS
pH	7.01	7.03	7.02	7.02	7	7.02	100	0.008	0.1	6.95 - 7.05	PASSED

9045FA: **ANALYTICAL RUN LOG**

PrepBatchID	LabSampleID	Aliquot	Unit	Temp (°C)	Initial pH	DateTime	Final pH	DateTime	Diff Initial &Final pH	pH	Notes

Calibration of pH Meter		InstrumentID:	
BufferID	Value	Reading	DateTime
	4		
	7		
	10		
	Initial CalVer 8		
	Closing CalVer 7		

☐ pH meter autocompensates sample temperature.

SampleDupID	ParentSample Result	SampleDup Result	Diff (+0.1pH Unit)

Applied SOP	Rev.No.	SDGs
EMAX-150.1		
EMAX-4500HB		
EMAX-9040		
EMAX-9045		

Analyzed By: _____

Checked By: _____

Date: _____

Eurofins Air Toxics, LLC
STANDARD OPERATING PROCEDURE

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN SUMMA™ POLISHED CANISTERS BY GC/MS SELECTIVE ION MONITORING

MODIFIED EPA METHODS TO-14A/TO-15

SOP #38

1.0 SCOPE AND APPLICATION

The procedures in this SOP describe the use of Modified EPA Methods TO-14A and TO-15 to determine the concentration of SIM level volatile organic compounds in ambient air using an evacuated specially treated stainless steel canister. In addition, it describes specific adaptations of the method to the analysis of a subset of the EPA Clean Air Act List of target compounds. Details of the analytical procedures and the required QC protocols are provided. A list of target compounds can be found in Appendix A.

2.0 METHOD SUMMARY

2.1 Description

EPA Methods TO-14A and TO-15 describe techniques for the analysis of airborne VOCs collected as whole air samples in evacuated, specially treated stainless steel canisters. An aliquot of up to 400 mL of air is withdrawn from the canister using a mass flow controller and loaded onto a multi-bed hydrophobic sorbent concentrator. After removing water, the focused air sample is then flash heated and swept onto a GC for separation of the VOCs. Compounds are detected using a mass spectrometer operating in the selective ion monitoring (SIM) mode. A summary of the method QC can be found in *Appendix B*.

2.2 Deviations

Modifications to EPA Methods TO-14A and TO-15 used to carry out the analyses of air samples are summarized in Tables 1 and 2.

Table 1. Summary of Method TO-14A Modifications

Requirement	TO-14A	EATL Modifications
Sample Drying System	Nafion Dryer	Multibed hydrophobic sorbent
ICAL %RSD acceptance criteria	≤ 30% RSD for listed 39 VOCs	Follow TO-15 requirements of <30%RSD with 2 of standard compound list allowed out to <40%RSD
Blank and standards	Zero air	UHP Nitrogen provides a higher purity gas matrix than zero air for trace level measurements.
BFB ion abundance criteria	Ion abundance criteria listed in Table 4 of TO-14A	Follow abundance criteria listed in TO-15.
BFB absolute abundance criteria	Within 10% when comparing to the previous daily BFB.	CCV internal standard area counts are compared to ICAL, corrective action when recovery is less than 60%

Table 2. Summary of Method TO-15 Modifications

Requirement	TO-15	EATL Modifications
Blank and standards	Zero air	UHP Nitrogen provides a higher purity gas matrix than zero air for trace level measurements.

3.0 HEALTH AND SAFETY

3.1 Normal laboratory safety precautions must be used when handling samples, preparing standards from neat materials, and analyzing samples. Appropriate eye wear, gloves, and lab coat must be worn when handling any chemical used in this method. All manipulation of standards, solvents, and solutions should be done with the utmost care in the hood. SDS for each chemical should be consulted for specific dangers and precautions.

3.2 Personnel must handle high pressure cylinders safely. This includes transport of cylinders fully secured on a cart. During storage, the cylinders must be secured at all times with a chain. When installing a pressure regulator, stand to the side of the cylinder.

3.3 Care must also be taken when handling syringes to ensure that a needle stick does not occur. ***All personnel installing or performing maintenance on a capillary column must wear eye protection***.

3.4 For information regarding pollution prevention and waste disposal, see Eurofins Air Toxics SOP #24: Storage and Disposal of Hazardous Wastes.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 Sample Containers

An air sample is collected in an evacuated stainless steel Summa™ / canister. Tedlar bags are not appropriate for the collection and analysis of compounds at SIM levels. Upon receipt, the canisters will be approximately in the range of 2.5 - 10" Hg vacuum. Canister receipt vacuum/pressure is reported on the first page of the data report.

4.2 Sample Handling

Prior to analysis, the canister is pressurized per project specific requirements.

4.3 Sample Storage

Samples are stored in the sample cage in the main laboratory. Analysis must occur within 30 days for canisters. Some projects may require a 14 day hold for canisters. The project profile must be reviewed prior to analysis to verify holding time requirements.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Interferences to this method generally include high levels of Carbon Dioxide, water and/or heavy hydrocarbons. Very high levels of moisture in the samples may cause low

internal standard responses which will interfere with accurate compound quantification. In these situations, dry lab blanks may be required between samples to maintain internal standard recoveries. Dilutions for an entire sample set may be required to insure instrumentation function is maintained and internal standard recoveries are acceptable.

5.2 Samples containing high levels of Carbon Dioxide and/or heavy hydrocarbons may need to be diluted greater than the target compound level requires. This ensures that internal standard recoveries meet QC requirements and/or that the system is not contaminated from carryover.

6.0 EQUIPMENT/APPARATUS

6.1 List of Equipment/Apparatus

- TO-15 Air Concentrator Systems
- Column: RTX-624 or Capillary Column or equivalent column
- Gas Chromatograph: Agilent 7890 or equivalent equipped with Electronic Pressure Control and a split/splitless injection port, and a cryo valve
- Mass Spectrometer: Agilent 5975, 5977 or equivalent
- Agilent Chemstation Software for Data Acquisition
- Thru-Put Target Software for Data Analysis
- NIST /NBS54.1K or NIST08.1 Library Search Software

  Certified NIST Traceable VOC cylinder blends
- Power Controllers
- Liquid Nitrogen certified VOC free by TO-15 analysis
- Ultra High Purity Helium (Local Supplier)
- Commercially purchased dilution system to blend working standards from stock cylinder standards utilizing mass flow controllers

6.2 Operating parameters for GC/MS

Since analytical equipment varies in configuration and as a function of intended specific target analytes, operating parameters are optimized on an instrument-by-instrument basis. Instrument specific operating parameters are recorded in the respective Instrument Maintenance Logbook at each work station.

Note: Only trained laboratory staff are authorized to vary the operating parameters under special circumstances. Any time a change is made, it is documented in the instrument maintenance logbook.

7.0 STANDARD PREPARATION

The formula used to calculate the volume required for the preparation of primary and second source standards for gas blends is the following:

C1*V1 = C2*V2,

Where, C1 is the concentration of the gas blend, V1 is the volume of standard to be used in the preparation, C2 is the desired concentration for the resulting standard and V2 is the final volume (at pressure*) of the newly blended standard.

*At 15 psi the final volume for a 6L canister is 12L and for a 1L canister it is 2L (volume reflects a twofold dilution as a function of pressurization).

7.1 Stock Standards

The TO-15 analytes are purchased from a qualified vendor at a concentration of 1-5 ppmv in a high pressure cylinder blend. The standard expires according to the manufacturer's expiration date on the certificate of analysis.

7.2 Working Standards

7.2.1 Starting with a clean evacuated canister, a volume of 50 microliters of purged deionized water is injected to humidify the standard prior to preparation.

7.2.2 Precision Diluter

The Precision Diluter is used to prepare primary and secondary standards for VOC analysis. The diluter prepares dilution of stock standards by monitoring small pressure changes digitally. There are a total of four positions on the diluter that can accommodate high pressure standard cylinders. There is an additional inlet to allow for secondary dilutions of working standards.

7.2.3 For the SIM analysis, the dynamic diluter is configured to blend the working standard at a concentration of 50 ppbv. The 50 ppbv standard is then further diluted to generate two additional lower concentration working standards .All working standards are pressurized to 15 psi with Nitrogen.

7.2.4 Working standards may be used for up to 3 months from the date of preparation. The TO-15 working standard may not be used past the expiration date of the NIST-traceable high pressure cylinder.

7.3 Internal Standard/Surrogate (IS/S) Mix

The IS/S blend may be prepared from a NIST traceable & ISO-17025 certified standard purchased from a qualified vendor. The standard is in the range of 10 ppmv and expires based on the manufacturer's date on the certificate of analysis. A working standard is prepared by taking a known aliquot of the standard and diluting it to achieve a concentration of 2.5 or 5.0 ppmv. As an alternative, the IS/S mixture may be prepared from neat chemicals.

7.4 BFB Check Mix

A certified 25 ng/µL 4-Bromofluorobenzene (BFB) standard purchased from a commercial vendor is utilized for systems allowing for direct injection into the GC injection port. Alternatively, a 1 to 10ppmv NIST-traceable cylinder containing BFB may be used to make a diluted working standard for TO-15 systems without access to the GC injection port.

8.0 CALIBRATION AND QUALITY CONTROL PROCEDURES

The following sections outline the laboratory's routine calibration and quality control procedures. Specific programs such as DoD QSM and projects may require additional QC samples and/or tighter QC criteria. .

8.1 Tuning Criteria

8.1.1 A daily (at the start of every 24 hours) tune check with 4-Bromofluorobenzene (BFB) at 50 ng is analyzed.

8.1.2 The relative abundances of selected ions are tabulated and compared against the criteria in *Appendix C*. Analysis cannot proceed unless all criteria of the tune check are met. The acceptance criteria are based on TO-15 which are wider than tuning criteria outlined in TO-14A.

8.2 Initial Calibration Procedures

Initial Calibration of the GC/MS is achieved via the internal standard technique. The concentrations used for standard analysis typically range from 0.01 to 20 ppbv. Other levels may be added to the calibration per specific client/project request.

8.2.1 Initial Calibration is performed using a minimum of five levels for standard TO14A/TO-15 SIM compounds. The initial calibration points must be analyzed from the lowest concentration point to the highest concentration point. The lowest concentration standard must be less than or equal to the reporting limit (Limit of Quantitation) and must be verified on a quarterly basis.

8.2.1.1 The percent relative standard deviations (%RSD) must be ≤ 30% for all standard compounds, with 2 compounds allowed out to ≤ 40%.

8.2.2 Compounds that are not included in Table A-2 are defined as non-standard compounds. Examples of Non-Standard TO-14A/TO-15 compounds are listed in Table A-2. Initial calibration criteria are established at the time of project set-up and documented in the Project Requirement Table.

8.2.3 After an Initial Calibration has been evaluated and meets laboratory criteria, the midpoint is copied by adding an extension onto the file identifier, and then it is requantified as a Continuing Calibration Verification. All calibration points are then requantified and the Internal Standard area counts and Retention Times for each file are compared to that of the midpoint. Internal Standards in each calibration point compared in this manner must meet laboratory criteria (area

count ± 40% and Retention Time ± 0.33 minutes compared to the CCV). If %D criterion for the CCV (see Section 8.4) is met, analysis of the Initial Calibration Verification Standard may proceed. The Internal Standard area counts and Retention Times in all subsequent samples and QC must meet laboratory criteria (area count ± 40% and Retention Time ± 0.33 minutes compared to the CCV).

8.2.4 The multipoint calibration is constructed by loading varying amounts of the working standards (see Section 7.2) onto the canister interface. The maximum load volume for the air interface system (e.g. 250 mL) defines the dilution factor.

8.2.5 The average relative response factor (RRF) from the initial calibration curve is used to quantitate results.

8.2.6 All Initial Calibrations needing re-analysis of a calibration level require explanation in the Initial Calibration Case Narrative Template. Only one calibration level is allowed for re-analysis per Initial Calibration Curve due to anomalous unacceptable linearity for compound(s). A bad load or unopened can does not count towards a re-analysis. If more than one standard was used for curving an instrument (i.e. 2 or more), and it is obvious that the RF is not linear relative to the other points of the curve, then all points ran from that standard must be re-analyzed. The reason for the reanalysis must be narrated and included with the ICAL raw data.

8.2.7 Initial calibration curves are generated in accordance to appropriate laboratory practices:

8.2.7.1 The lowest or highest calibration levels of an analyte may be dropped from the curve to achieve linearity. This will affect the analyte's reporting limit and/or calibration range. The minimum number of calibration levels must still be used for the curve.

8.2.7.2 An additional mid-range calibration level may be added to achieve linearity.

8.2.7.3 Alternate calibration levels may be used to meet project specific requirements.

8.2.7.4 It is not acceptable to drop a calibration level that is found somewhere in the middle of the curve to achieve linearity. The calibration level that is in question may be re-analyzed and re-quanted into the curve.

8.2.8 All current Initial Calibrations and Method Detection Limit (MDL) studies are kept in a folder near the instrument.

8.2.9 The indicated flow on the instrument must be measured and its units must be calibrated with a NIST flow meter to provide a true indication of the actual flow through the unit before each time an Initial Calibration is performed and/or on a quarterly basis, whichever is more frequent.

8.3 Initial Calibration Verification (ICV) and Laboratory Control Standard (LCS)

8.3.1 To verify that the standards are correct and the accuracy of the calibration, an independently prepared (i.e., same vendor, different lot number, or second vendor) standard containing all target compounds is analyzed after each Initial Calibration Curve and daily prior to sample analysis. The acceptance criterion for the ICV and LCS recoveries are listed in Table A-1. All compounds must recover within 70-130% with the exception of Carbon Tetrachloride and Naphthalene which must recover within 60-140%. Corrective action is initiated when more than 15% of the list exceeds these limits and/or any compound exceeds 50-150%. If the stated criteria are not met, the system is checked and the same standard or a different standard is re-analyzed. In the event that the criteria cannot be met, only short list compounds that meet the criteria may be analyzed, or the instrument is re-calibrated.

8.3.2 ICV and LCS criterion for non-standard compounds is subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon ICV and LCS requirements prior to sample analysis.

8.3.3 Recovery of any compound in the ICV or LCS that is ≤ 50% of the expected value will result in either re-calibration, or analysis on a different instrument. Recovery of any compound that is ≥ 150% of expected value is acceptable if there are no reported detections in related sample analysis. If recovery of any compound is ≥ 150% and there are detected hits for that compound in the sample(s), the samples must be re-analyzed on a different instrument that meets criteria. Analysis may continue, however the system and/or standard preparation should be evaluated. If the problem is determined to be systematic (i.e. occurs on multiple days and/or on multiple instruments), a CAR must be filed to document corrective actions taken.

8.3.4 Some projects require the LCS to be evaluated using DoD QSM specified control limits or statistical limits derived from historical data.

8.3.5 An LCS is analyzed in duplicate (LCSD) daily prior to sample analysis. Refer to section 8.8.1 for the RPD acceptance criteria between the LCS and LCSD.

8.3.6 LCSD % Recovery acceptance criterion must be met for common risk driver compounds and compounds of concern including Benzene, Toluene, Ethyl Benzene, m,p-xylene, Vinyl Chloride, 1,1-Dichloroethene, cis-1,2-Dichloroethene, Trichloroethene, Tetrachloroethene or any other client specified risk driver compounds.

8.3.7 The reporting limit (Limit of Quantitation – LOQ) must be verified quarterly on each instrument that performs the methods the lab is accredited for by DoD-ELAP (Refer to DoD scope at O:\QA\Certifications). The LOQ is verified by evaluating the point of the initial calibration that corresponds to the LOQ. The concentration recovered is used to calculate the precision and bias of each compound. A

minimum of three points is required to perform the calculations. If there are insufficient points, a primary source standard is analyzed at the LOQ. Precision and bias is determined by calculating the % relative standard deviation (% RSD) and the % bias of the mean concentration recovered. The acceptance criterion for the LOQ verification is ≤30% RSD and ≤50% bias for each compound. If more than 10% of the compound list exceeds the acceptance criteria, the instrument and standard used will evaluated to determine the source of the error. Quarterly LOQ verifications are evaluated by the QA Department.

8.4 Continuing Calibration Verification (CCV)

8.4.1 A Continuing Calibration Verification (CCV) is performed at the start of each day after analysis of the BFB tune check. This is an analysis of the primary source at a concentration between the low point and the midpoint of the initial calibration.

8.4.2 The acceptance criteria for the percent difference (%D) between the daily CCV response and average response from the Calibration Curve is as follows:

8.4.2.1 All compounds must be ≤ 30% D with Carbon Tetrachloride and Naphthalene ≤40%D Compounds exceeding this criterion and associated data will be flagged and narrated. If more than 10% of compounds from the standard list exceed SOP criteria, corrective action will be taken. Corrective action may include instrument maintenance, re-calibration, and/or re-preparation of the calibration standards (See 8.4.3). If any compound exceeds 60-140%, samples are not analyzed unless data meets project needs. The QA Manager or Lab Manager may approve exceedance of a compound under special circumstances after reviewing the impact to the data quality. Regardless, associated data will be flagged and narrated. See Table A-1 for the standard compound list.

8.4.2.2 CCV criterion for non-standard compounds is subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon CCV requirements prior to sample analysis.

8.4.3 If the CCV fails to meet the performance criteria, the CCV is re-analyzed and/or another standard is analyzed. If the CCV fails again, maintenance should be performed and the test repeated. If the system still fails the calibration check, a new Initial Calibration is performed. If the CCV passes following maintenance to the instrument, then the LCS must also pass before samples can be analyzed.

8.4.4 Recovery of any compound in the CCV that is ≤ 50% of the expected value will result in either re-calibration, or analysis on a different instrument. Recovery of any compound that is ≥ 150% of expected value is acceptable if there are no reported detections in related sample analysis. If recovery of any compound is ≥ 150% and there are detected hits for that compound in the sample(s), the samples must be re-analyzed on a different instrument that meets criteria. Analysis may continue, however the system and/or standard preparation should be evaluated. If

the problem is determined to be systematic (i.e. occurs on multiple days and/or on multiple instruments), a CAR must be filed to document corrective actions taken.

8.4.4.1 Certain projects have different CCV acceptance criteria (e.g., ≤ 25% for 100% of compounds). A specific list of target analytes is requested when the CCV acceptance criteria required are different from the EATL standard criteria noted above.

8.5 Laboratory Blanks

8.5.1 A humidified Nitrogen Blank is analyzed upon completion of all required QC including calibration standards at the beginning of each day and at least once in every 24-hour shift. A Lab Blank is also analyzed in the event saturation-level concentrations are incurred to demonstrate that contamination does not exist in the chromatographic system. Each system has been evaluated for carryover, and a specific sample load volume or on-column concentration has been determined as the trigger for a Lab Blank. The acceptance criterion for the Lab Blanks is a result less than the laboratory reporting limit (Limit of Quantitation) *(*see *Appendix A,* Table A-1).

8.5.1.1 In the event that the Laboratory Blank is contaminated by target compounds, the Laboratory Blank should be re-analyzed to eliminate the possibility of an anomalous indication. If re-analysis is acceptable, analysis of client samples may continue.

8.5.1.2 Client samples that have analyte sublists that include the compound(s) in question should be substituted for short list samples that do not include the compounds in question. As these samples are analyzed, the operator should monitor the results to determine if the contamination has been removed from the system.

8.5.1.3 If there are no samples in-house that meet the criteria described in 8.5.1.2, the nature of the contamination should be evaluated.

8.5.1.4 Methylene Chloride, Acetone, and 2-Butanone are acknowledged as common laboratory contaminants. While not on the standard SIM list, if these compounds are included as targets, their presence at a concentration of < 5X the reporting limit is acceptable as long as the associated samples are not being analyzed for these analytes only. The associated sample will be flagged. If the contamination persists for more than two analytical batches, the issue should be escalated for evaluation in order to identify and remove the source of contamination.

8.5.1.5 If other target compounds are present in the Laboratory Blank, operations management will determine the impact on the usability of the data based on project requirements. If analysis proceeds, the associated data will be flagged to indicate detection above the RL in the Laboratory Blank.

8.5.1.6 If the analysis request contains Acetone, laboratory blanks are not needed to continue running samples for Acetone above the lab blank 'trigger'. This compound can be present at high levels in air samples and is not a critical risk driver for client projects. If the sample or samples following the high level analysis contain this compound below 5x the reporting limit then a narration must be used describing the potential for high bias of these results. If the result for this compound in the subsequent samples is greater than 5x the reporting limit then no narration is necessary. If a shortened list of analytes is requested and contains this compound, blanks must be used to demonstrate cleanliness of the system.

8.6 Internal Standards (IS)

8.6.1 The Internal Standard retention times for the blanks, QC samples and samples must be within ± 0.33 minutes of the retention times in the continuing calibration check. In addition, the internal standard area must be within ± 40% of the CCV's internal standard area for all blanks, QC samples, and samples. If the area count is below the lower limit as established by the CCV then the IS is deemed to have failed acceptance criteria and the steps in sections 8.6.2 and 8.6.2.1 are followed.

8.6.2 If the Internal Standards for the blank do not pass the acceptance criteria, the system is inspected and the blank re-analyzed. Analyses do not continue until the blank meets the Internal Standard Criteria. If the Internal Standard acceptance criterion is not met during sample analysis, the sample must be reanalyzed. If the ISs are within limits in the reanalysis, the second analysis will be reported.

8.7 Surrogates

8.7.1 The acceptance limits for Surrogate recoveries are 70 - 130%. If the Surrogate recoveries for the QC samples (i.e. CCV, LCS or blank) are outside of these limits, the system is inspected and the QC sample re-analyzed. Analysis does not continue until the QC sample meets the Surrogate recovery criteria. If the recovery limits are not met for sample analysis, the sample must be re-analyzed unless obvious matrix interference is documented. If the surrogate recoveries are within limits in the re-analysis, the second analysis will be reported. If the limits are still not met, then the data from the first analysis is reported with a narrative indicating the acceptance criteria for surrogate recoveries were exceeded. Additionally, the system must be shown to be in control by the analysis of a blank or sample with acceptable surrogate recoveries. Upon request, the data from the matrix effect confirmation analysis is provided to the client.

8.7.2 Some projects require the Surrogates to be evaluated using control limits that are derived from historical data.

8.8 Laboratory Duplicates

8.8.1 Every daily analytical batch must include an LCS and an LCSD to evaluate instrument precision. The acceptance criteria for the relative percent difference

(RPD) between the LCS and LCSD analyses should meet ≤25%. Sample analysis can continue so long as no more than 5% of the compound list exceeds the 25% RPD criterion. No compound should exceed 40% RPD. Any compound exceeding 25% RPD is narrated in the lab report. If a compound exceeds 40%RPD, the LCS is analyzed a 3rd time. If the limit is exceeded again, the system is evaluated. The evaluation includes verification of LCS canister pressure and instrument flow rates.

8.8.2 A duplicate sample analysis is performed only when specifically required by the Project Profile and/or Project Requirement Table associated with the workorder. The Relative Percent Difference (RPD) between the two analyses must be ≤25% for all compounds detected at greater than 5 times the reporting limit (Limit of Quantitation). If this limit is exceeded, the sample is re-analyzed a third time, or analyzed on a different analytical system. If the limit is exceeded again, the cause is investigated and the system brought back to working order. If no problem is found on the system, the data is flagged to note the non-conforming event.

8.8.2.1 When three analyses do not result in acceptable precision (≤ 25% RPD for all compounds > 5X LOQ), the instrument shall be eliminated as a potential source of the failure. This may be accomplished by choosing another sample and performing a duplicate analysis to determine if precision is possible in that instance. To whatever extent is possible, the original analytical conditions should be duplicated as well. This would imply use of the same syringe if relevant etc. Details to ensure duplication of original analytical conditions are left to the judgment of a scientist, Lab Manager, or the QA Manager. Do not dispose of the sample until the issue is resolved.

8.9 Field QC samples

8.9.1 Neither TO-14A nor TO-15 describes field QC sample collection or acceptance criteria. However, clients may collect Field Blanks, Trip Blanks, and Field Duplicates. While there is no acceptance criteria established, analysts must monitor performance and note anomalies. For example, a positive result in the Field or Trip Blank requires closer inspection to ensure the anomaly wasn't incurred during sample handling and loading. Inspection may include verification of the canister analyzed and verification that the analytical system was clean.

8.9.2 Likewise, a field duplicate sample which shows an inconsistent chromatographic pattern as compared to the paired sample or concentrations differing by more than 40%RPD for many of the detections requires further investigation at the time of sample loading. Verification of the canisters identification and load volume and comparison of the FID screens are all appropriate to verify results.

8.9.3 Notate on the data checklist the anomaly and the items verified. If an anomaly was uncovered, then the field QC should be reanalyzed. If there are no findings then an SDR is generated informing the appropriate project manager following a

note on the Data Review Checklist indicating the non conformance. Narrate the presence of a detection in the Trip Blank and Field Blank.

9.0 CALCULATIONS

9.1 Response Factor:

$$\textit{Relative Response Factor (RRF)} = \frac{\text{Area of Compound}}{\text{Area of Int. Standard}} \text{ X } \frac{\text{Conc. IS (ppbv)}}{\text{Conc. of Compound (ppbv)}}$$

9.2 Sample Results:

$$\textit{Results Calculation (ppbv on-column)} = \frac{\text{Area of Compound in Sample}}{\text{Area of Int. Standard in Sample}} \text{ X } \frac{\text{Conc. IS (ppbv)}}{\text{ICAL RRF}}$$

ppbv in sample = ppbv on-column X Dilution Factor

Dilution Factor = Pressurization Factor* x Analytical Dilution Factor

Analytical Dilution Factor = $\frac{\text{Max load volume of unit (ml)}}{\text{Sample volume loaded (ml)}}$ x (Off-line dilution factor)

** The pressurization factor is determined by the lab measured canister receipt vacuum and final pressure.*

10.0 SAMPLE ANALYSIS

10.1 Analytical Batch

The analytical batch is defined as all samples, up to 20, analyzed within 24 hours on one instrument. All QC and samples must be processed with the method associated with the daily analytical batch.

*Samples per batch are reportable analytical runs excluding QC samples, dilutions, duplicate analysis and confirmation analysis.

10.3 Analytical Sequence

Initial 24-hour period	Subsequent 24-hour period
BFB Tune Check	BFB Tune Check
Initial Calibration	CCV
LCS/LCSD	LCS/LCSD
Laboratory Blank	Laboratory Blank
Samples (up to 20)	Samples (up to 20)

The "Subsequent 24-hour" sequence is followed each 24-hour period (every 12 hours when specified by the project) during which samples are analyzed until the system fails quality control acceptance limits.

Additional QC samples may be required by the program or project to be analyzed within the analytical batch. These details are included in the project PRT.

10.5 Validation of Reporting Limit (Limit of Quantitation)

10.5.1 Method Detection Limit (MDL) studies are analyzed following procedures as described in 40 CFR Pt. 136 App. B and Eurofins Air Toxics SOP #39. The reporting limit (Limit of Quantitation) must be greater than the MDL before sample analysis can occur. If this is not achieved, corrective action, including raising the reporting limit is taken prior to continuing with sample analysis. See Appendix A for the TO-14A/TO-15 Reporting Limits. In general, the reporting limit (Limit of Quantitation) is typically up to 10 times greater than the MDL but may be up to 50 times greater. If the reporting limit exceeds this range, the Laboratory Manager and/or the QA Department will evaluate the RL to determine the reason and its approval.

10.5.2 The MDL verification sample must be analyzed on a quarterly basis. Refer to SOP #39 MDL Procedure for the acceptance criterion.

10.5.3 A Method Detection Limit study will be performed for non-standard compounds only when prior arrangements with the client have been made are documented in the Project Requirement Table. The Reporting Limit must be greater than the MDL before sample analysis can occur.

10.6 Analytical Procedures

The sample containers are connected to the inlet line of the TO-15 concentrator. Following the work instructions for the specific system, the sample connection is checked for leaks, and the sample is loaded onto the system. During the load step, a 1 mL gas sample loop filled with IS/S is swept onto the sorbent trap.

10.7 Sample Dilutions

10.7.1 To obtain analyte concentrations within the calibrated range of the detectors and to prevent contamination of the system, samples may be screened prior to analysis on a GC/MS or GC/FID. The VOC concentrations are used to determine the volume of sample loaded for TO-14A/TO-15 SIM analysis.

10.7.2 An undiluted analysis involves loading the maximum load volume for the unit generally 250mL for the manual systems or 400mL for the autosampler systems. The Dilution Factor is obtained by dividing the full load volume by the sample volume loaded. All samples submitted for TO-15 SIM are screened prior to analysis. If samples contain high concentrations of target and/or non-target VOCs, samples may be analyzed by an alternative TO-15 method (i.e. Standard or 5&20) with a higher dynamic calibration range.

If this is not possible, an increased dilution factor will be incurred. As well, if full scan instruments are not available, samples that contain over approximately 20,000 ppbv of target analytes are diluted by adding a measured aliquot of the sample to a Tedlar bag that has a metered volume of Nitrogen as the diluent gas. For this alternative dilution technique, up to 250 mL or 400mL of the diluted sample is loaded. The final dilution factor includes canister pressurization dilution as well.

10.8 Compound Identification

10.8.1 There are three criteria that must be satisfied to justify positive identification of a given compound.

1. The retention time must match the daily standard within a factor of less than 0.1 minutes for all characteristic ions (quantitation and qualifier ions) present in the spectrum. Shifts in target compound retention times that exceed this limit must be accompanied by a corresponding shift in retention time for the corresponding Internal Standard.
2. The peak shape should be Gaussian for all monitored ions of interest with the exception of those at or near the reporting limit.
3. The mass ion fragmentation pattern should match the reference spectrum unless obvious interference is noted. There are two tools in the Target data processing software to determine a match – the compound's spectral pattern (relative ion intensities) and the characteristic ion peak area ratios. In general, the visual assessment of the spectral pattern is sufficient to proceed with the identification; however, the ion area ratios can provide quantitative confirmation when interference is not present and qualifier ions are properly integrated.

10.8.2 Because only a limited number of mass ions are monitored in a given retention time window in the SIM mode, it may be challenging to determine the presence of an interfering peak. If the unit is operating in synchronous SIM/Scan (TO-15 Hi/Lo), the chemist can evaluate the scan file to determine whether an interfering peak is eluting at a similar retention time and affecting the quantitation and/or qualifier ion.

Background subtraction can be used by the laboratory staff as a data evaluation tool to remove interfering ion masses from co-eluting compounds that may be masking the target compound. If background subtraction is needed in order to make a decision regarding the presence of a target compound then the subtraction should be included in the data package. The background subtraction of the spectra should be within ± 20 scans of the target peak. If the decision can be made without the use of background subtraction then it is not required to be included in the data package.

10.8.3 If the above criteria are met then the compound has been positively identified. However, matrix interference may make it impossible to satisfy one or all of the

above conditions. In these instances the chemist must evaluate the available data and determine whether the compound should be positively identified on the basis of the chemist's "best judgment". Advanced data analysis tools are available in the data processing software to aid in this evaluation as needed.

10.9 Quantitative Analysis

10.9.1 Quantitation is based on the integrated abundance of the primary quantitation ion for each analyte. If the response for any primary quantitation ion exceeds the Initial Calibration range of the GC/MS system, the sample is diluted and re-analyzed, excluding detections of the compounds noted in Section 10.10. If the response for any primary quantitation ion results in a value that rounds to the equivalent of the upper calibration range when expressing the result with 2 significant figures, re-analysis is not necessary.

10.9.2 When interference with the primary quantitation ion occurs, the result is flagged with a "M" indicating matrix and a probable high bias. This is also noted in the laboratory narrative included in the report.

10.10 Detections Outside of the Calibrated Range

10.10.1 Per client request only, compounds detected between current instrument MDL and the Limit of Quantitation are reported and given "J" flags (Estimated Concentration).

10.10.2 Unless specifically directed otherwise by a client, detections of the following non-critical risk driver compounds: Acetone, 2-Butanone, 2,2,4-Trimethylpentane, Hexane and Heptane above the high level of the curve are reported with an "E" flag and do not result in further dilution.

10.11 Manual Integrations

At times performing manual integrations on Initial Calibrations standards, QC, Laboratory Blanks and samples may be necessary. To accurately document ion area ratios on the Target report page, if manual peak integration for quantitation ion is needed in the ICAL or QC samples, then the qualifier ion peak shape is evaluated as well and properly integrated if needed.

11.0 CORRECTIVE ACTION PROCEDURES

A request for corrective action (CAR) is initiated any time either the EATL SOPs or client-prescribed QC protocol are not followed, or in any other instance that sample results are adversely affected. The corrective action procedure is documented in EATL SOP #61.

12.0 DATA REVIEW

12.1 Analytical Data Review

As the analytical sequence is analyzed throughout the day, the data is reviewed by the analyst or scientist using the following steps:

12.1.1 Check for any project-specific requirements.

12.1.2 Verify holding time.

12.1.3 Verify the BFB tune check, CCV, LCS, LCSD

12.1.4 Verify that the method blank has no hits above the reporting limit.

12.1.5 Verify sample results:

a) Verify the retention time.

b) Verify that correct amount of sample was analyzed.

c) Verify the automated peak integration.

d) Verify that result concentrations are within linear range of calibration curve (upper 60% for dilutions).

12.1.6 Initial and date raw data and/or logbook entry to indicate that the data is acceptable.

12.1.7 Apply appropriate data flags.

12.1.8 Describe unusual events on Data Review Checklist.

12.1.9 Verify results of the data validation report from Lumen, make corrections to the raw data as needed to remove errors identified by Lumen.

Notes:

- *A secondary review of the analytical runs is required when the analyst or scientist is not signed off on the analysis.*
- *Preparation and review of laboratory narratives are carried out.*

12.2 Write-up and Final Report Review

The analyst or scientist performing the data write-up and final client report reduces the data by reviewing the Target data files. The peaks in each sample are reviewed for the correct integration and identification. The criteria for compound identification are described in section 10.8. The data is evaluated for the project required sublist only.

When the sample Target review is complete, the sample files are transferred to the Atlas database. The client report is compiled in the Atlas workorder editor. Check the following when compiling the report:

- Prepare and review narrative, detailing QC non-compliance as needed.
- Evaluate data package from a data user perspective. Does the data make sense?

After the Atlas report is complete, LUMEN, a rules-based data validation tool, is used to verify QC criteria, hold time, data qualifier flags, manual integration documentation, tune clock time, retention time, and appropriate ICAL. Review all errors, and correct or insure discrepancies are addressed on the Data Review sheet or explained in the Lab Narrative if data quality is impacted.

When complete sign and date the 'Write-up' field on the Data Review Checklist and email the report to the client.

12.3 Technical Data Review

The Scientist or designated personnel performs a Technical Data Review on 100% reports if the write-up review analyst or scientist is not signed off for the method. This review follows all the steps mentioned in the Analytical/Write-Up Data Review (see Section 12.1).

12.4 Report Review

The Report Review represents a 3rd level of review which is required for DoD and client specific projects only. Refer to the Data Review Checklist for the specific items requiring review.

12.5 QA Data Review

A thorough QA data review is performed by the QA Department on final data packages requesting 100% review. The QA review entails verification that project and QC requirements are met. Failure to meet QC and/or project requirements results in a Corrective Action Request (CAR) and documentation. Dilution factors, analyte retention times, peak integration areas, concentration calculations, unit conversions, and reporting limits are also checked. Field and Trip Blanks are checked and trends are observed.

13.0 INSTRUMENT MAINTENANCE

13.1 Instruments are monitored on a daily basis by the bench analyst for any potential failure. The analysis of blanks and control standards at the start of the day and as analysis continues helps to provide real time feedback to the analyst on the condition of the instruments. Routine maintenance includes: mass spectrometers, sample introduction system and gas chromatograph.

13.1.1 The bench analyst will document any routine or major non-routine maintenance in the bound instrument logbook assigned to each instrument. The date of the maintenance, what work was performed and analyst initials are included.

13.2 Mass Spectrometers

- Daily check of vacuum ion gauge (Increase in ion count indicates a potential leak)
- Daily (every 24 hours) tune check with BFB
- Cleaning of ion source on as needed basis
- The pump oil level and quality is visually checked every month and at the time of source cleaning to ensure proper vacuum pump function, and oil is changed as needed.
- A sensitivity check must follow routine maintenance to ensure that a standard representing the low point concentration of the curve meets criteria.

13.3 Sample Introduction System

- To ensure a clean sample introduction system, if necessary, the lines and trap are "steam-cleaned" by analyzing a humidified system blank. This takes place every day or as needed based on the compound list following standards (i.e., CCV, LCS) analysis. Humidified system blanks are also analyzed after saturation-level detections in samples.

13.4 Gas Chromatograph

Routine maintenance includes the following:

13.4.1 As needed, clip 3 feet off the front end of the capillary column, and if necessary, the backend as well.

13.4.2 Replace the injection port liner as needed.

13.4.3 Visually inspect the septum on the valve syringe injection port and replace as needed.

13.4.4 The column is replaced when chromatograph peak shape or resolution degrades. Similarly, if the column bleed profile rises with age then the column needs replacing.

14.0 DELIVERABLES

Data reporting packages are prepared as described in SOP #78 – Generation of Eurofins Air Toxics Data Deliverables, Electronic Conversion, and Archival.

15.0 REFERENCES

EPA Method TO-14A

Compendium of Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Second Edition, January 1999. *EPA/625/R-96/010b*

EPA Method TO-15

Compendium of Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Second Edition, January 1999. *EPA/625/R-96/010b*

SW-846 Method 8000B

Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final Update III, Revision 1, December 1996.

Volatile Organic Analysis of Ambient Air in Canisters - *Draft Method*

USEPA Contract Laboratory Program, Revision VCAA01.0, December 1991

Eurofins Air Toxics Laboratory Quality Assurance Manual (LQAM)

Definitions and Terms, Appendix A.

http://www.air-dispersion.com/formulas.html. Author Milton R. Beychok (accessed October 29, 2009).

List of Appendices

Appendix A. Compound List with Reporting Limits and Quantitation Ions

Table A-1 Reporting Limits and QC Acceptance Criteria for Standard TO-14A/15 SIM List

Table A-2 Example Reporting Limits and Quantitation Ions for Non-standard TO-14A/15 SIM List

Appendix B. Summary of Calibration and QC Procedures for Modified Methods TO-14A/TO-15 SIM

Appendix C. BFB Tune Criteria and Check Mix

Appendix A

Appendix A

Table A-1. Reporting Limits and QC Acceptance Criteria for Standard TO-14A/15 SIM List

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Dichlorodifluoromethane (Fr12)	0.020	≤30%	70 - 130	70 - 130	± 25
Freon 114	0.020	≤30%	70 - 130	70 - 130	± 25
Chloromethane	0. 50	≤30%	70 - 130	70 - 130	± 25
Vinyl Chloride	0.010	≤30%	70 - 130	70 - 130	± 25
Chloroethane	0.050	≤30%	70 - 130	70 - 130	± 25
Freon 11	0.020	≤30%	70 - 130	70 - 130	± 25
Freon 113	0.020	≤30%	70 - 130	70 - 130	± 25
1,1-Dichloroethene	0.010	≤30%	70 - 130	70 - 130	± 25
Trans-1,2-Dichloroethene	0.100	≤30%	70 - 130	70 - 130	± 25
Methyl tert-Butyl Ether	0.100	≤30%	70 - 130	70 - 130	± 25
1,1-Dichloroethane	0.020	≤30%	70 - 130	70 - 130	± 25
cis-1,2-Dichloroethene	0.020	≤30%	70 - 130	70 - 130	± 25
Chloroform	0.020	≤30%	70 - 130	70 - 130	± 25
1,1,1-Trichloroethane	0.020	≤30%	70 - 130	70 - 130	± 25
Carbon Tetrachloride	0.020	≤40%	60 - 140	60 - 140	± 25
Benzene	0.050	≤30%	70 - 130	70 - 130	± 25
1,2-Dichloroethane	0.020	≤30%	70 - 130	70 - 130	± 25
Trichloroethene	0.020	≤30%	70 - 130	70 - 130	± 25
Toluene	0.050	≤30%	70 - 130	70 - 130	± 25
1,1,2-Trichloroethane	0.020	≤30%	70 - 130	70 - 130	± 25
Tetrachloroethene	0.020	≤30%	70 - 130	70 - 130	± 25
1,2-Dibromoethane	0.020	≤30%	70 - 130	70 - 130	± 25
Ethyl Benzene	0.020	≤30%	70 - 130	70 - 130	± 25
m,p-Xylene	0.040	≤30%	70 - 130	70 - 130	± 25
o-Xylene	0.020	≤30%	70 - 130	70 - 130	± 25
1,1,2,2-Tetrachloroethane	0.020	≤30%	70 - 130	70 - 130	± 25
1,4-Dichlorobenzene	0.020	≤30%	70 - 130	70 - 130	± 25

Naphthalene	0.050	≤40%	60 - 140	60 - 140	± 25

Appendix A (continued)

Table A-2. Reporting Limits and QC Acceptance Criteria for TO-14A/15 Extended SIM List

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
1,2,3-Trichloropropane	0.040	≤30%	70 - 130	70 - 130	± 25
1,2,4-Trichlorobenzene	0.050	≤30%	70 - 130	70 - 130	± 25
1,2,4-Trimethylbenzene	0. 040	≤30%	70 - 130	70 - 130	± 25
1,2-Dichlorobenzene	0.050	≤30%	70 - 130	70 - 130	± 25
1,2-Dichloropropane	0.040	≤30%	70 - 130	70 - 130	± 25
1,3,5-Trimethylbenzene	0.040	≤30%	70 - 130	70 - 130	± 25
1,3-Butadiene	0.200	≤30%	70 - 130	70 - 130	± 25
1,3-Dichlorobenzene	0.100	≤30%	70 - 130	70 - 130	± 25
1,4-Dioxane	0.100	≤30%	70 - 130	70 - 130	± 25
2-Butanone	0.200	≤30%	70 - 130	70 - 130	± 25
2-Hexanone	0.200	≤30%	70 - 130	70 - 130	± 25
2-Propanol	0.040	≤30%	70 - 130	70 - 130	± 25
4-Ethyltoluene	0.040	≤30%	70 - 130	70 - 130	± 25
4-Methyl-2-Pentanone	0.040	≤30%	70 - 130	70 - 130	± 25
Acetone	0.200	≤30%	70 - 130	70 - 130	± 25
Acetonitrile	0.200	≤30%	70 - 130	70 - 130	± 25
Acrolein	0.200	≤30%	70 - 130	70 - 130	± 25
Acrylonitrile	0.010	≤30%	70 - 130	70 - 130	± 25
alpha-Chlorotoluene	0.100	≤30%	70 - 130	70 - 130	± 25
Bromodichloromethane	0.012	≤30%	70 - 130	70 - 130	± 25
Bromoform	0.040	≤30%	70 - 130	70 - 130	± 25
Bromomethane	0.040	≤30%	70 - 130	70 - 130	± 25
Chlorobenzene	0.020	≤30%	70 - 130	70 - 130	± 25
cis-1,3-Dichloropropene	0.020	≤30%	70 - 130	70 - 130	± 25
Cyclohexane	0.040	≤30%	70 - 130	70 - 130	± 25
Dibromochloromethane	0.010	≤30%	70 - 130	70 - 130	± 25
Ethyl Acetate	0.040	≤30%	70 - 130	70 - 130	± 25

Heptane	0.040	≤30%	70 - 130	70 - 130	± 25
Hexachlorobutadiene	0.020	≤30%	70 - 130	70 - 130	± 25
Hexane	0.040	≤30%	70 - 130	70 - 130	± 25
Methylene Chloride	0.200	≤30%	70 - 130	70 - 130	± 25
Propylene	0.200	≤30%	70 - 130	70 - 130	± 25
Styrene	0.030	≤30%	70 - 130	70 - 130	± 25
Tetrahydrofuran	0.040	≤30%	70 - 130	70 - 130	± 25
trans-1,3-Dichloropropene	0.020	≤30%	70 - 130	70 - 130	± 25
Vinyl Acetate	0.040	≤30%	70 - 130	70 - 130	± 25

Appendix B

Summary of Calibration and QC Procedures for Modified Methods TO-14A/TO-15 SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours.	TO-15 Ion Abundance criteria	Correct problem then repeat tune.
Multi-Point Calibration (minimum of 5 points)	Prior to sample analysis.	≤30% for standard compounds with 2 compounds allowed out to ≤ 40% RSD.	Correct problem then repeat Initial Calibration Curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each initial calibration curve, and daily, prior to sample analysis.	Recoveries for 85% of Standard compounds must be 70-130%. No recovery may be <50%. ICV evaluated on a full list basis at time of calibration. Project may require use of in-house statistical or DoD QSM specified control limits for LCS	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) for Non-Standard Compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60-140%. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Continuing Calibration Verification (CCV)	At the start of each day after the BFB Tune check.	≤30%D ≤40%D Carbon Tetrachloride and Naphthalene	Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than two compounds from the standard list recover outside of 70-130%, corrective action will be taken. If any compound exceeds 60-140%, samples are not analyzed unless data meets project needs. Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV for Non-Standard Compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60-140%. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit (Tables A-2, A-3).	Inspect the system and Re-analyze the blank. B-flag data for common contaminants.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±40% of the area counts of the daily CCV internal standards.	**For blanks:** inspect the system and reanalyze the blank. **For samples:** re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.
Surrogates	As each standard, blank, and sample is being loaded.	70 - 130%. If specified by project, in-house generated control limits may be used.	**For blanks:** inspect the system and reanalyze the blank. **For samples:** re-analyze the sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %Rs are out-of-limits a second time, report data from first analysis and narrate.
Laboratory Duplicates - Laboratory Control Spike Duplicate (LCSD)	One per analytical batch.	RPD ≤25%.	Narrate exceedances. If more than 5% of compound list outside criteria or if compound is >40%RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

Appendix C

BFB Tune Check Criteria

4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

m/e	Ion Abundance Criteria
50	8.0 to 40.0% of mass 95
75	30.0 to 66.0% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	<2.0% of mass 174
174	50.0 to 120.0% of mass 95
175	4.0 to 9.0% of mass 174
176	93.0% to 101.0% of mass 174
177	5.0 to 9.0% of mass 176

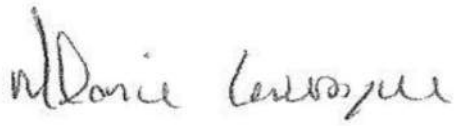
Document Owner/Laboratory Director:

Date: 04/30/19

Technical Director:

Date: 04-29-19

Quality Assurance:



Date: 05/01/19

Eurofins Air Toxics, LLC
STANDARD OPERATING PROCEDURE

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN SUMMA™ POLISHED CANISTERS BY GC/MS LOW LEVEL

MODIFIED EPA METHODS TO-14A/TO-15

SOP # 83

CONFIDENTIAL

1.0 SCOPE AND APPLICATION

The procedures in this SOP describe the use of Modified EPA Methods TO-14A and TO-15 to determine the concentration of volatile organic compounds in ambient air using an evacuated specially treated stainless steel canister. Details of the analytical procedures and the required QC protocols are provided. A list of target compounds can be found in *Appendix A*.

2.0 METHOD SUMMARY

2.1 Description

EPA Methods TO-14A and TO-15 describe techniques for the analysis of airborne VOCs collected as whole air samples in evacuated, specially treated stainless steel canisters. An aliquot of up to 400 mL of air is withdrawn from the canister using a mass flow controller. This volume is loaded onto a hydrophobic multibed sorbent trap to remove water and carbon dioxide and to concentrate the vapor sample. The focused sample is then flash heated to sweep adsorbed VOCs onto a GC/MS for separation and detection. Compounds are detected using a mass spectrometer operating in the full scan mode.

2.2 Deviations

EPA Methods TO-14A and TO-15 were written for ambient air applications targeting VOC concentrations at and above 0.5 ppbv. To provide VOC measurements below 0.5 ppbv, several modifications were implemented to accommodate a 5-fold reduction in reporting limits.

Modifications to EPA Methods TO-14A and TO-15 used to carry out the analyses of air samples are summarized in Tables 1 and 2.

Table 1. Summary of Method TO-14A Modifications

Requirement	TO-14A	EATL Modifications
Sample Drying System	Nafion Dryer	Multibed hydrophobic sorbent
Blank acceptance criteria	< 0.2 ppbv	< RL
BFB ion abundance criteria	Ion abundance criteria listed in Table 4 of TO-14A	Follow abundance criteria listed in TO-15.
BFB absolute abundance criteria	Within 10% when comparing to the previous daily BFB.	CCV internal standard area counts are compared to ICAL, Corrective action when recovery is less than 60%.
Blanks and standards	Zero Air	UHP Nitrogen provides a higher purity gas matrix than zero

Requirement	TO-14A	EATL Modifications
		air for trace level measurements.
Initial Calibration	≤ 30% RSD for listed 39 VOCs	≤ 30% RSD with 4 compounds allowed out to ≤ 40%.

Table 2. Summary of Method TO-15 Modifications

Requirement	TO-15	EATL Modifications
Initial Calibration	≤ 30% RSD with 2 compounds allowed out to < 40% RSD.	≤ 30% RSD with 4 compounds allowed out to ≤ 40%.
Blanks and standards	Zero Air	UHP Nitrogen provides a higher purity gas matrix than zero air.

3.0 HEALTH AND SAFETY

3.1 Normal laboratory safety precautions must be used when handling samples, preparing standards from neat materials, and analyzing samples. Appropriate eye wear, gloves, and lab coat must be worn when handling any chemical used in this method. All manipulation of standards, solvents, and solutions should be done with the utmost care in the hood. SDS for each chemical should be consulted for specific dangers and precautions.

3.2 Personnel must handle high pressure cylinders safely. This includes transport of cylinders fully secured on a cart. During storage, the cylinders must be secured at all times with a chain. When installing a pressure regulator, stand to the side of the cylinder.

3.3 Care must also be taken when handling syringes to ensure that a needle stick does not occur. ***All personnel installing or performing maintenance on a capillary column must wear eye protection***.

3.4 For information regarding pollution prevention and waste disposal, see Eurofins Air Toxics SOP #24: Storage and Disposal of Hazardous Wastes.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 Sample Containers

An air sample is collected in an evacuated stainless steel Summa™ / canister or Tedlar bag. Tedlar bags are not included as acceptable media in either Compendium Method TO-14A or TO-15, but are accepted for analysis per client request. Upon receipt, the canisters will be approximately in the range of 2.5 – 10" Hg vacuum.

4.2 Sample Handling

Prior to analysis, the canister is pressurized per project specific requirements.

Samples pressurized with Helium cannot be analyzed by low level analysis. The flow controllers used in the laboratory are calibrated for Nitrogen only. Helium pressurized samples typically have a higher flow rate than Nitrogen pressurized samples. Please see SOP 6 or 91 for analysis of Helium pressurized samples.

4.3 Sample Storage

Samples are stored in the sample cage in the main laboratory. Analysis must occur within 30 days for canisters. Some projects may require a 14 day hold for canisters. The project profile must be reviewed prior to analysis to verify holding time requirements.

4.4 Holding time in the case of Tedlar bags is limited to a maximum of 3 days as losses of VOCs are observed specifically for compounds with low vapor pressure. Holding time may be extended to a maximum of 30 days by transferring the sample from a Tedlar bag to a clean, evacuated Summa™ canister prior to the expiration of the 3 day hold. The transfer must be documented using the Tedlar bag transfer form and documented in the case narrative as per Eurofins Air Toxics SOP #69.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Interferences to this method generally include high concentrations of water. Very high levels of moisture in the samples may cause low internal standard and surrogate responses which will interfere with accurate compound quantification. In these situations, dry lab blanks may be required between samples to maintain internal standard recoveries. In extreme cases, sample dilution may be required.

5.2 When a sample has high levels of acidic gases such as HCl and SO_2 and/or high levels of non-target compounds, the analyst may have to dilute the sample more than the target compound level requires. This ensures that internal standard recoveries meet QC requirements and/or that the system is not contaminated from carryover or damaged by high levels of acidic gases.

5.3 See Eurofins Air Toxics WI38_83 C2 interface and WI38_83 PE interface (MSD-E) work instructions for examples when to red tag canisters due to high level concentrations.

5.4 Tedlar bags are not appropriate sample collection media for low vapor pressure compounds such as naphthalene due to severe adsorption and subsequent losses on the Tedlar surface. A sample discrepancy report (SDR) must be submitted when naphthalene measurements are requested from a Tedlar bag. All naphthalene results are flagged as estimated values.

6.0 EQUIPMENT/APPARATUS

6.1 List of Equipment

- TO-15 Air Concentrator Systems (
- Column: RTX-624 Capillary Column or equivalent column
- Gas Chromatograph: Agilent 6890 equipped with Electronic Pressure Control and a split/splitless injection port, and a cryo valve
- Mass Spectrometer: Agilent 5973 or equivalent
- Agilent Chemstation Software for Data Acquisition
- Thru-Put Target Software (UNIX Operating System) for Data Analysis
- NIST/NBS54.1K or NIST08.1 Library Search Software
- Certified NIST Traceable VOC cylinder blends
- High Purity Neat Chemicals
- Tedlar Bags (various sizes) SKC
- Heating Tapes- Various Lengths
- Power Controllers (Cole Palmer)
- Liquid Nitrogen certified VOC free by TO-15 analysis
- Ultra High Purity Helium (Local Supplier)
- Commercially purchased dilution system to blend working standards from stock cylinder standards utilizing mass flow controllers

6.2 Operating Parameters for GC/MS

Since analytical equipment varies in configuration and as a function of intended specific target analytes, operating parameters are optimized on an instrument-by-instrument basis. Instrument specific operating parameters are recorded in the respective Instrument Maintenance Logbook at each work station.

Note: Only trained laboratory staff are authorized to vary the operating parameters under special circumstances. Any time a change is made, it is documented in the instrument maintenance logbook.

7.0 STANDARD PREPARATION

The formula used to calculate the volume required for the preparation of primary and second source standards for gas blends is the following:

C1*V1 = C2*V2,

Where, C1 is the concentration of the gas blend, V1 is the volume of standard to be used in the preparation, C2 is the desired concentration for the resulting standard and V2 is the final volume (at pressure*) of the newly blended standard.

*At 15 psi the final volume for a 6L canister is 12L and for a 1L canister it is 2L

(volume reflects a twofold dilution as a function of pressurization).

7.1 Stock Standards

7.1.1 The TO-15 analytes are purchased from a qualified vendor at a concentration of 1-5 ppmv in a high pressure cylinder blend. The standard expires according to the manufacturer's expiration date on the certificate of analysis.

7.1.2 Analytes not present in the commercially available blends are purchased in neat form from a commercial vendor. These compounds are then blended into the gas phase. Once the neat compound exceeds the manufacturer's expiration date, these standards must be purity-checked by the laboratory annually.

7.2 Working Standards

7.2.1 Starting with a clean evacuated canister, a volume of 50 microliters of purged deionized water is injected to humidify the standard prior to preparation.

7.2.2 Precision Diluter

The Precision Diluter is used to prepare primary and secondary standards for VOC analysis. The diluter prepares dilution of stock standards by monitoring small pressure changes digitally. There are a total of four positions on the diluter that can accommodate high pressure standard cylinders. There is an additional inlet to allow for secondary dilutions of working standards.

7.2.3 Working standards prepared from source standards originating from neat or liquid mixes are prepared by injecting appropriate amounts of standard into an evacuated canister that contains 50 µL of water. These working standards typically contain 'non-routine' compounds which are blended in canisters separate from the routine TO-15 VOC working calibration standard.

7.2.4 All working standards are pressurized to 15 psi with Nitrogen. Concentrations of working standard canisters are typically prepared at 0.05 ppbv (if needed), 2.0 ppbv, and 50 ppbv. (Some compounds may be blended at concentrations 10x lower, e.g. Naphthalene).

7.2.5 Working standards can be re-pressurized with Nitrogen when pressure becomes near ambient. This re-pressurization step is recorded in the standard preparation logbook. The concentration of the working standard is then adjusted to reflect the addition of Nitrogen.

Working standards may be used for up to 3 months from the date of preparation. The TO-15 working standard may not be used past the expiration date of the NIST-traceable high pressure cylinder. *Note: Tedlar bags are used for the static dilution medium due to their inherent inertness to polar analytes vs. glass dilution jars. Standards made using a Tedlar bag should not, however, be stored in the Tedlar bags beyond one day. Fresh calibration standards are prepared and then transferred to Summa™ canisters for storage. The standards prepared from neat materials are stable in Summa™ canisters for 6 months.*

7.3 BFB Check Mix

A certified 25 ng/µL 4-Bromofluorobenzene (BFB) standard purchased from a commercial vendor is utilized for systems allowing for direct injection into the GC injection port. Alternatively, a 1 to 10ppmv NIST-traceable cylinder containing BFB may be used to make a diluted working standard for TO-15 systems without access to the GC injection port.

7.4 Internal Standard/Surrogate (IS/S) Mix

The IS/S blend is an internal reference material which may be prepared from a Certified Working Class standard purchased from a qualified vendor. The standard is in the range of 10 ppmv and expires based on the manufacturer's date on the certificate of analysis. A working standard is prepared by taking a known aliquot of the standard and diluting it to achieve a concentration ranging from 1.25ppmv to 2.0ppmv. As an alternative, the IS/S mixture may be prepared from neat chemicals.

8.0 CALIBRATION AND QUALITY CONTROL PROCEDURES

The following sections outline the laboratory's routine calibration and quality control procedures. Specific programs such as DoD QSM and projects may require additional QC samples and/or tighter QC criteria.

8.1 Tuning Criteria

8.1.1 At the start of every 24 hours , a tune check with 4-Bromofluorobenzene (BFB).

8.1.2 The relative abundances of selected ions are tabulated and reported as outlined in *Appendix B*. Analysis cannot proceed unless all criteria of the tune check are met. The acceptance criteria are based on TO-15 which are wider than tuning criteria outlined in TO-14A.

8.2 Initial Calibration Procedures

Initial Calibration of the GC/MS is achieved via the internal standard technique. The concentrations used for standard analysis typically range from 0.1 to 40 ppbv. Other levels may be added to the calibration per specific client/project request.

8.2.1 Standard Compound criteria

Initial Calibration is performed using a minimum of five levels for standard TO14A/TO-15 compounds. The low-level standard must be less than or equal to the reporting limit (Limit of Quantitation) and must be verified on a quarterly basis.

The percent relative standard deviations (%RSD) for all standard compounds must be ≤ 30%, with four exceptions not to exceed ≤ 40%.

Non-Standard Compound criteria

Compounds that are not included in Table A-1 are defined as non-standard compounds. Examples of Non-Standard TO-14A/TO-15 compounds are listed in Table A-1b. A one, three or five point calibration may be performed for non-standard compounds with documented client approval. The requirements for TPH are described in SOP #111. The lowest level standard must be less than or equal to the reporting limit (Limit of Quantitation). In general, LOQ (Limit of Quantitation) evaluations are not maintained for non-standard compounds which are not part of DoD accreditation.

Initial calibration criterion for non-standard compounds is ≤ 40% RSD subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon Initial Calibration requirements prior to sample analysis as more stringent criteria than ≤ 40% RSD may be required. If linearity requirements are not met a new Initial Calibration Curve is performed.

8.2.2 After an Initial Calibration has been evaluated and meets laboratory criteria, the midpoint for standard compounds is copied by adding an extension onto the file identifier, and then it is re-quantified as a Continuing Calibration Verification. All calibration points are then re-quantified and the Internal Standard area counts and Retention Time Windows for each file are compared to that of the midpoint. Internal Standards in each calibration point compared in this manner must meet laboratory criteria (area count ± 40% and Retention Time ± 0.33 minutes compared to the CCV). If %D criterion for the CCV (see Section 8.4) is met, analysis of the Initial Calibration Verification Standard may proceed. On days when an Initial Calibration is not performed, the Internal Standard area counts and Retention Time Windows in all subsequent samples and

QC must meet laboratory criteria (area count ± 40% and Retention Time ± 0.33 minutes compared to the CCV analyzed at the beginning of the analytical sequence).

8.2.3 The multipoint calibration is constructed by loading varying amounts of the combined calibration blend (Section 7.2) onto the canister interface. The maximum load volume for the air interface system (e.g. 250 mL) defines the dilution factor.

8.2.4 The average relative response factor (RRF) from the Initial Calibration Curve is used to quantitate results.

8.2.5 Initial calibration curves are generated in accordance to appropriate laboratory practices:

8.2.5.1 The lowest or highest calibration levels of an analyte may be dropped from the curve to achieve linearity. This will affect the analyte's reporting limit and/or calibration range. The minimum number of calibration levels must still be used for the curve.

8.2.5.2 An additional mid-range calibration level may be added to achieve linearity.

8.2.5.3 Alternate calibration levels may be used to meet client requirements.

8.2.5.4 It is not acceptable to drop a calibration level that is found somewhere in the middle of the curve to achieve linearity (i.e. dropping the 50ppbv or the 25ppbv calibration level). The calibration level that is in question may be re-analyzed and re-quanted into the curve.

8.2.6 All Initial Calibrations needing re-analysis of a calibration level require explanation in the Initial Calibration Case Narrative Template. Only one calibration level is allowed for re-analysis per Initial Calibration Curve due to anomalous unacceptable linearity for compound(s). A bad load or unopened can does not count towards a re-analysis. If more than one standard was used for curving an instrument (i.e. 2 or more), and it is obvious that the RF is not linear relative to the other points of the curve, then all points ran from that standards must be re-analyzed. The reason for the reanalysis must be narrated and included with the ICAL raw data.

8.2.7 The reporting limit (Limit of Quantitation – LOQ) must be verified quarterly on each instrument that performs the methods the lab is accredited for by DoD-ELAP (Refer to DoD scope at

O:\QA\Certifications). The LOQ is verified by evaluating the point of the initial calibration that corresponds to the LOQ. The concentration recovered is used to calculate the precision and bias of each compound. A minimum of three points is required to perform the calculations. If there are insufficient points, a primary source standard is analyzed at the LOQ. Precision and bias is determined by calculating the % relative standard deviation (% RSD) and the % bias of the mean concentration recovered. The acceptance criterion for the LOQ verification is ≤30% RSD and ≤50% bias for each compound. If more than 10% of the compound list exceeds the acceptance criteria, the instrument and standard used will evaluated to determine the source of the error. Quarterly LOQ verifications are evaluated by the QA Department.

8.2.8 All current Initial Calibrations and Method Detection Limit (MDL) studies are kept in a folder near the instrument.

8.2.9 The indicated flow on the instrument must be measured and its units must be calibrated with a NIST flow meter to provide a true indication of the actual flow through the unit before each time an Initial Calibration is performed and/or on a quarterly basis, whichever is more frequent.

8.3 Initial Calibration Verification (ICV)

8.3.1 Standard Compound criteria

An independently prepared (i.e., same vendor, different lot number, or second vendor) standard containing all target compounds is analyzed after each Initial Calibration Curve, to verify that the standards are correct and the calibration is accurate. The acceptance criterion for the ICV recoveries are as follows: For the compounds listed in Table A-1, recoveries for 85% of the compounds must meet the listed acceptance criteria with no recoveries <50%. For the compounds listed in Table A-1, recoveries for 90% of the compounds must be ± 30%. For the compounds listed in Table A-1b recoveries for 80% of the compounds must be ± 40%.

8.3.2 Recoveries of any compound that is found to exceed these criteria, but are within 50-150%, are narrated on the ICAL Narrative sheet that is attached to the ICAL packet. However, exceeding the acceptance criterion of 150% recovery permits for a reanalysis of an ICV.

8.3.3 Recovery of any compound in the ICV that is < 50% of the expected value will result in standard re-preparation, system maintenance if needed and re-calibration, or sample analysis on a different instrument. Recovery of any compound that is ≥ 150% of expected value, analysis may continue with manager approval after evaluation of whether the data meets client project needs. However, the system and/or standard preparation should be

evaluated. If the problem is determined to be systematic (i.e. occurs on more than three consecutive days), corrective action outlined above should be conducted to resolve the issue.

8.3.4 Non-Standard Compound criteria

An Initial Calibration Verification will be performed for non-standard compounds only when prior arrangements with the client have been made and are documented in the Project Requirement Table. Initial calibration verification criterion for these analytes is 60 – 140 % Recovery and is subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify the agreed upon ICV requirements prior to sample analysis as more stringent criteria than 60 – 140 % may be required.

8.3.5 See SOP #111 for requirements for TPH.

8.4 Continuing Calibration Verification (CCV)

8.4.1 A Continuing Calibration Verification (CCV) is performed at the start of each day after analysis of the BFB Tune Check. This is an analysis of the primary source at a concentration between the low point and the midpoint of the initial calibration.

8.4.2 The acceptance criteria for the percent Difference (%D) between the daily CCV response and average response from the Calibration Curve is as follows:

8.4.2.1 The acceptance criteria for the percent Difference (%D) between the daily CCV response and average response from the Calibration Curve is as follows: All standard CCV compounds must be ≤ 30% D. Any compounds exceeding this criterion will be flagged and associated data likewise flagged and narrated. If more than four compounds from the standard list recover outside of 70-130%, corrective action will be taken. Corrective action may include instrument maintenance, re-calibration, and/or re-preparation of the calibration standards (See 8.4.4). If any compound recovery exceeds 60-140%, samples are not analyzed unless data meets project needs. The QA Manager or Lab Manager may approve exceedance of a compound under special circumstances after reviewing the impact to the data quality. Regardless, associated data will be flagged and narrated.

8.4.2.2 If a list of ≤ 40 target compounds is requested, no more than 10%

of the compounds may be outside 70-130% criteria. For example, if a client is requesting 6 compounds then 0 compounds are allowed out; or if 15 compounds are requested then only 1 compound may be outside acceptance criteria.

8.4.3 Non-Standard Compound criteria

Compounds that are not included in Tables A-1 are defined as non-standard compounds. Examples of Non-Standard TO-14A/TO-15 compounds are listed in Table A-1b. CCV criterion for these analytes is ≤ 40 %D subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon CCV requirements prior to sample analysis as more stringent criteria than ≤ 40 %D may be required.

8.4.4 If the CCV fails to meet the performance criteria, the CCV is re-analyzed and/or another standard is analyzed. If the CCV fails again, system maintenance should be performed and the test repeated. If the system still fails the calibration check, a new Calibration Curve is performed. If the CCV passes following maintenance to the instrument then the LCS must also pass before samples can be analyzed.

8.5 Laboratory Control Spike (LCS)

8.5.1 A Laboratory Control Spike is analyzed daily prior to sample analysis. Recovery limits are listed in Table A-1. Recoveries for 85% of standard compounds must be 70-130% with no recovery at <50Alternatively, projects may require in-house generated or DoD QSM specified control limits.

8.5.2 If the stated criteria are not met, the system is checked and the same standard or different standards are re-analyzed. In the event that the criteria cannot be met for the full TO-14A/TO-15 list of compounds, a review of the data will be done by a QA Manager or Lab Manager and may approve the exceedances of a compound under special circumstances after reviewing the impact to the data quality. If approved, the data will be flagged and narrated.

8.5.3 LCS criterion for Non-Standard analytes is 60 – 140 % recovery subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon LCS requirements because in some cases, a commitment has been made to criteria more stringent than 60 – 140%.

8.5.4 Recovery of any compound in the LCS that is ≤ 50% of the expected value will result in either re-calibration, or analysis on a different instrument. Recovery of any compound that is ≥ 150% of expected value, analysis may continue; however the system and/or standard preparation should be

evaluated. If the problem is determined to be systematic (i.e. occurs on more than three consecutive days and/or on multiple instruments), the instrument must be evaluated and recalibrated if needed.

8.5.5 Some projects require the LCS to be evaluated using control limits that are derived from historical data. Recovery of any compound in the LCS that is outside of the historically derived control limits but within the default limits of 70-130%, analysis may continue and the excursions flagged and narrated.

8.5.6 An LCS is analyzed in duplicate (LCSD) daily prior to sample analysis. Refer to section 8.9.1 for the RPD acceptance criteria between the LCS and LCSD.

8.6 Internal Standards

8.6.1 One mL of the IS blend is injected into the canister interface as each standard, blank, and sample is being loaded.

- Bromochloromethane
- Chlorobenzene-d_5
- 1,4-Difluorobenzene

8.6.2 Internal Standards' retention times for the blanks, QC samples and samples must be within ± 0.33 minutes of the retention times in the Continuing Calibration Check. In addition, the IS area must be within ± 40% of the CCV's IS area for the blanks, QC samples and samples. If the area count is below the lower limit as established by the CCV then the IS is deemed to have failed acceptance criteria and the steps in sections 8.6.2.1 and 8.6.2.2 are followed.

8.6.2.1 If the IS's for the blank do not pass the acceptance criteria, the system is inspected and the blank re-analyzed. Analysis is discontinued until the blank meets the IS criteria.

8.6.2.2 If the IS's in a sample do not pass the acceptance criteria, the sample must be re-analyzed. If the IS's are within limits in the re-analysis, the second analysis will be reported. If the IS's are out-of-limits a second time, then the sample will be diluted to get the IS's within limits.

8.7 Surrogates

One mL of the Surrogate blend is injected into the canister interface as each standard, blank, and sample is being loaded. The acceptance limits for Surrogate recoveries are 70 to 130%. Concentrations of Surrogates are equivalent to internal standard concentrations:

- 1,2-Dichloroethane-d4
- Toluene-d8
- 4-Bromofluorobenzene

8.7.1 If the Surrogate recoveries for the QC samples (i.e. CCV, LCS, or blank) do not pass the acceptance criteria, the system is inspected and the QC sample is re-analyzed. Analysis is discontinued until the QC sample meets the Surrogate recovery criteria.

8.7.2 If the surrogate recoveries for a sample are outside of these limits, the sample is re-analyzed unless obvious matrix interference is documented. If the Surrogate recoveries are within limits in the re-analysis, the second analysis will be reported. If the Surrogate recoveries are out-of-limits a second time, the data from the first analysis will be reported with a narrative indicating the acceptance criteria for Surrogate recoveries were exceeded. Additionally, the system must be shown to be in control by the analysis of a blank or sample with acceptable surrogate recoveries.

8.7.3 Some projects require the Surrogates to be evaluated using control limits that are derived from historical data.

8.8 Laboratory Blank

8.8.1 A humidified Nitrogen Laboratory Blank is analyzed upon completion of all required QC including calibration standards at the beginning of each day and at least once in every 24 hour shift. A Lab Blank is also analyzed in the event saturation-level concentrations are incurred to demonstrate that contamination does not exist in the chromatographic system. Each system has been evaluated for carryover, and a specific sample load volume or on-column concentration has been determined as the trigger for a Lab Blank. The acceptance criterion for Laboratory Blanks is a result less than the laboratory reporting limit (Limit of Quantitation) (see *Appendix A*).

8.8.1.1 In the event that the Laboratory Blank is contaminated by target compounds, tentatively identified compounds (TICs), non-Methane organic hydrocarbons (NMOC), or petroleum hydrocarbons (TPH), the following protocol should be observed. Initially, the Laboratory Blank should be re-analyzed to eliminate the possibility of an anomalous indication. If re-analysis is acceptable, analysis of client samples may continue.

8.8.1.2 Client samples that have analyte sublists that do not include the compound(s) in question should be substituted for full list samples. As these samples are analyzed, the operator should monitor the results to determine if the contamination has been removed from

the system. In the event non-target contamination is present in the Laboratory Blank, analysis of samples not requiring TIC's, NMOC, or TPH calculations can continue.

8.8.1.3 If there are no samples in-house that meet the criteria described in 8.8.1.2, the nature of the contamination should be evaluated. Samples that have been analyzed that cannot be re-analyzed will be flagged to note the non-conforming result.

8.8.1.4 Methylene Chloride, Acetone, Acetonitrile, Tetrahydrofuran, Ethanol, Bromomethane and 2-Butanone are acknowledged as common laboratory contaminants. Presence of these compounds at a concentration of < 5X the reporting limit is acceptable as long as the associated samples are not being analyzed for these analytes only. The associated samples will be flagged. If the contamination persists for more than two analytical batches, the issue should be escalated for evaluation in order to identify and remove the source of contamination.

8.8.1.5 If other target compounds are present in the Laboratory, operations management will determine the impact on the usability of the data based on project requirements. If analysis proceeds, the associated data will be flagged to indicate detection above the RL in the Laboratory Blank.

8.8.1.6 If any Lab Blank contains either target or non-target hits at or above the reporting limit that are questionable, the blank should be re-analyzed to confirm the contamination. Manual integration of blanks is not permitted if the manual integration causes the analyte of interest to be below the reporting limit, except in the case of baseline contribution to the total ion current of a Surrogate (usually Bromofluorobenzene) when calculating NMOC/TPH-G concentrations and baseline contribution such as described in SOP #52 for Manual Integrations by GC/MS analyses. The Laboratory Manager, QA Manager or designated personnel must sign all manual integrations on Lab Blanks.

8.8.1.7 If the analysis request is for the full list of compounds laboratory blanks are not needed to continue running samples for the following compounds at the given concentration; Acetone at or above 60 ppbv, 2-Propanol at or above 80 ppbv, and Ethanol at or above 60 ppbv. If a sample contains any or all of these compounds above these concentrations, analysis of samples may continue. If the sample(s) following the high level analysis contain these compounds below 5x the reporting limit then a narration must be

used describing the potential for high bias of these results. If the result for these compounds in the subsequent sample(s) is greater than 5X the reporting limit then no narration is necessary.
If 10 or fewer analytes are requested and the target list contains these compounds, blanks must be used to demonstrate cleanliness of the system if concentrations are above the values listed previously.

8.8.2 Some projects require specific acceptance criteria for Method Blank as follow:

No analytes detected at ≥ ½ the RL. For common laboratory contaminants, no analytes detected ≥ the RL.

If an analyte in the laboratory blank fails these criteria, a different Laboratory Blank canister is analyzed to rule out canister contamination. If the analyte in the Laboratory Blank still fails these criteria, the data is flagged with the appropriate data qualifying code (B) and the non-conformance is narrated unless, the analyte resulted in a non-detect in the samples.

8.8.3 In general, manual integration of target peaks in lab blanks is not permitted if the manual integration causes the analyte of interest to be below the reporting limit (Limit of Quantitation), except in the case of baseline contribution to the total ion current of a TIC or surrogate (usually Bromofluorobenzene) when calculating NMOC/TPH-G concentrations. However, there may be times that integration of compounds in the Lab Blank is necessary to accurately represent the data. All manual integrations on Lab Blanks must be signed by either a qualified Lab Manager or the QA Manager. Refer to SOP #52 "Manual Peak Integration and Background Subtraction for GC/MS Analyses".

8.9 Laboratory Duplicates

8.9.1 Every daily analytical batch must include an LCS and an LCSD to evaluate instrument precision. The acceptance criteria for the relative percent difference (RPD) between the LCS and LCSD analyses should meet ≤25%. Sample analysis can continue as long as no more than 5% of the compound list (3 VOCs for a 62 compound list) exceeds the 25% RPD criterion. No compound should exceed 40%RPD. Any compound exceeding 25% RPD is narrated in the lab report. If a compound exceeds 40%RPD, the LCS is analyzed a 3rd time. If the limit is exceeded again, the system is evaluated. This evaluation includes verification of LCS canister pressure and instrument flow rates.

8.9.2 A duplicate sample analysis is performed only when specifically required by the Project Profile and/or Project Requirement Table associated with the workorder. The Relative Percent Difference (RPD) between the two analyses must be ≤25% for all compounds detected at greater than 5 times the reporting limit (Limit of Quantitation). If this limit is exceeded, the sample is re-analyzed a third time, or analyzed on a different analytical system. If the limit is exceeded again, the cause is investigated and the system brought back to working order. If no problem is found on the system, the data is flagged to note the non-conforming event.

8.9.2.1 When three analyses do not result in acceptable precision (≤ 25% RPD for all compounds > 5X LOQ), the instrument shall be eliminated as a potential source of the failure. This may be accomplished by choosing another sample and performing a duplicate analysis to determine if precision is possible in that instance. To whatever extent is possible, the original analytical conditions should be duplicated as well. This would imply use of the same syringe if relevant etc. Details to ensure duplication of original analytical conditions are left to the judgment of a scientist, Lab Manager, or the QA Manager. Do not dispose of the sample until the issue is resolved.

8.10 Field QC Samples

8.10.1 Neither TO-14A nor TO-15 describes field QC sample collection or acceptance criteria. However, clients may collect Field Blanks, Trip Blanks, and Field Duplicates. While there is no acceptance criteria established, analysts must monitor performance and note anomalies. For example, a positive result in the Field or Trip Blank requires closer inspection to ensure the anomaly wasn't incurred during sample handling and loading. Inspection may include verification of the canister analyzed and verification that the analytical system was clean.

8.10.2 Likewise, a field duplicate sample which shows an inconsistent chromatographic pattern as compared to the paired sample or concentrations differing by more than 40%RPD for many of the detections requires further investigation at the time of sample loading. Verification of the canisters identification and load volume and comparison of the FID screens are all appropriate to verify results.

8.10.3 Notate on the data checklist the anomaly and the items verified. If an anomaly was uncovered, then the field QC should be reanalyzed. If there are no findings then an SDR is generated informing the appropriate project manager following a note on the Data Review Checklist indicating the non

conformance. Narrate the presence of a detection in the Trip Blank and Field Blank.

9.0 CALCULATIONS

9.1 Response Factor

$$\text{Relative Response Factor (RRF)} = \frac{\text{Area of Compound}}{\text{Area of Int. Standard}} \times \frac{\text{Conc. Int. Standard (ppbv)}}{\text{Conc. of Compound (ppbv)}}$$

9.2 Sample Results

$$\underset{\text{(ppbv on-column)}}{\text{Results Calculation}} = \frac{\text{Area of Compound in Sample}}{\text{Area of Int. Standard in Sample}} \times \frac{\text{Conc. Int. Standard (ppbv)}}{\text{ICAL RRF*}}$$

** The average RRF from the Initial Calibration Curve is used to quantitate results.*

ppbv in sample = ppbv on-column X Dilution Factor

Dilution Factor = Pressurization Factor* x Analytical Dilution Factor

$$\text{Analytical Dilution Factor} = \frac{\text{Max load volume of unit (ml)}}{\text{Sample volume loaded (ml)}} \times \text{(Off-line dilution factor)}$$

** The pressurization factor is determined by the lab measured canister receipt vacuum and final pressure.*

9.3 Total Petroleum Hydrocarbon (TPH) and Non-Methane Organic Compound (NMOC) calculations by GC/MS

The calculations performed for TPH and NMOC as well as additional TPH characterization including the hydrocarbon fractionation is beyond the scope of this SOP and is detailed in SOP #111.

10.0 SAMPLE ANALYSIS

10.1 Analytical Batch

The analytical batch is defined as all samples* up to 20 analyzed within 24 hours on one instrument. All QC and samples must be processed with the method associated with the daily analytical batch.

* Samples per batch are reportable analytical runs excluding QC samples, duplicate analysis and confirmation analysis.

10.2 Analytical Sequence

Initial 24-hour period:	***Subsequent 24-hour period:***
BFB Tune Check	BFB Tune Check
Initial Calibration	CCV
LCS/LCSD	LCS/LCSD
Laboratory Blank	Laboratory Blank
Samples (up to 20)	Samples (up to 20)

The "Subsequent 24-hour" sequence is followed every 24 hour period during which samples are analyzed until the system fails quality control acceptance limits.

Additional QC samples may be required by the program or project to be analyzed within the analytical batch. These details are included in the project PRT.

10.3 Validation of Reporting Limit (Limit of Quantitation)

10.3.1 Method Detection Limit (MDL) studies are analyzed for all standard TO-14A/TO-15 compounds following procedures as described in 40 CFR Pt. 136 App. B and Eurofins Air Toxics SOP #39.

10.3.2 The reporting limit (Limit of Quantitation) must be greater than the MDL before sample analysis can occur. If this is not achieved, corrective action, including raising the reporting limit is taken prior to continuing with sample analysis. See Appendix A for the TO-14A/TO-15 Reporting Limits.

10.3.3 In general, the reporting limit (Limit of Quantitation) is typically up to 10 times greater than the MDL but may be up to 50 times greater. If the reporting limit (Limit of Quantitation) exceeds this range, the Laboratory Manager and/or the QA Department will evaluate the RL to determine the reason and its approval.

10.3.4 The MDL verification sample must be analyzed on a quarterly basis. Refer to SOP #39 and WI39 MDL Procedure for the acceptance criterion.

10.3.5 A Method Detection Limit study will be performed for non-standard compounds only when prior arrangements with the client have been made are documented in the Project Requirement Table. The reporting limit must be greater than the MDL before sample analysis can occur.

10.4 Quantitative Analysis

10.4.1 Quantitation is based on the integrated abundance of the primary ion for each analyte. If the response for any primary quantitation ion exceeds the

Initial Calibration range of the GC/MS system, the sample is diluted and re-analyzed, excluding detections of the compounds noted in Section 10.7.2. If the response for any primary quantitation ion results in a value that rounds to the equivalent of the upper calibration range when expressing the result with 2 significant figures, re-analysis is not necessary.

10.4.2 When interference with the primary quantitation ion occurs, either the result is flagged with an "M" indicating matrix and a probable high bias or quantitation on the secondary ion is carried out after a new response factor (using the secondary ion) is generated from the initial calibration. Therefore, the same ion used to establish the response factor is used to quantify target analytes in the sample. This is noted in the laboratory narrative included in the report.

10.4.3 The criterion for using the secondary ion for quantitation is a difference in the reported result of 50% or more. Discussion with the project manager and/or QA Manager may determine that quantitation using the secondary ion is not necessary given the objectives of the project and flagging will suffice. In the case when the difference is less than 50% and/or there is interference with the primary and secondary ion, the possibility of high bias is notated on the Data Review Checklist and in the final report.

10.5 Detections Outside of the Calibrated Range

10.5.1 Compounds detected in between current instrument MDL values and the low point of the calibration curve are reported only by client request and are flagged to indicate that the concentration is estimated.

10.5.2 Unless specifically directed otherwise by a client, detections of the following non-critical risk driver compounds: Acetone, Cyclohexane, Ethanol, Heptane, Hexane, Methyl Ethyl Ketone (2-Butanone), 2-Propanol, Propylene, 2,2,4-Trimethylpentane and Tetrahydrofuran above the high level of the curve are reported with an "E" flag and do not result in further dilution.

10.6 Tentatively Identified Compounds (TICs)

10.6.1 By project request, based on the computer generated searches, the identification of the ten highest non-target unknown peaks is reported. The spectra of these peaks are searched against the NIST library of greater than 50,000 compounds.

10.6.2 The total ion current is used for quantitation and calculation of TIC results. A TIC is determined to be present when the quantitated concentration is

found to be greater than one tenth the concentration of the spiked Internal Standard.

10.6.3 The total ion current of the closest (by retention time, RT) non-interfered Internal Standard is used to calculate results (per SW-846 protocol). If all Internal Standards have interference, the Internal Standards in the Lab Blank are used to calculate results. A relative response factor of "1" is assumed. Match quality is useful in determining whether or not tentative identification should be reported, but is not the only criteria.

10.6.4 There are cases in which the NIST library match contains masses for a compound that are not within the conventional scanning range. In this instance match quality may appear to be poor when that is not the case. Eurofins Air Toxics protocol is to defer to the analyst's judgment and experience with regard to identification.

10.7 Compound Identification

10.7 There are three criteria that must be satisfied to justify positive identification of a given compound.

1. The retention time must match the daily standard within a factor of less than 0.1 minutes for all characteristic ions (quantitation and qualifier ions) present in the spectrum. Shifts in target compound retention times that exceed this limit must be accompanied by a corresponding shift in retention time for the corresponding Internal Standard.

2. The peak shape should be Gaussian for the compound's characteristic ions with the exception of those at or near the reporting limit.

3. The mass ion fragmentation pattern should match the reference spectrum unless obvious interference is noted. There are two tools in the Target data processing software to determine a match – the compound's spectral pattern (relative ion intensities) and the characteristic ion peak area ratios. In general, the visual assessment of the spectral pattern is sufficient to proceed with the identification; however, the ion area ratios can provide quantitative confirmation when interference is not present and qualifier ions are properly integrated.

10.7.2 To evaluate interferences and aid in comparison of the peak spectra to the reference spectral pattern, background subtraction can be used by the laboratory staff as a data evaluation tool to remove interfering ion masses from co-eluting compounds that may be masking the target compound. If

background subtraction is needed in order to make a decision regarding the presence of a target compound then the subtraction should be included in the data package. The background subtraction of the spectra should be within ± 20 scans of the target peak. If the decision can be made without the use of background subtraction (i.e. retention time, characteristic ion pattern or area ratio match, and Gaussian peak shape) then it is not required to be included in the data package. Some ions in the reference spectra are not within the scan range (35-350 amu) and as such are not considered to be relevant to a positive identification.

10.7.3 If the above criteria are met then the compound has been positively identified. However, matrix interference may make it impossible to satisfy one or all of the above conditions. In these instances the chemist must evaluate the available data and determine whether the compound should be positively identified on the basis of the chemist's "best judgment". Advanced data analysis tools are available in the data processing software to aid in this evaluation as needed.

10.8 Manual Integrations

At times performing manual integrations on Initial Calibrations standards, QC, Laboratory Blanks and samples may be necessary. To accurately document ion ratios on the Target report page, if manual peak integration for the quantitation ion is needed in the ICAL or QC samples, then the qualifier ion peak shape is evaluated as well and properly integrated if needed.

10.9 Analytical Procedures

The sample containers are connected to the inlet line of the TO-15 concentrator. Following the work instructions for the specific system, the sample connection is checked for leaks, and the sample is loaded onto the system. During the load step, a 1 mL gas sample loop filled with IS/S is swept onto the sorbent trap. If samples are pressurized with Helium, see work instructions for adjusting and verifying accurate mass flow controller operation.

10.10 Sample Dilutions

10.12.1 To obtain analyte concentrations within the calibrated range of the detectors and prevent contamination of the system, samples are typically screened prior to analysis on a GC/MS. Occasionally, a GC/FID unit may be used specifically when high petroleum concentrations are expected. Dilutions are determined based on estimated concentrations of the highest target and/or non-target compounds.

10.12.2 Following the screening process, if the sample or samples is deemed to require a dilution factor of more than 10, the sample(s) can be analyzed via TO-14A/TO-15 (5&20 ppbv) following SOP #91 with the following stipulations:

- 5&20 instrumentation must be validated via Initial Calibration, MDL and daily CCV/LCS/Lab Blank for all of the target compounds associated with the Project Profile and workorder in question.

10.12.3 Other necessary changes may be made on a project by project basis in order to make the transition from SOP #83 to SOP #91 analytically consistent.

10.12.4 An undiluted analysis involves loading the standard load volume as identified by the ICAL. In general this standard load volume is 250 mL for the LL analysis, or 400ml on the autosampler systems. The dilution factor is obtained by dividing the full load volume by the sample volume loaded. All samples submitted for TO-15 LL are screened prior to analysis. If samples contain high concentrations of target and/or non-target VOCs, samples may be analyzed by an alternative TO-15 method (i.e. standard or 5&20) with a higher dynamic calibration range.

10.12.5 When determining the load volume for the sample, both target and non-target compounds are evaluated by reviewing the screening data.

Second, the screening results should also be evaluated for non-target compounds since high concentrations can contaminate the loading interface and interfere with the analysis. Non-target compounds eluting early in the screen do not require as aggressive of a dilution since carryover and interference is expected to be minimal. Non-target compounds with a lower vapor pressure (eluting mid and late on the GC screening run) require dilution to minimize interference and carryover. In general, mid- and late-eluting non-target compounds require dilution when concentrations are above approximately 100 ppbv.

10.12.6 Dilution for the target and non-target compounds is required if screening data indicates that they may saturate the detector.

10.12.7 In the event the Project Profile requires that all analytical runs be reported, the analytical runs must be evaluated at the time of analysis to ensure that the RPD for compounds within linear range and greater than 5 times the RL do not exceed 25%. Re-analysis or system inspection/maintenance may be required to resolve any discrepancies.

11.0 CORRECTIVE ACTION PROCEDURES

A Request for Corrective Action (CAR) is initiated any time either the EATL SOPs or client-prescribed QC protocol are not followed, or in any other instance that sample results are adversely affected. The corrective action procedure is documented in EATL SOP #61.

12.0 DATA REVIEW

12.1 Analytical Data Review

As the analytical sequence is analyzed throughout the day, the data is reviewed by the analyst or scientist using the following steps:

- Check for any project-specific requirements.
- Verify holding time.
- Verify the BFB Tune check, CCV, LCS, LCSD.
- Verify that Method Blank has no hits above reporting limit (with the exceptions outlined in 8.7).
- Verify sample results:
  a. Verify the retention time.
  b. Verify that correct amount of sample was analyzed.
  c. Verify the automated peak integration.
  d. Verify that result concentrations are within linear range of Calibration Curve (generally in the upper 60% for dilutions).
- Initial and date raw data and /or logbook entry to indicate that the data is acceptable.
- Apply appropriate data flags.
- Describe unusual events on Data Review Checklist.
- Verify results of the data validation report from Lumen, make corrections to the raw data as needed to remove errors identified by Lumen.

Notes:

- *A secondary review of the analytical runs is required when the analyst or scientist is not signed off on the analysis.*
- *Preparation and review of Laboratory Narrative are carried out.*

12.2 Write-up and Final Report Review

The analyst or scientist performing the data write-up and final client report reduces the data by reviewing the Target data files. The peaks in each sample are reviewed for the correct integration and identification. The criteria for compound identification are described in section 10.9. The data is evaluated for the project required sublist only.

When the sample Target review is complete, the sample files are transferred to the Atlas database. Check the following when compiling the report:

- Prepare and review narrative, detailing QC non-compliance as needed.
- Evaluate data package from a data user perspective. Does the data make sense?

After the Atlas report is complete, LUMEN, a rules-based data validation tool, is used to verify QC criteria, hold time, data qualifier flags, manual integration documentation, tune clock time, retention time, and appropriate ICAL. Review all errors, and correct or insure discrepancies are addressed on the Data Review sheet or explained in the Lab Narrative if data quality is impacted.

When complete sign and date the 'Write-up' field on the Data Review Checklist and email the report to the client.

12.3 Technical Data Review

The Scientist or designated personnel performs a technical data review on 100% reports if the write-up review analyst or scientist is not signed-off for the method. This review follows all the steps mentioned in the Analytical data review (see Section 12.1).

12.4 Report Review

The Report Review represents a 3rd level of review which is required for DoD and client specific projects only. Refer to the Data Review Checklist for the specific items requiring review.

12.5 QA Data Review

A thorough QA data review is performed by the QA Department on final data packages requesting 100% review. The QA review entails verification that project and QC requirements are met. Failure to meet QC and/or project requirements results in a Corrective Action Request (CAR) and documentation. Dilution factors, analyte retention times, peak integration areas, concentration calculations, unit conversions, and reporting limits are also checked. Field and Trip Blanks are checked and trends are observed.

13.0 INSTRUMENT MAINTENANCE

13.1 Instruments are monitored on a daily basis by the bench analyst for any potential failure. The analysis of blanks and control standards at the start of the day and as analysis continues helps to provide real-time feedback to the analyst on the

condition of the instruments. Routine maintenance includes: mass spectrometers, sample introduction system and gas chromatograph.

13.1.1 The bench analyst will document any routine or major non-routine maintenance in the bound instrument logbook assigned to each instrument. The date of the maintenance, what work was performed and analyst initials are included.

13.2 Mass Spectrometers

- Periodic check of vacuum ion gauge (Increase in ion count indicates a potential leak)
- Daily (every 24 hours) tune check with BFB
- Cleaning of ion source on quarterly basis or as needed
- The pump oil level and quality is visually checked every month and at the time of source cleaning to ensure proper vacuum pump function, and oil is changed as needed.
- A sensitivity check must follow a routine maintenance to ensure that a standard representing the low point concentration of the curve meets criteria.

13.3 Sample Introduction System

13.3.1 To ensure a clean sample introduction system, if necessary, the lines and trap are "steam-cleaned" by analyzing a humidified system blank. This takes place every day or as needed based on the compound list following standard's (i.e., CCV, LCS) analysis. Humidified system blanks are also analyzed after saturation-level detections in samples.

13.4 Gas Chromatograph

Routine maintenance includes the following:

13.4.1 As needed, clip approximately 3 feet off the front end of the capillary column, and if necessary, the back end as well.

13.4.2 Replace the injection port liner as needed.

13.4.3 Visually inspect the septum on the valve syringe injection port and replace as needed.

13.4.4 The column is replaced when chromatography peak shape or resolution degrades. Similarly, if the column bleed profile rises with age then the column needs replacing.

14.0 DELIVERABLES

Data reporting packages are prepared as described in SOP #78 – Generation of Eurofins Air Toxics Data Deliverables, Electronic Conversion, and Archival.

15.0 REFERENCES

EPA Method TO-14A
Compendium of Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Second Edition, January 1999. *EPA/625/R-96/010b*

EPA Method TO-15
Compendium of Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Second Edition, January 1999. *EPA/625/R-96/010b*

SW-846 Method 8000B
Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final Update III, Revision 1, December 1996

Volatile Organic Analysis of Ambient Air in Canisters - *Draft Method* USEPA Contract Laboratory Program, Revision VCAA01.0, December 1991

Eurofins Air Toxics NELAP Quality Manual
Definitions and Terms, Appendix A.

http://www.air-dispersion.com/formulas.html. Author Milton R. Beychok (accessed October 29, 2009).

List of Appendices

Appendix A. Reporting and QC Limits

Table A-1. Method TO-14ATO-15 Standard Compounds

Table A-1b. Method TO-14A/TO-15 Example Non-Standard Compounds
Table A-2. Internal Standards
Table A-3. Surrogates
Table A-4. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15 for Volatile Organic Compounds

Appendix B. BFB Tune Criteria

Appendix A

Reporting and QC Limits

Table A-1. Method TO-14A/TO-15(Standard Compounds)

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS* (%R)	Precision Limits (%RPD)
1,1,2,2-Tetrachloroethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,1,2-Trichloroethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,1-Dichloroethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,1-Dichloroethene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,2,4-Trichlorobenzene	0.5	≤30%	70 - 130	70 - 130	≤ 25
1,2,4-Trimethylbenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,2-Dibromoethane (EDB)	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,2-Dichlorobenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,2-Dichloroethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,2-Dichloropropane	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,3,5-Trimethylbenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,3-Dichlorobenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,4-Dichlorobenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Benzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Bromomethane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Carbon Tetrachloride	0.1	≤30%	70 - 130	70 - 130	≤ 25
Chlorobenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Chloroethane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Chloroform	0.1	≤30%	70 - 130	70 - 130	≤ 25
Chloromethane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Chlorotoluene (Benzyl Chloride)	0.1	≤30%	70 - 130	70 - 130	≤ 25
cis-1,2-Dichloroethene	0.1	≤30%	70 - 130	70 - 130	≤ 25
cis-1,3-Dichloropropene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Dichloromethane (Methylene Chloride)	0.2	≤30%	70 - 130	70 - 130	≤ 25
Ethylbenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Freon 11 (Trichlorofluoromethane)	0.1	≤30%	70 - 130	70 - 130	≤ 25
Freon 113 (Trichlorotrifluoroethane)	0.1	≤30%	70 - 130	70 - 130	≤ 25
Freon 114	0.1	≤30%	70 - 130	70 - 130	≤ 25
Freon 12 (Dichlorodifluoromethane)	0.5	≤30%	70 - 130	70 - 130	≤ 25
Hexachlorobutadiene	0.5	≤30%	70 - 130	70 - 130	≤ 25
m,p-Xylene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Methyl Chloroform (1,1,1-Trichloroethane)	0.1	≤30%	70 - 130	70 - 130	≤ 25
o-Xylene	0.1	≤30%	70 - 130	70 - 130	≤ 25

Styrene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Tetrachloroethene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Toluene	0.1	≤30%	70 - 130	70 - 130	≤ 25
trans-1,3-Dichloropropene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Trichloroethene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Vinyl Chloride	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,3-Butadiene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,4-Dioxane	0.1	≤30%	70 - 130	70 - 130	≤ 25
2-Butanone (Methyl Ethyl Ketone)	0.5	≤30%	70 - 130	70 - 130	≤ 25
2-Hexanone	0.5	≤30%	70 - 130	70 - 130	≤ 25
4-Ethyltoluene	0.1	≤30%	70 - 130	70 - 130	≤ 25
4-Methyl-2-Pentanone (MIBK)	0.1	≤30%	70 - 130	70 - 130	≤ 25
Acetone	1.0	≤30%	70 - 130	70 - 130	≤ 25
Bromodichloromethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
Bromoform	0.1	≤30%	70 - 130	70 - 130	≤ 25
Carbon Disulfide	0.5	≤30%	70 - 130	70 - 130	≤ 25
Cumene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Cyclohexane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Dibromochloromethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
Ethanol	0.5	≤30%	70 - 130	70 - 130	≤ 25
Heptane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Hexane	0.5	≤30%	70 – 130	70 – 130	≤ 25
Isopropanol	0.5	≤30%	70 – 130	70 – 130	≤ 25
Methyl t-Butyl Ether (MTBE)	0.1	≤30%	70 – 130	70 – 130	≤ 25
Propylbenzene	0.1	≤30%	70 – 130	70 – 130	≤ 25
Tetrahydrofuran	0.5	≤30%	70 – 130	70 – 130	≤ 25
trans-1,2-Dichloroethene	0.1	≤30%	70 – 130	70 – 130	≤ 25
2,2,4-Trimethylpentane	0.5	≤30%	70 – 130	70 – 130	≤ 25
3-Chloroprene	0.5	≤30%	70 – 130	70 – 130	≤ 25

Appendix A (continued)

Table A-1b. Method TO-14A/TO-15 Example Non-Standard Compounds

Analyte	RL/LOQ (ppbv)	QA Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD
Acrolein	0.5	≤40	60 – 140	60 – 140	± 25
Butane	0.5	≤40	60 – 140	60 – 140	± 25
Ethyl tert-Butyl Ether	0.5	≤40	60 – 140	60 – 140	± 25
Isopentane	0.5	≤40	60 – 140	60 – 140	± 25
Isopropyl Ether	0.5	≤40	60 – 140	60 – 140	± 25
Methylcyclohexane	0.5	≤40	60 – 140	60 – 140	± 25
Naphthalene	0.5	≤40	60 – 140	60 – 140	± 25
Propylene	1.0	≤40	60 – 140	60 – 140	± 25
tert-Amyl Methyl Ether	0.5	≤40	60 – 140	60 – 140	± 25
Vinyl Acetate	0.5	≤40	60 – 140	60 – 140	± 25
tert-Butyl Alcohol	0.5	≤40	60 – 140	60 – 140	± 25
TPH (Gasoline)	10	1- Point Calibration	NA	ICV only: 60 – 140	± 25
NMOC (Hexane/Heptane)	2.0	1- Point Calibration	NA	NA	± 25

Table A-2. Internal Standards

Analyte	Recovery Limits (%R)
Bromochloromethane	60 – 140
1,4-Difluorobenzene	60 – 140
Chlorobenzene-d_5	60 – 140

Table A-3. Surrogates*

Analyte	Recovery Limits (%R)
1,2-Dichloroethane-d_4	70 – 130
Toluene-d_8	70 – 130
4-Bromofluorobenzene	70 – 130

* In-house generated control limits may be used per client's request.

Appendix A (continued)

Table A-4. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15 (VOC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours,	TO-15 ion abundance criteria	Correct problem then repeat tune.
Minimum 5-Point Initial Calibration (ICAL)	Prior to sample Analysis.	% RSD ≤ 30 with four compounds allowed out to ≤ 40% RSD.	Correct problem then repeat initial calibration curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each initial calibration curve, and daily prior to sample analysis.	Recoveries for 85% of Standard compounds must be 70-130%. No recovery may be <50%. ICV evaluated on a full list basis at time of calibration. * If specified by the project, in-house generated or DoD specified control limits may be used for the LCS.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) for Non-Standard Compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60-140%. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Continuing Calibration Verification (CCV) for Standard compounds	At the start of each day and 24-hour clock	70-130%.	Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than four compounds from the standard list recover outside of 70-130% or > 10% of VOCs if short list is used (40 compounds or less), corrective action will be taken. If any compound exceeds 60-140%, samples are not analyzed unless data meets project needs. Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV for Non-Standard Compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60-140%. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit (Tables A-1).	Inspect the system and Re-analyze the blank. B-flag data for common contaminants.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ± 40% of the area counts of the daily CCV internal standards.	**For blanks:** inspect the system and reanalyze the blank. **For samples:** re-analyze the sample unless obvious matrix interference is documented. If the Iss are within limits in the reanalysis, report the second analysis. If Iss are out-of-limits a second time, report data from first analysis and narrate.
Surrogates	As each standard, blank, and sample is being loaded.	70 – 130% R. * If specified by the project, in-house generated control limits may be used.	**For blanks:** inspect the system and reanalyze the blank **For samples:** reanalyze the sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %Rs are out-of-limits a second time, report data from first analysis and narrate.
Laboratory Duplicates – Laboratory Control Spike Duplicate (LCSD)	One per analytical batch.	RPD ≤ 25%.	Narrate exceedances. If more than 5% of compound list outside criteria or if compound is >40%RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

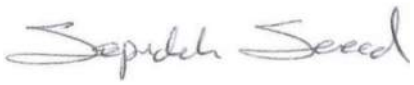
Appendix B

BFB Tune Criteria

4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

m/e	Ion Abundance Criteria
50	8.0 to 40.0% of mass 95
75	30.0 to 66.0% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	<2.0% of mass 174
174	50.0 to 120.0% of mass 95
175	4.0 to 9.0% of mass 174
176	93.0% to 101.0% of mass 174
177	5.0 to 9.0% of mass 176

Document Owner/Laboratory Director:



Date: -

Technical Director:

Date: -

Quality Assurance:

Date: 02/28/20

Eurofins Air Toxics, LLC

STANDARD OPERATING PROCEDURE

ANALYSIS OF VOCS BY GC/MS COLLECTED ON CHARCOAL-BASED PASSIVE SAMPLERS USING MODIFIED EPA TO-17

SOP #100

1.0 SCOPE AND APPLICATION

The procedures in this SOP describe the protocols for GC/MS determination of VOCs collected using charcoal-based passive samplers. Extraction procedures for the following samplers are provided: Radiello (RAD130), Waterloo Membrane Samplers (WMS), and SKC 575 and charcoal Ultra series samplers. These samplers can be used to measure vapor-phase VOCs in a variety of gaseous matrices including indoor air, outdoor air, workplace environments, soil gas, and emissions from materials.

2.0 METHOD SUMMARY

2.1 Description

VOCs in the sampling environment pass through the diffusive barrier or permeable membrane of the sampler at a known, controlled rate (defined as the sampling rate) and adsorb to the charcoal-based sorbent bed of the sampler. The sorbent is extracted using a volume of carbon disulfide, and the extract is directly injected into a gas chromatograph equipped with a mass spectrometer. The retention time and spectral pattern of a compound are compared with that of known standard. Concentrations of the analytes are calculated from the average relative response factors of calibration curves obtained from analysis of standard solutions. The results are reported in units of μg/sample or $\mu g/m^3$ if the sampling rate and duration are known.

2.2 Deviations

There are currently no EPA methods for the preparation and analysis of charcoal-based passive samplers for environmental monitoring of VOCs in air. The reference method used for this procedure is EPA TO-17, which describes the collection of VOCs in ambient air using sorbents and analysis by GC/MS. Because TO-17 describes active sample collection using a pump and thermal desorption as the preparation step, several modifications are required. Specifically, the extraction steps using carbon disulfide and internal standard addition are based on the recommended procedures published by Radiello (FSM). The modifications taken to EPA Method TO-17 are outlined in Table 1.

Table 1. Summary of Method TO-17 Modifications

Requirement	TO-17	EATL Modifications
Sample collection	Pump pulls measured air volume through sorbent tube	VOCs in air adsorbed onto sorbent bed passively through diffusion

Sample preparation	Thermal extraction	Solvent extraction
Sorbent tube conditioning	Condition newly packed tubes prior to use.	Charcoal-based sorbent is a single use media and conditioning is conducted by vendor.
Instrumentation	Thermal desorption system	Liquid injection system
Internal Standard	Gas-phase internal standard introduced on the tube or focusing trap during analysis	Liquid-phase internal standard introduced on the tube at the time of extraction
Media and sample storage	<4 deg C, 30 days	Media shelf life is determined by vendor; sample hold-time is 30 days for the RAD130 and WMS. Sample preservation requirements are storage in a cool, solvent-free refrigerator and optional use of ice during shipping.
Internal Standard Recovery	+/-40% of daily CCV area	-50% to +100% of daily CCV area

3.0 HEALTH AND SAFETY

Normal laboratory safety precautions must be used when handling samples, preparing standards from neat materials, and analyzing samples. Appropriate eye wear, gloves, and lab coat should be worn when handling any chemical used in this method. All manipulation of standards, solvents, and solutions should be done with the utmost care in the hood. The MSDS for each chemical should be consulted for specific dangers and precautions. Specifically, the extraction solvent, carbon disulfide, is flammable and extremely hazardous in case of skin contact, ingestion or inhalation. The MSDS of carbon disulfide is required reading prior to performing this procedure.

Care must also be taken when handling syringes to ensure that a needle stick does not occur. All personnel installing or performing maintenance on a capillary column must wear eye protection. For information regarding pollution prevention and waste disposal, see Eurofins Air Toxics SOP #24: Storage and Disposal of Hazardous Wastes.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Sorbent media is stored according to manufacturer recommendations. The media and sample storage requirements are summarized in Table 2.

Table 2. Storage Requirements

Sorbent	Media Shelf-life	Sample Hold-time	Storage	Shipping
RAD130	2 years – use expiration date provided by FSM	30 days	Cool (<10°C); solvent-free refrigerator	Expedited shipping recommended, use of ice optional
SKC 575 /Ultra Series	2 years- use expiration date provided by SKC	At least 2 weeks.	Room temperature acceptable, SKC recommends storing at 4 deg C for trace level analysis.	Expedited shipping recommended, use of ice optional
*WMS	6 months from certification date, then re-certify	30 days	Room Temperature (<25°C) in solvent-free environment	Standard shipping is acceptable

*The WMS samplers are prepared by SiREM

The vendors FSM (Radiello) and SKC (Ultra and 575) provide expiration dates on the packaging. SiREM provides samplers directly to the laboratory and the laboratory establishes media expiration date of 6 months after certifying the lot of prepared samplers.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

Interferences to this method generally include high background VOC contribution from carbon disulfide solvent and interference of the carbon disulfide (CS2) peak with closely eluting target compounds. It is extremely important to use high purity grade carbon disulfide. The presence of benzene in CS2 is common and care must be exercised to ensure concentrations are below the reporting limit. Each lot of carbon disulfide must be certified prior to use and also with each extraction batch. Additionally, target VOC peaks elute both prior to and after the CS2 peak. To protect the detector, the filament is turned off during the elution of CS2, limiting the analysis of VOCs eluting during or near the solvent delay.

Based on laboratory spiking studies and literature references, 1,1,2,2-Tetrachloroethane can degrade into Trichloroethene during storage on Anasorb 747 which is used in the WMS devices. Appropriate narratives and qualifiers are included in the method quotes and laboratory reports.

Whenever a high concentration sample is encountered (i.e. > 2000 ug/mL), the sample analyzed next must be re-analyzed to ensure no carry-over effects (unless the following sample is < RL for all target compounds).

6.0 MEDIA/EQUIPMENT/ REAGENTS

6.1 List of Media

- WMS samplers manufactured by SiREM: Three versions of the WMS samplers are available – standard, low-uptake, and thick membrane. The standard version is denoted on the sampler as #-AN-R-# and in Atlas as WMS-SE. The low-uptake version is notated on the sampler as #-AN-LU-# and in Atlas as WMS-LU.

  A third version of WMS may be employed and is denoted on the sampler as #-RTM-#. "RTM" stands for regular thick membrane and utilizes a membrane 10.2 times thicker than the normal 100 micron membrane.

- Radiello® Adsorbing Cartridge code 130

- SKC 575 or Ultra Series Badge

6.2 List of Equipment

- Agilent 7683B Series Injector and Autosampler Tray

- Agilent MSD 5975C operating using synchronous SIM/Scan

- RTX-624

- Ultra High Purity Helium (Local Supplier)

- Agilent 7890A Gas Chromatograph

- HP Chemstation and Target Thru-put system software.

- 10 μL, 25 μL, 100uL, 500 μL, and 1 to 5 mL cemented needle gastight syringes and microliter syringes (Hamilton or equivalent vendor) Hamilton syringes have an accuracy of +/- 1%.

- Glass vial with screw top cap: 2 mL , 4mL

- Wheaton Mini-nert Vial: 0.3 and 1.0 mL.

- Disposable pipettes

6.3 List of Reagents

- Certified Custom made VOC Mixes in CS2 qualified vendor
- Carbon Disulfide, Assay: GC; Purity: 99.99%; qualified vendor

7.0 STANDARDS PREPARATION

The standards are prepared by syringe dilution technique. The aliquot volume (V_2) of the stock standard is calculated using the following equation:

$$V_2 = (C_1 * V_1)/C_2$$

Solvent Volume = V_1-V_2

Where C_1 = Concentration of the diluted standard (ug/mL)

C_2 = Concentration of the stock standard (ug/mL)

V_1 = Final volume of the diluted standard (mL)

V_2 = Volume of the stock standard (mL)

Using a gastight/microliter syringe, add the calculated solvent volume to a labeled vial. The calculated stock standard volume (V_2) is then injected via syringe into the vial. The vial is sealed with light blue cap and inverted multiple times to mix completely. Most standards are prepared to a final volume of 2.0 mL using a 2 mL screw top vial equipped with a light blue color cap.

The syringe dilution technique has the following advantages over using volumetric flasks for the preparation of VOC mixtures:

- No transfer of the prepared standard from a volumetric flask to a storage vial is required which minimizes the potential loss of volatile compounds.
- Headspace in the preparation/storage vial is minimized which helps to keep volatile compounds in solution.

7.1 Stock Standards

7.1.1 A stock solution containing the majority of the target analytes is purchased from Absolute Standards or equivalent at concentration of 5000 ug/mL.

7.1.2 A stock solution containing target analytes that are not in the above mixed standard is prepared by aliquoting a calculated amount of each individual stock into a 2-mL vial using a calibrated syringe. This solution should yield 10,000 ug/mL in Carbon Disulfide for each

analyte. Standard vials are stored in freezers at <-10 °C. The stock standard expires one year after the preparation.

7.2 Working Standards

7.2.1 A minimum of five calibration level standards is prepared by aliquoting the appropriate amount of stock standard mixture and diluting with Carbon Disulfide to yield desired concentrations. Working standards are stable for 6 months. If standard degradation is observed (e.g. poor recoveries), new standards are prepared. The instrument is calibrated using a range of concentrations from 0.05 ug/mL to 50 ug/mL.

7.3 BFB Tuning Standard

7.3.1 25 µg/mL solution of EPA 8240B/8260 GC/MS Tuning Mix standard is purchased from Supelco or equivalent. This standard should be stored at < 4 °C and expires one year after opening, or manufacturer's expiration date, whichever comes first.

7.4 Internal Standard Solution (IS)

7.4.1 A custom made 2-Fluorotoluene standard in Carbon Disulfide may be purchased from a equivalent qualified vendor at a concentration of 125ug/mL. 4 uL is injected into CCV standard and into all ICAL levels. This standard is stored at < -10 °C and expires 1 year after opening the container, or manufacturer's expiration date, whichever comes first.

7.4.2 Alternately, a standard stock solution can be purchased from a qualified vendor.

7.5 Surrogate Solutions

7.5.1 Neat Toluene–d8 is purchased from a equivalent qualified vendor. A 10,000 ug/mL stock solution of Toluene-d8 is prepared from neat standard. This standard is stored at <-10 °C and expires 1 year after preparation or when the parent standard expires. Alternate concentrations of surrogate solution may be prepared. The specific mass of material, final volume of solvent, and resulting concentration should be documented in the standard preparation book.

7.5.2 Alternately, a standard stock solution can be purchased from a qualified vendor.

7.6 Internal Standard/Surrogate (IS/S) Mix

7.6.1 A mix of Internal standard and Surrogate standard in Carbon Disulfide at a concentration of 5000 ug/mL may be purchased froma qualified vendor. This mix is then diluted to prepare a working standard at a concentration of 125 ug/mL The IS/S mix (125ug/mL) is stored tightly sealed at < -10 °C and expires six months after preparation or when the parent standard expires.

7.6.2 Alternately, standard mixes may be purchased from a qualified vendor.

7.7 Second Source Standards

7.7.1 Extraction Spike Standard

A standard at 500ug/mL is prepared by aliquoting the appropriate amount of stock standard mixture and diluting with Carbon Disulfide. The second source stock standards are either from different manufacturer or the same manufacturer but prepared by a different chemist. This standard is stored tightly sealed at < -10 °C and expires six months after the preparation date. If standard degradation is observed prior to 6 months expiration or when the parent standard expires, (e.g. poor recoveries) new standards are prepared.

7.7.2 Independent Source for Calibration Verification

The standard solution has a concentration of 5.0 ug/mL. This standard is stored tightly sealed at < -10 °C and expires six months after the preparation date or when the parent standard expires.

8.0 CALIBRATION AND QUALITY CONTROL PROCEDURES

Additional Calibration and QC procedures may be required for specific projects and programs (e.g. DoD QSM). Refer to the appropriate Project Requirement Tables attached to the project profile for the specific acceptance criteria for initial calibration, frequency and acceptance criteria for CCVs, method blanks, LCS, etc.

8.1 Tuning Criteria

A daily (every 12 hours) tune check with 4-Bromofluorobenzene is achieved by directly injecting 2 µL of the BFB Check Standard into the GC in accordance with Method TO-17 tuning criteria. The relative abundance of selected ions is tabulated and reported *(see Appendix B)*. Analysis cannot proceed unless all criteria of the tune check are met.

8.2 Internal Standards (IS)

The Internal Standard used by EATL for this analysis is 2-Fluorotoluene. The base peak ion from the specific Internal Standard is used for quantitation. If interferences are noted, then a secondary ion may be selected.

8.2.1 Acceptance criteria for Internal Standards are as follows:

8.2.1.1 Continuing Calibration Verifications (CCVs): The area counts must be within 50 to 200% recovery and the retention times must be within 30 seconds of the mid-level standard from the most recent Initial Calibration.

8.2.1.2 Method Blanks and Samples and all non-CCV QC Checks: The Internal Standards' retention times must be within ±0.33 min (20 seconds) of the retention times in the Continuing Calibration Check, and the area counts within 50-200% of the IS area counts in the CCV.

8.2.1.3 The EICP area counts for the Internal Standard in the Continuing Verification (CCV) must be within -50% to +100% of the mid -level standard from the most recent Initial Calibration. The acceptance criterion is not subject to rounding. If the EICP area for the Internal Standard is not within –50% to +100% of that in CCV, sample must be re-aliquoted and re-analyzed. If the Internal Standard response is within limits in the re-analysis, the second analysis will be reported. If the re-analysis result is outside the limit a second time, the sample will be diluted until the internal standard meets the acceptance criteria. Because the IS is added at the time of extraction, the IS will be dilute along with the sample, see Section 14.1 for dilution protocols. The other option is to use secondary ion for quantitation, if there are no matrix interferences. The results from the last run will be reported with a narrative indicating the presence of matrix.

8.2.1.4 If the Internal Standards for the system blank do not pass the acceptance criteria, the system is inspected and the blank re-analyzed. Analyses are discontinued until the blank meets the Internal Standard criteria.

8.3 Calibration Procedures

8.3.1 Calibration of the GC/MS is achieved via the Internal Standard technique. Relative response factors (RRFs) are generated based on the ratio of the standard quantitation ion area over the Internal Standard quantitation ion area and the ratio of the Internal Standard concentration over the standard concentration. See Section 10 for the calculation of RRF.

8.3.1.1 The Initial Calibration Curve includes a minimum of five concentration levels for each analyte and the surrogate Toluene–d8. Calibration is achieved by injecting a fixed volume using the autosampler microsyringe for all calibration levels.

8.3.1.2 The percent relative standard deviations (%RSD) are listed in Table A-1. If these criteria are not met, a new Initial Calibration Curve must be performed.

8.3.1.3 After an Initial Calibration has been evaluated and meets laboratory criteria, the midpoint is copied by adding an extension onto the file identifier, and then it is requantified as a Continuing Calibration Verification. All calibration points are then requantified and Retention Time Windows for each file are compared to that of the midpoint. If %D criterion for the CCV (see Section 8.5.1) is met, analysis of the Initial Calibration Verification Standard may proceed. On days when an Initial Calibration is not performed, the Retention Time Windows in all subsequent samples and QC must meet laboratory criteria (compared to the CCV analyzed at the beginning of the analytical sequence).

8.3.2 The reporting limit (Limit of Quantitation – LOQ) must be verified quarterly on each instrument that performs the methods the lab is accredited for by DoD (Refer to DoD scope at O:\QA\Certifications). LOQ standard preparation follows the same extraction procedure as samples (See Sections 10.2 and 10.3). The LOQ is verified by evaluating the point of the initial calibration that corresponds to the LOQ. The concentration recovered is used to calculate the precision and bias of each compound. A minimum of three points is required to perform the calculations. If there are insufficient points, a primary source standard is analyzed at the LOQ. Precision and bias is determined by calculating the % relative standard deviation (% RSD) and the % bias of the mean concentration recovered. The acceptance criterion for the LOQ verification is ≤30% RSD and ≤50% bias for

each compound. If more than 10% of the compound list exceeds the acceptance criteria, the instrument and standard used will evaluated to determine the source of the error. Quarterly LOQ verifications are evaluated by the QA Department.

8.4 Initial Calibration Verification (ICV)

8.4.1 Following Initial Calibration and with each analytical batch, a 500ug/mL standard which contains the majority of target compounds is prepared from a separate source. This independent source standard is analyzed to verify accuracy of the primary source standards used for the Initial Calibration. The acceptance criteria for the ICV recoveries are listed in Table A-1. If the noted limits are exceeded, the problem is investigated and if warranted, the system is re-calibrated.

8.5 Continuing Calibration Verification (CCV)

8.5.1 A midlevel standard is analyzed at the start of every clock immediately after the tune check. The CCV level should be varied periodically with one standard analyzed above the mid-point of the ICAL and one point below the mid-point. See table A-1 for the CCV acceptance criteria.

8.5.2 *Software Verification:* A manual verification of the computer-generated results is performed on one randomly chosen Surrogate and one target compound for each CCV.

8.6 Laboratory Control Spike (LCS)

8.6.1 An LCS standard which contains the majority of target compounds is prepared at 500 ug/mL from a separate source. The LCS standard is spiked and extracted with each set of up to 20 extracted samples.

8.6.2 If the LCS exceeds the limits listed in Table A-1, the analyst will re-aliquot and re-analyze the extract. It is not possible to re-extract charcoal sorbents. If the LCS recoveries are within limits in the re-analysis, the second analysis will be reported. If the LCS recoveries are out-of-limits a second time, the results from the first analysis will be reported with a narrative indicating the acceptance limits were exceeded for the associated LCS.

8.7 Laboratory Control Spike Duplicate (LCSD)

To provide a measure of instrument precision, the LCS is analyzed twice to provide an LCSD. The LCSD is analyzed once per analytical batch. The RPD between duplicates must be ≤ 25% for detections greater than 5 times the

reporting limit. If this limit is exceeded, the sample is re-analyzed for a third time. If the limit is exceeded again, the cause is investigated. If no problem is found on the system, the last two results are reported, and the data is narrated to note the non-conforming event.

8.8 Surrogate

8.8.1 An Internal Standard and Surrogate mix standard typically containing 125 µg/mL each of 2-Fluorotoluene and Toluene-d8 is spiked into all samples, standards and blanks prior to extraction. Toluene-d8 recovery must be within 70-130% to meet the acceptance criteria.

8.8.2 If Surrogate recovery exceeds these limits, the analyst will re-aliquot and re-analyze the extract. It is not possible to re-extract the samples. If the Surrogate recoveries are within limits in the re-analysis, the second analysis will be reported. If the Surrogate recoveries are out of limits a second time, the results will be reported from the first analysis with a narrative indicating the acceptance criteria for Surrogate recoveries were exceeded.

8.9 Method and Solvent Blanks

8.9.1 A Method Blank is extracted with each set of up to 20 extracted samples and is analyzed to ensure the extraction process is free from contamination. Either a Method Blank or Solvent Blank is analyzed immediately after the calibration standard to ensure the instrument is free from contamination.

8.9.2 If any target compound is detected above the reporting limit, re-aliquot and re-analyze the extract to confirm the presence of the target compound. If any target compound is detected above the reporting limit in the re-analysis, flag and report. If it doesn't confirm, investigate and correct the problem before re-analyzing all the affected samples.

9.0 CALCULATIONS

Relative Response Factor (RRF) =

$$\frac{\text{Area of Compound}}{\text{Area of Int. Standard}} \text{ X } \frac{\text{Conc. Int. Standard (µg/mL)}}{\text{Conc. of Compound (µg/mL)}}$$

Results Calculation (µg/mL) =

$$\frac{\text{Area of Compound in Sample}}{\text{Area of Int. Standard in Sample}} \times \frac{\text{Conc. Int. Standard (µg/mL)}}{\text{ICAL RRF*}}$$

*The average RRF from the initial calibration curve is used to quantitate results.

µg/Sample = µg/mL On Column × Final Volume (mL) × Dilution Factor

Media Type	Final Volume (mL)
Radiello	2.0
SKC 575/Ultra	2.0
WMS	1.0

All results are reported in units of microgram per sampler in Target using the extract volume to convert the on-column µg/mL results to total mass per sample. Results are not corrected for desorption efficiency unless requested by the client. Once the data has been transferred to the Atlas database, concentrations in $\mu g/m^3$ and/or ppbv are automatically calculated using the following formulas;

$$\mu g/m^3 = \text{Mass}(\mu g) \times 10^6 (m^3/mL)/[Q\ (mL/min) \times t(min)]$$

Q = Sampling rate (varies by compound and by sampler, see Appendix A)

t = Sample duration

ppbv = $\mu g/m^3$ x 24.45/Molecular Weight

The passive sample calculator is set up to provide temperature adjusted sampling rate by entering the average field temperature into the Atlas Log-in module. The relationship between temperature and sampling rate is detailed below:

$Q_k = Q_{298} * (K/298)^{1.5}$

K= average temperature during sampling in Kelvin

Q_{298} = Sampling rate at 298 K

Q_K = Sampling rate at sampling temperature K

Temperature corrections are not performed on the WMS samplers since the sampling rate does not vary significantly as a function of temperature.

The WMS media type logged into Atlas will pull the correct uptake rate (WMS-SE, WMS-LU, or WMS-TM) In order to correctly calculate the concentration.

9.1 *Software Verification*: A manual verification of the computer-generated results is performed on one randomly chosen target compound for each CCV.

10.0 SAMPLE PREPARATION AND ANALYSIS

10.1 WMS Sample Extraction

10.1.1 Before extracting, tap the top of the vial to remove any of the sorbent adhering to the membrane. Remove the cap using the de-crimper.

10.1.2 A 40 uL of the internal standard and surrogate STD mix at 125ug/mL is added to each sample and QC sample vials to yield a final concentration of 5.0ug/mL.

10.1.3 Add a 1 mL aliquot of Carbon Disulfide slowly to each sample to avoid solvent evaporation due to the generation of heat.

10.1.4 After the solvent and IS/S addition, re-crimp a cap onto the vial and place on a shaker to agitate for approximately 30 minutes.

10.1.5 After extraction, an aliquot (> 350uL*) of the extract is transferred to an insert in an autosampler vial for GC/MS analysis.

* limit headspace in the insert to eliminate VOC loss.

10.1.6 Extracted Quality Control Samples: With each batch of samples, a Method Blank and a Laboratory Control Spike (LCS) are prepared. To prepare the LCS, a standard solution containing target compounds is spiked directly onto the sorbent contained in an un-sampled WMS vial (a typical spike level is 5.0ug/mL.) Both the Method Blank and LCS are extracted and analyzed in the same manner as the field samples.

10.2 RAD 130 Sample Extraction

10.2.1 An 80uL of an internal standard and surrogate STD mix at 125ug/mL is directly added to each sample and QC sample vials to yield a final concentration of 5.0ug/mL.

10.2.2 A 2.0 mL aliquot of Carbon Disulfide is also added to the RAD 130 tubes.

10.2.3 After the addition, the tube is capped and placed on a shaker and agitated for approximately 30 minutes.

10.2.4 After extraction, an aliquot (>350uL*) of the extract is transferred to an insert with bottom spring in an autosampler vial for GC/MS analysis.

* limit headspace in the insert to eliminate VOC loss.

10.2.5 Extracted Quality Control Samples: With each batch of samples, a Method Blank and a Laboratory Control Spike (LCS) are prepared. To

prepare the LCS, a standard solution containing target compounds is spiked directly onto the sorbent contained in an un-sampled RAD 130 tube (a typical spike level is 5.0ug/mL.) Both the Method Blank and LCS are extracted and analyzed in the same manner as the field samples.

10.3 SKC 575 Sample Extraction

10.3.1 Cut tubes on back of sampler to provide access to the charcoal sorbent bed.

10.3.2 Add 80ul an internal standard and surrogate std mix at 125ug/ml is directly added to each sample and qc sample vials to yield a final concentration of 5.0ug/ml.

10.3.3 Slowly add 2.0 mL Carbon Disulfide and tightly press plugs in place.

10.3.4 Place on a shaker and agitate for approximately 30 minutes.

10.3.5 After extraction, an aliquot (> 350uL*) of the extract is transferred to an insert with bottom spring in an autosampler vial for GC/MS analysis.

* limit headspace in the insert to eliminate VOC loss.

10.3.6 Extracted Quality Control Samples: With each batch of samples, a Method Blank and a Laboratory Control Spike (LCS) are prepared. To prepare the LCS, a standard solution containing target compounds is spiked directly onto the sorbent contained in an un-sampled badge (a typical spike level is 5.0ug/mL.) Both the Method Blank and LCS are extracted and analyzed in the same manner as the field samples.

10.4 SKC Ultra Sample Extraction

10.4.1 Remove the back plugs of the sample bed and the blank bed and transfer charcoal from each bed into separate labeled 4.0 mL vials.

10.4.2 Add 80ul an internal standard and surrogate std mix at 125ug/ml is directly added to each sample and qc sample vials to yield a final concentration of 5.0ug/ml.

10.4.3 Slowly add 2.0 mL Carbon Disulfide and cap vials tightly

10.4.4 Place on a shaker and agitate for approximately 30 minutes.

10.4.5 After extraction, an aliquot (> 350uL*) of the extract is transferred to an insert with bottom spring in an autosampler vial for GC/MS analysis.

* limit headspace in the insert to eliminate VOC loss.

10.4.6 Extracted Quality Control Samples: With each batch of samples, a Method Blank and a Laboratory Control Spike (LCS) are prepared. To prepare the LCS, a standard solution containing target compounds is spiked directly onto the sorbent contained in an un-sampled SKC Ultra

badge (a typical spike level is 5.0ug/mL.) Both the Method Blank and LCS are extracted and analyzed in the same manner as the field samples.

10.5 Extraction/Analytical Batch

10.5.1 The extraction batch is defined as up to 20 samples and associated QC which is extracted together on one day.

10.5.2 The analytical batch is defined as up to 20 samples and associated QC that are analyzed together in one day on one instrument. Samples are logged into a work order as noted on the Chain-of-Custody. If samples extend to more than one analytical batch, appropriate QC will be reported. All QC and samples must be processed with the method associated with the daily analytical batch.

10.5.3 Analytical Sequence

Initial 12-hour period:	Subsequent 12-hour period:
BFB Tune Check	BFB Tune Check
Initial Calibration	CCV
ICV	Extracted LCS
Laboratory Blank	Extracted LCS duplicate
Extracted QC samples (LCS, LCSD, Method Blank)	Method Blank
Samples (up to 20)	Samples (up to 20)

The "Subsequent 12-hour" sequence is followed every 12-hour period that samples are analyzed, until the system is found to be out of calibration.

10.6 Validation of Reporting Limit (Limit of Quantitation)

10.6.1 Method Detection Limit (MDL) studies are analyzed for all standard compounds following procedures described in 40 CFR Pt. 136 App. B and Eurofins Air Toxics SOP #39.

10.6.2 The reporting limit (Limit of Quantitation) must be greater than the MDL before sample analysis can occur. If this is not achieved, corrective action, including raising the reporting limit is taken prior to continuing with sample analysis. See Appendix A for the Reporting Limits.

10.6.3 In general, the reporting limit (Limit of Quantitation) is typically up to 10 times greater than the MDL but may be up to 50 times greater. If the reporting limit (Limit of Quantitation) exceeds this range, the Lab Manager and/or the QA Department will evaluate the reporting limit to determine the reason and its approval.

10.6.4 The MDL verification sample must be analyzed on a quarterly basis. Refer to SOP #39 and WI39 MDL Procedure for the acceptance criterion.

10.7 Qualitative Target Compound Identification

10.7.1 There are three criteria that must be satisfied to justify positive identification of a given compound. 1.) The retention time must match the daily standard within a factor of less than 0.1 minutes for all ions present in the spectrum. Shifts in target compound retention times that exceed this limit must be accompanied by a corresponding shift in retention time for the corresponding Internal Standard. 2.) The peak shape should be Gaussian for all monitored ions of interest with the exception of those at or near the reporting limit. 3.) The ion abundance ratios should match the library reference spectrum unless obvious interference is noted on the full scan run. (See 11.9).

10.7.2 As guidance for determining proper ion ratios, relative intensities of characteristic ions of a compound should be within approximately 30% of the relative intensities of these ions in the reference spectrum. The spectrum of an individual compound in the CCV may be used as the reference to determine the ion ratio target.

10.7.3 Since the MS is configured to collect both SIM and full scan data for each run, background subtraction is primarily helpful when evaluating peaks on the full scan data file and can be used by the laboratory staff as a data evaluation tool to remove interfering ion masses from co-eluting compounds that may be masking the target compound. If background subtraction is needed in order to make a decision regarding the presence of a target compound then the subtraction should be included in the data package. The background subtraction of the spectra should be within $\pm$20 scans of the target peak. If the compound identification can be made without the use of background subtraction, i.e. retention time, ion ratio match, and Gaussian peak shape, then documentation is not required to be included in the data package.

10.7.4 If the above criteria are met then the compound has been positively identified. However, severe matrix interference may make it impossible to satisfy one or all of the above conditions. In these instances the scientist or analyst must evaluate the interference and be able to document the reason that the criteria cannot be satisfied. If this can be done, then the compound may be positively identified on the basis of analyst's "best judgment".

10.8 Quantitative Analysis

MSD instrument is set to acquire both SIM and full scan data simultaneously. This generates two separate data files in the analytical software. One file contains SIM data and the other contains full scan data. This allows a lower reporting limit for the selected SIM compounds. The data generated from SIM is used to report target compounds. The data generated by full scan is used as a reference for full spectra evaluation and may be used when non-standard compounds and/or Total Volatile Organic Compound (TVOC) determination are requested. Both the primary and secondary ions for each target compound are selected for selected ion monitoring by GC/MS software. Compounds are identified using both the ion profiles generated and the retention time windows.

The average RRF from the Initial Calibration Curve is used to quantitate the VOC results. Quantitation is based on the integrated abundance of the quantitation ion for each analyte. If the response of any quantitation ion exceeds the initial Calibration range of the GC/MS system, samples are diluted following procedures outlined in section 11.10. If interference with the quantitation ion is present, the potential bias is noted in the narrative.

10.9 Sample Dilutions

If the concentration is above the linear range of the working standards, dilute with a Carbon Disulfide solution containing 5.0ug/mL of internal standard, re-analyze, and apply the appropriate dilution factor in calculations.

10.10 Manual Integrations

At times, performing manual integrations on Initial Calibration standards, QC, Laboratory Blanks and samples may be necessary due to limitations with the data processing software. It is strongly discouraged to manually integrate QC samples.

11.0 CORRECTIVE ACTION PROCEDURES

A request for corrective action (CAR) is initiated any time either the EATL SOPs or client-prescribed QC protocol are not followed, or in any other instance that sample results are adversely affected. The corrective action system is documented in Eurofins Air Toxics SOP #61.

12.0 DATA REVIEW

12.1 Analytical Review

The GCMS unit is equipped with an autosampler. The run is reviewed by the chemist using the following steps:

12.1.1 Check for any project-specific requirements.

12.1.2 Verify holding time.

12.1.3 Verify Tune Check, CCV, LCS, LCSD, and Method Blank have met criteria.

12.1.4 Verify sample results:

a. Verify internal standard, retention time, and surrogate recoveries are acceptable.

b. Verify reasonableness of the automated peak integration and spectral verification

c. Verify result concentrations are within linear range of calibration curve.

d. Verify that all samples were analyzed within the project of method specified clock

12.1.5 Initial and date sequence log to indicate that the data is acceptable.

12.1.6 Transfer the BFB, CCV, LCS, LCSD, and Method Blank to the Atlas database.

12.1.7 Mark the associated workorder samples analyzed as complete in the Atlas database.

- Describe unusual events on the Data Review Checklist and sign and date the Analytical Review field. (If the runs are checked simultaneously with data write-up and report generation, then the chemist simply competes the steps in section 13.2 and signs and dates the 'Write-up' field only.)

12.2 Write-up and Final Report Review

The chemist performing the data write-up and final client report reduces the data by reviewing the Target data files. The peaks in each sample are reviewed for the correct integration and identification. The criteria for compound identification are described in section 11.7. The data is evaluated for the project required sublist only.

When the sample Target review is complete, the sample files are transferred to the Atlas database. The client report is compiled in the Atlas workorder editor following EATL SOP#78. The following is checked when compiling the report:

- Ensure the sample duration is accurately listed on the Log-in page. For the blank, the longest duration is used to convert to units of concentration.

- Ensure the correct sampler type is selected on the log-in page. It is critical that the correct sampler type is selected to insure that the proper uptake rates are used to convert mass units to concentration. If WMS samplers are utilized, verify that Atlas has the correct type – WMS-SE, WMS-LU, or WMS-TM depending on whether the standard, low uptake, or thick membrance version was used for sample collection.

- Prepare and review narrative , detailing QC non-compliance as needed.

- Evaluate data package from a data user perspective. Does the data make sense?

- Review the final pdf with the calculated concentrations to insure that the uptake rates are properly applied and durations are entered.

12.3 Technical Data Review

The Scientist or designated personnel performs a Technical Data Review on 100 % of the reports if the write-up review analyst is not signed-off for the method. This review follows all the steps mentioned in the Analytical/Write-up Data Review (see Section 13.1 and 13.2).

12.4 Report Review

The Report Review represents a 3rd level of review which is required for DoD and client specific projects only. Refer to the Data Review Checklist for the specific items requiring review.

12.5 QA Data Review

A thorough QA data review is performed by the QA Department on final data packages requesting 100% review and at the discretion of the QA department. The QA review entails verification that project and QC requirements are met. Failure to meet QC and/or project requirements results in a Corrective Action Request (CAR) and documentation. Dilution factors, analyte retention times, peak integration areas, concentration calculations, unit conversions, and reporting limits are also checked. Sample trends and field QC performance are observed.

13.0 INSTRUMENT MAINTENANCE

Instruments are monitored on a daily basis by the bench analyst for any potential failure. The analysis of blanks and control standards at the start of the day and as analysis continues helps to provide real time feedback to the analyst on the condition of the instruments. Routine maintenance includes:

13.1 Mass Spectrometers

- Prior to start of analytical batch, check of vacuum ion gauge (increase in ion count indicates a potential leak)
- At the beginning of the analytical batch, perform a tune check with BFB
- Cleaning of ion source as needed
- The oil level and quality is visually checked approximately monthly to ensure proper vacuum pump function, and oil is changed approximately every 6 months.

13.2 Gas Chromatograph

Basic maintenance includes the following:

- Clip about six inches to one foot off the front end of the capillary column, and if necessary, the back end as well.
- Replace the injection port liner. The liner is replaced by removing the inlet cap using a wrench and releasing the liner from the inlet body using a pair of tweezers. Care should be taken not to get finger prints on any inside surface.
- Change the septa on the GC and on the internal standard and surrogate loading station as needed. Always use a high temperature/low bleed septa and take care not to leave finger prints on any inside heated surface. Wear a pair of white cotton gloves or use tweezers to handle the septa. Lower the oven temperature to 40°C. Remove the inlet cap with a wrench, remove the old septa with a pair of tweezers and insert the new septa.
- The column is replaced when chromatography peak shape or resolution degrades. Similarly, if the column-bleed profile rises with age then the column needs replacing. Use new black graphite ferrules each time and clip off approximately 1" of column after inserting it through the ferrule. This will remove any graphite particles that may have scrapped off into the column. Tighten the column nut and ferrule finger tight and one quarter turn with a wrench. Tightening any more only crushes the ferrule and may damage the column.
- The bench analyst will document any routine or major maintenance in the bound instrument logbook assigned to each instrument. The date of the maintenance, what work was performed and analyst initials are included.

14.0 DELIVERABLES

Data reporting packages are prepared as described in SOP #78: Generation of Eurofins Air Toxics Data Deliverables, Electronic Conversion, and Archival.

15.0 REFERENCES

- EPA Method TO-17: Determination of volatile organic compounds in ambient air using active sampling onto sorbent tubes, EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-17, January, 1999.
- NIOSH Manual of Analytical Methods, 3rd Edition, Volume 2
- FSM Radiello manual (http://www.sigmaaldrich.com/etc/medialib/docs/Supelco/Application_Notes/radiellomanual_final.Par.0001.File.dat/radiello_manual_final.pdf) accessed 6/6/09.
- SKC 575 Series Passive samplers, Form #37009
- SKC sampling rates (www.skcinc.com) Publication 1811 Rev 1201 accessed 12-19-12
- Eurofins Air Toxics Laboratory Quality Assurance Manual (LQAM): Definitions and Terms, Appendix A.

16.0 LIST OF APPENDICES

Appendix A

Summary of Calibration and QC Procedures

Table A-1 Target compounds, QC & Reporting Limits

Table A-2 Special Target compounds, QC & Reporting Limits

Table A-3 Internal Standard

Table A-4 Surrogate

Table A-5 Sampling Rates for "Standard" target compounds (RAD 130)

Table A-6 Sampling Rates for "Standard" target compounds (SKC 575)

Appendix B

BFB Tuning Criteria

Note: Sampling rates for WMS samplers are proprietary information of SiREM.

Appendix A
Summary of Calibration and QC Procedure

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at the start of every 12-hour clock.	Method TO-17 tuning criteria.	Correct problem then repeat tune.
Initial 5-Point Calibration	Prior to sample analysis. For DoD projects, the surrogate Toluene-d8 must be supported by a multi-point ICAL over a concentration range. Calibration at a single level concentration is allowed only for non-DoD projects.	Compound criteria in Table A-1.	Correct problem then repeat initial calibration. Analysis may proceed if no more than 2 VOCs exceed criteria or 5% of VOCs if short list is used. Narrate exceedances.
Initial Calibration Verification (ICV)	Once per initial calibration.	See Table A-1	Verify concentrations and standard preparation. Analysis may proceed if no more than 2 VOCs exceed criteria or 5% of VOCs if short list is used. Narrate exceedances.
Continuing Calibration Verification (CCV)	At the start of every shift immediately after the BFB tune check.	See "CCV criteria" column in Table A-1	Investigate and correct the problem, up to and including recalibration if necessary. Analysis may proceed if no more than 2 VOCs exceed criteria or 5% of VOCs if short list is used. Associated results are flagged.
Internal Standards (IS)	IS is added at the time of extraction to all samples and QC samples.	**For CCVs:** area counts 50% - 200%, RT w/in 30 sec of mid-point in ICAL. **For blanks, samples and non-CCV QC Checks:** area counts 50 – 200%, RT w/in 20 sec. of RT in CCV.	**CCV:** inspect and correct system prior to sample analysis. **For blanks:** inspect the system and re-analyze the blank. **For samples:** re-analyze; if out again, flag data.
Surrogate	Surrogate is added at the time of extraction to all samples and QC samples.	70-130%	Same as for Internal Standards.
Solvent Blanks	Immediately after the calibration standard or after samples with high concentrations	Results less than laboratory reporting limit (see Table A-1).	Re-aliquot and re-analyze solvent blank. If detections remain, flag concentrations in associated samples.
Extracted Laboratory Blank	Each set of up to 20 samples	Results less than the reporting limit.	Flag sample concentrations in associated extraction batch.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Extracted LCS	Each set of up to 20 samples.	See Table A-1	Analysis may proceed if no more than 2 VOCs exceed criteria (or 5% for short list exceed criteria). Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise, narrate.
LCSD	Each set of up to 20 samples	%RPD ≤ 25%	Analysis may proceed if no more than 2 VOCs exceed criteria (or 5% for short list exceed criteria). Narrate as appropriate.

Table A-1. Target Analytes QC & Reporting Limit

Analytes	Reporting Limit (µg/mL)	Acceptance Criteria			
		ICAL (%RSD)	ICV (% R)	LCS (% R)	CCV (%D)
Chloromethane	0.2	30	70 – 130	50 – 140	≤40%
Vinyl Chloride	0.2	30	50– 140	50 – 140	≤40%
Ethanol	0.5	30	70 – 130	50 – 130*	≤30%
1,1-Dichloroethene	0.2	30	70 – 130	70 – 130	≤30%
MTBE	0.05	30	70 – 130	70 – 130	≤30%
trans-1,2-Dichloroethene	0.1	30	70 – 130	70 – 130	≤30%
Hexane	0.05/0.20***	30	70 – 130	70 – 130	≤30%
1,1-Dichloroethane	0.05	20	80 – 120	70 – 130	≤20%
Ethyl Acetate	0.2	30	70 – 130	70 – 130	≤30%
2-Butanone	0.05/0.10***	30	70 – 130	70 – 130	≤30%
cis-1,2-Dichloroethene	0.05	20	80 – 120	70 – 130	≤20%
Chloroform	0.05	20	80 – 120	70 – 130	≤20%
Cyclohexane	0.05	30	70 – 130	70 – 130	≤30%
1,1,1-trichloroethane	0.05	20	80 – 120	70 – 130	≤20%
Carbon Tetrachloride	0.05	20	80 – 120	70 – 130	≤20%
Benzene	0.2	30	70 – 130	70 – 130	≤30%
1,2-Dichloroethane	0.05	20	80 – 120	70 – 130	≤20%
Heptane	0.05	20	80 – 120	70 – 130	≤20%
Trichloroethene	0.05	20	80 – 120	70 – 130	≤20%
4-Methyl-2-pentanone	0.1	30	70 – 130	70 – 130	≤30%
Toluene	0.05	20	80 – 120	70 – 130	≤20%
1,1,2-Trichloroethane	0.05	20	80 – 120	70 – 130	≤20%
Tetrachloroethene	0.05	20	80 – 120	70 – 130	≤20%
Chlorobenzene	0.05	20	80 – 120	70 – 130	≤20%
Ethylbenzene	0.05	20	80 – 120	70 – 130	≤20%
m,p-Xylene	0.05	20	80 – 120	70 – 130	≤20%
o-Xylene	0.05	30	70 – 130	70 – 130	≤30%
Styrene	0.05	30	70 – 130	20 – 100*	≤30%
1,1,2,2-Tetrachloroethane	0.05	30	70 – 130	60 – 130	≤30%
Propylbenzene	0.05	20	80 – 120	70 – 130	≤20%

1,3,5-Trimethylbenzene	0.05	20	80 – 120	70 – 130	≤20%
1,2,4-Trimethylbenzene	0.05	20	80 – 120	70 – 130	≤20%
1,3-Dichlorobenzene	0.05	30	70 – 130	50 – 110**	≤30%
1,4-Dichlorobenzene	0.05	30	70 – 130	50 – 110**	≤30%
1,2-Dichlorobenzene	0.05	30	70 – 130	50 – 110**	≤30%
Naphthalene	0.05	30	70 – 130	5 – 80*	≤30%

Acceptance limits based on Desorption efficiency studies.
** 60 – 130% for WMS, ***RL for WMS

Table A-2
Non-standard Analytes

Analyte	Reporting Limits (ug/mL)	ICAL (%RSD)	ICV (%R)	LCS (%R)	CCV (%D)
Halothane	0.1	30	70-130	70-130	≤30%
2-Chloroethyl vinyl ether*	1.0	30	70-130	60-130	≤30%
Methyl Methacrylate	0.05	30	70-130	70-130	≤30%
a-Pinene	0.05	30	70-130	60-130	≤30%
R-(+)-Limonene	0.05	30	70-130	70-130	≤30%
1,4-Dithiane	0.05	30	70-130	50-110	≤30%
1,2,4-Trichlorobenzene	0.05	30	70-130	20-110	≤30%
1,2,3-Trichlorobenzene	0.05	30	70-130	20-110	≤30%

*Need special approval, compound is unstable.

Table A-3
Internal Standard

Analyte	CCV IS (%R)	Sample IS (%R)
2-Fluorotoluene	-50 to +200	-50 to +200

Table A-4
Surrogate

Analyte	%R
Toluene-d_8	70-130

Table A-5
Sampling Rates for Routinely Calibrated target compounds (RAD 130)

Analytes	Reporting Limit (µg/mL)	Reporting Limit (µg/sampler)	Sampling Rates for Radiello 130 Sampler
Vinyl Chloride[1]	0.2	0.4	90*
Ethanol	0.5	1.0	102
1,1-Dichloroethene	0.2	0.4	76*
MTBE	0.05	0.1	65
trans-1,2-Dichloroethene	0.1	0.2	60*
Hexane	0.05	0.1	66
1,1-Dichloroethane	0.05	0.1	63*
Ethyl Acetate	0.2	0.4	78
2-Butanone	0.1	0.2	79
cis-1,2-Dichloroethene	0.05	0.1	62*
Chloroform	0.05	0.1	75
Cyclohexane	0.05	0.1	54
1,1,1-trichloroethane	0.05	0.1	62
Carbon Tetrachloride	0.05	0.1	67
Benzene	0.2	0.4	80
1,2-Dichloroethane	0.05	0.1	77
Heptane	0.05	0.1	58
Trichloroethene	0.05	0.1	69
4-Methyl-2-pentanone	0.1	0.2	67
Toluene	0.05	0.1	74
1,1,2-Trichloroethane	0.05	0.1	66*
Tetrachloroethene	0.05	0.1	59
Chlorobenzene	0.05	0.1	68
Ethylbenzene	0.05	0.1	68
m,p-Xylene	0.05	0.1	70
o-Xylene	0.05	0.1	65
Styrene	0.05	0.1	61
1,1,2,2-Tetrachloroethane	0.05	0.1	60*
Propylbenzene	0.05	0.1	57
1,3,5-Trimethylbenzene	0.05	0.1	53*
1,2,4-Trimethylbenzene	0.05	0.1	50
1,3-Dichlorobenzene	0.05	0.1	59*
1,4-Dichlorobenzene	0.05	0.1	51
1,2-Dichlorobenzene	0.05	0.1	58*
Naphthalene	0.05	0.1	25

1. Vinyl chloride is included in the calibration standard; however, it is not a "standard" compound as it is not recommended on the RAD130 cartridge due to poor retention. Applications using the RAD130 for VC measurements for extended durations (>8 hours) will result in significant low bias using the theoretical uptake rate. All lab reports with vinyl chloride reported must have the appropriate narration regarding poor retention and potential low bias.

*Estimated using the diffusion coefficient in air and the geometric constant 14.145 cm.

Note: Sampling rates provided are appropriate for the 'white' diffusive body, code 120.

Table A-6
Sampling Rates for "Standard" target compounds
(SKC 575/Ultra)

Analytes	Reporting Limit (µg/mL)	Reporting Limit (µg/sampler)	Sampling Rates for Indoor Air Applications 'Zero Face velocity'	Sampling Rates for Outdoor/worker exposure (ml/min)
Vinyl Chloride[1]	0.2	0.4	17.4*	21.2*
Ethanol	0.5	1.0	11.7	20.0
1,1-Dichloroethene	0.2	0.4	9.74	12.3
MTBE	0.05	0.1	9.84	13.6
trans-1,2-Dichloroethene	0.1	0.2	10.2	14.8
1,1-Dichloroethane	0.05	0.1	13.14	12.3
Ethyl Acetate	0.2	0.4	9.26	13.75
2-Butanone	0.05	0.1	6.27	17.1
cis-1,2-Dichloroethene	0.05	0.1	11.54*	14.8*
Chloroform	0.05	0.1	10.14	13
Cyclohexane	0.05	0.1	7.76	15.6
1,1,1-trichloroethane	0.05	0.1	9.40	14.1
Carbon Tetrachloride	0.05	0.1	10.41	14.1
Benzene	0.2	0.4	10.69	16
1,2-Dichloroethane	0.05	0.1	11.79	14.2
Heptane	0.05	0.1	9.38	13.9
Trichloroethene	0.05	0.1	11.47	14.9
4-Methyl-2-pentanone	0.1	0.2	7.29	13.5
Toluene	0.05	0.1	8.90	14.5
1,1,2-Trichloroethane	0.05	0.1	9.64	12.5
Tetrachloroethene	0.05	0.1	10.02	13.1
Chlorobenzene	0.05	0.1	8.23*	18.74*
Ethylbenzene	0.05	0.1	9.02	12.9
m,p-Xylene	0.05	0.1	8.1	12.65
o-Xylene	0.05	0.1	8.11	11.9
Styrene	0.05	0.1	9.04	13.7
1,1,2,2-Tetrachloroethane	0.05	0.1	9.98	11.8
Propylbenzene	0.05	0.1	6.41*	11.69*
1,3,5-Trimethylbenzene	0.05	0.1	7.29*	12.1*
1,2,4-Trimethylbenzene	0.05	0.1	9.92*	12.1*
1,3-Dichlorobenzene	0.05	0.1	5.79*	12.7*
1,4-Dichlorobenzene	0.05	0.1	10.74*	12.7*
1,2-Dichlorobenzene	0.05	0.1	4.97*	12.6*
Naphthalene	0.05	0.1	2.71*	13.7*

*Calculated by SKC

1. Vinyl chloride is included in the calibration standard; however, it is not a "standard" compound as it is not recommended on the SKC badges due to poor retention.

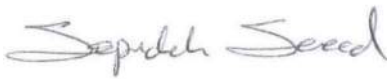
Appendix B

BFB Tune Criteria

4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	8 to 40% of mass 95
75	30 to 66% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	< 2% of mass 174
174	50 to 120% of mass 95
175	4 to 9% of mass 174
176	93 to 101% of mass 174
177	5 to 9% of mass 176

Document Owner/Laboratory Director:

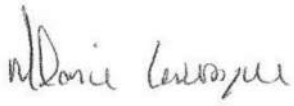


Date:05/22/19

Technical Director:

Date: 05/20/19

Quality Assurance:



Date: 03-26-19