F I N A L

Quality Assurance Project Plan
OU-2 Remedial Investigation
700 South 1600 East PCE Plume
Salt Lake City, Utah

Contract No. W912DQ-15-D-3014 Task Order 0005
Version 0

Prepared for

U.S. Army Corps of Engineers

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Title and Approval Sheet (A1)

Quality Assurance Project Plan (QAPP) Version 0

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Operable Unit 2 – 700 South and 1600 East PCE Plume, Salt Lake City, Utah

CH2M HILL Project Manager: _____ Date: _____

CH2M HILL (Prime)

CH2M HILL Technical Reviewer: _____ Date: _____

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CH2M HILL Independent Technical Reviewer: _____ Date: _____

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USACE Project Manager: _____

USACE Date: _____

VA COR/Remedial Program Manager, CERCLA: _____ Date: _____

Department of Veteran's Affairs

EPA Project Manager: _____ Date: _____

EPA Region 8

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EPA Region 8

UDEQ Project Manager: _____

Utah Department of Environmental Quality Date: _____

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Acronyms and Abbreviations

µg/L	microgram(s) per liter
µg/m^3	microgram(s) per cubic meter
°C	degrees Celsius
%D	percent difference
%R	percent recovery
AOU	Accelerated Operable Unit
aq	aqueous
BHC	benzene hexachloride
CA	California
CAR	corrective action report
CAS	Chemical Abstracts Service
CC-ACA	Certified Chemist – American Chemical Society
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response Compensation and Liability Act
CGWP	Certified Groundwater Professional
CH	Certified Hydrogeologist
CH2M	CH2M HILL, Inc.
COPC	constituent of potential concern
CSM	conceptual site model
DAF	dilution attenuation factor
DCV	daily calibration verification
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethene
DDT	dichlorodiphenyltrichloroethane
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
GC/MS	gas chromatograph/mass spectrometer
GIS	geographic information system
GPS	geographic positioning system
H_2SO_4	sulfuric acid
HCl	hydrochloric acid
HNO_3	nitric acid

ICAL	initial calibration
ICS	interference check solutions
ID	identification
IGES	Intermountain GeoEnvironmental Services, Inc.
IS	internal standard
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 quality assurance project plan specified reporting limit
L	liter
LCL	lower control limit
LCS	laboratory control sample
LIMS	laboratory information management system
m^3/kg	cubic meter(s) per kilogram
MB	method blank
MCL	maximum contaminant level
MDL	method detection limit
mg/kg	milligram(s) per kilogram
mg/L	milligram(s) per liter
mL	milliliter(s)
MO	Missouri
MPC	measurement performance criteria
MS	mass spectrometer; matrix spike
MSA	method of standard addition
MSD	matrix spike duplicate
NA	not applicable
NaOH	sodium hydroxide
NCR	non-conforming report
NFG	National Functional Guideline
OU	operable unit
PB	performance based
PCE	tetrachloroethene
PDS	post digestion spike
PE	Professional Engineer
PG	Professional Geologist
PM	project manager
PMP	Project Management Professional
ppbV	part(s) per billion by volume
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
QCSM	quality control systems manager
qPCR	quantitative polymerase chain reaction
QSM	quality systems manual

R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified
RDL	representative detection limit
RI	remedial investigation
RIWP	remedial investigation work plan
RL	reporting limit
RPD	relative percent difference
RRF	relative response factor
RSD	relative standard deviation
RSL	regional screening level
RT	retention time
SDG	sample delivery group
SIRFER	Stable Isotope Ratio Facility for Environmental Research
SOP	standard operating procedure
SRM	standard reference material
SSL	soil screening level
SVOC	semivolatile organic compound
TDS	total dissolved solids
TN	Tennessee
TOC	total organic carbon
TSA	technical system audit
U	The compound was analyzed for, but was not detected above, the reported method detection limit
UCL	upper control limit
UDEQ	Utah Department of Environmental Quality
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
USACE	U.S. Army Corps of Engineers
USACE CQM	U.S. Army Corps of Engineers Certified Construction Quality Manager
USACE-KC	U.S. Army Corps of Engineers, Kansas City District
USCS	Unified Soil Classification System
UT	Utah
VHA	Veterans Health Administration
VISL	vapor intrusion screening level
VOA	volatile organic analytic
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 remedial investigation (RI) being conducted at the 700 South 1600 East PCE Plume Superfund Operable Unit 2 (OU-2) site in Salt Lake City, Utah. The QAPP is provided as part of the remedial investigation work plan (RIWP) (CH2M, 2018) and can be found in Appendix A-2 of the RIWP. The QAPP, its data quality objectives (DQOs), and its supporting document, the field sampling plan (FSP) (Appendix A-1 of the RIWP [CH2M, 2018]), were developed to document the type and quality of data needed for environmental decisions.

The QAPP follows U.S. Environmental Protection Agency (EPA) guidelines contained in *EPA Guidance for Quality Assurance Project Plans* (EPA, 2002), and *EPA Requirements for Quality Assurance Project Plans* (EPA, 2001, reissued 2006). The contents of the QAPP also meet the requirements of the *Uniform Federal Policy for Quality Assurance Project Plans* (EPA, 2005). 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 ensure 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, 2001, 2006).
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)

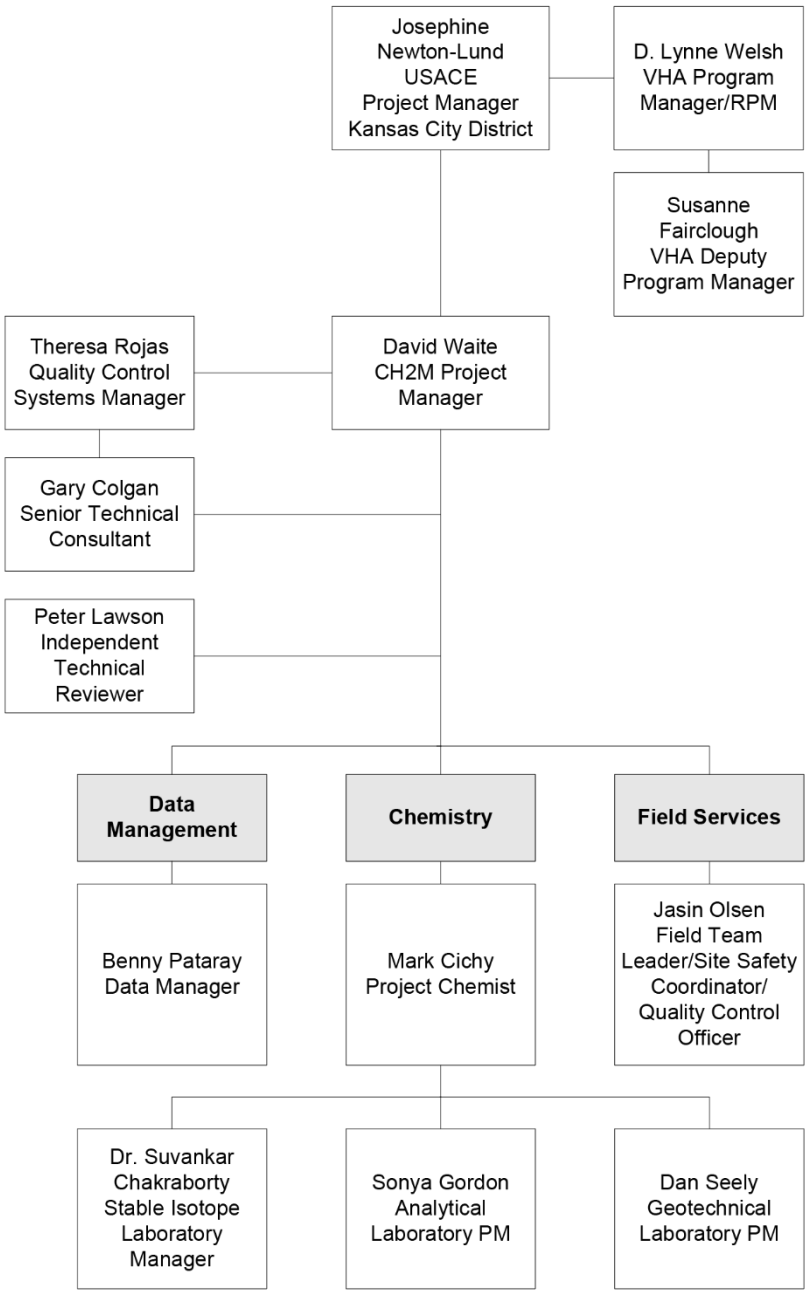
The Comprehensive Environmental Response Compensation and Liability Act (CERCLA) investigations at OU-2 will be conducted by CH2M HILL, Inc. (CH2M). The investigations will be staffed with experienced personnel with proper training to provide consistent technical support for activities defined in this QAPP and the associated FSP (Appendix A-1 of the RIWP [CH2M, 2018]).

The project organization and lines of authority for CH2M 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.

Table 2-1. Project Team Members
Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Name	Title	Location	Telephone Number
D. Lynne Welsh	VHA Program Manager/RPM	VHA, Salt Lake City, UT	801-582-1565, ext. 2021
Susanne Fairclough	VHA Deputy Program Manager	VHA, Salt Lake City, UT	801-582-1565, ext. 1952
Josephine Newton-Lund, PMP	USACE Project Manager	USACE-KC, Kansas City, MO	816-289-3912
David Waite, PE	Project Manager	CH2M, Taylorsville, UT	385-474-8560
Theresa Rojas, USACE CQM	Quality Control Systems Manager	CH2M, Atlanta, GA	678-530-4297
Gary Colgan, PG (UT, WY), CGWP	Senior Technical Consultant	CH2M, Taylorsville, UT	385-474-8512
Peter Lawson, PG (CA), CH (CA)	Independent Technical Reviewer	CH2M, Redding, CA	530-229-3383
Benny Pataray	Data Manager	CH2M, Taylorsville, UT	385-474-8545
Mark Cichy, CC-ACA	Project Chemist	CH2M, Redding, CA	530-229-3274
Jasin Olsen	Field Team Leader/Site Safety Coordinator/Quality Control Officer	CH2M, Taylorsville, UT	801-660-9741
Sonya Gordon	Analytical Laboratory Project Manager	Empirical Laboratories, Nashville, TN	877-345-1115, ext. 238
Dan Seely, PE	Geotechnical Laboratory Project Manager	IGES	801-270-9400
Dr. Suvankar Chakraborty	Stable Isotope Laboratory Manager	SIRFER	801-581-4654

Notes:
CA = California
CC-ACA = Certified Chemist – American Chemical Society
CERCLA = Comprehensive Environmental Response Compensation and Liability Act
CGWP = Certified Groundwater Professional
CH = Certified Hydrogeologist
GA = Georgia
IGES = Intermountain GeoEnvironmental Services, Inc.
MO = Missouri
PE = Professional Engineer
PG = Professional Geologist
PMP = Project Management Professional
SIRFER = Stable Isotope Ratio Facility for Environmental Research
TN = Tennessee
USACE = U.S. Army Corps of Engineers
USACE CQM = U.S. Army Corps of Engineers Certified Construction Quality Manager
USACE-KC = U.S. Army Corps of Engineers, Kansas City District
UT = Utah
VHA = Veterans Health Administration
WY = Wyoming



1
2 Figure 2-1. Organizational Chart

2.1.1 Veterans Health Administration Program Manager and Deputy Program Manager

Ms. D. Lynne Welsh, as Program Manager, will serve as the Veterans Health Administration (VHA) environmental restoration manager for the OU-2 RI. Ms. Susanne Fairclough will serve as the VHA deputy program manager. The VHA will be involved in the day-to-day project delivery. Where quality assurance (QA) problems or deficiencies in this QAPP requiring special action are identified, the CH2M project manager and quality control systems manager (QCSM), in conjunction with VHA program managers, will identify the appropriate corrective action to be initiated by the field team leaders (FTLs) or the laboratory, and ensure the QAPP is amended as necessary.

2.1.2 U.S. Army Corps of Engineers Project Manager

Ms. Josephine Newton-Lund provides overall project management for the OU-2 RI. Ms. Newton-Lund will coordinate all project matters with VHA and CH2M. A multidisciplinary team will review and provide comment on written deliverables through the project manager (PM).

2.1.3 Project Manager

Mr. David Waite will coordinate project responsibilities and financial, schedule, quality, and technical aspects of the project. The PM, in consultation with the QCSM, will maintain and revise the official approved FSP, RIWP, and QAPP documents, and ensure distribution of revisions to project members.

2.1.4 Quality Control Systems Manager

Ms. Theresa Rojas will administer a comprehensive QA/quality control (QA/QC) program that includes procedures needed to ensure the required levels of data quality and project QC are achieved. This QA/QC program includes conducting quarterly reviews of the project technical issues, quality, cost, and schedule; selecting, approving, and training subject-specific senior technical reviewers; ensuring a lead senior technical reviewer has been assigned to the project; and administering performance audits of work undertaken during the investigation of OU-2. If the QCSM or PM identify quality issues associated with work conducted by subcontractors, the QCSM will conduct a quality system review of that subcontractor and will require and manage a corrective action plan.

2.1.5 Senior Technical Consultant

Mr. Gary Colgan, a hydrogeologist with more than 30 years of experience, will serve as the senior technical consultant and subject matter expert for hydrogeology including groundwater characterization and aquifer testing, and provide input on all hydrogeological-related documents, identify other subject matter experts that may be required, and ensure the project's compliance with current state of the science. Mr. Colgan will serve as a primary technical reviewer on CH2M deliverables, and will sign off on the Statement of Technical Review form. Mr. Colgan will also provide technical oversight and direction to the field team leader.

2.1.6 Independent Technical Reviewer

Mr. Peter Lawson, a senior hydrogeologist, will provide independent technical review for the project and of CH2M client deliverables, providing the team technical input on project strategy and direction, and ensuring that proper technical reviews are completed. Mr. Lawson will also sign off on the Statement of Technical Review form.

2.1.7 Data Manager

Benny Pataray will ensure field and lab data is managed appropriately, maintain the field and analytical data in EQuIS, and archive data as required by the contract. He will communicate with the PM, QCSM, project chemist, and field staff about any issues or problems with field or laboratory data.

2.1.8 Project Chemist

Mr. Mark Cichy will manage, coordinate, and oversee the analytical laboratories. He will also ensure that laboratory data are delivered complete and are validated in compliance with the QAPP.

2.1.9 Field Team Leader/Site Safety Coordinator/Quality Control Officer

Mr. Jasin Olsen, a geologist with more than 5 years of field experience, will ensure all field investigation activities performed by CH2M and its subcontractors comply with the requirements in the approved work planning documents. Mr. Olsen will also ensure that field activities meet the quality metrics outlined in the work planning documents and that appropriate QC inspections are performed and properly documented.

2.1.10 Laboratory Project Managers

Ms. Sonya Gordon (Empirical Laboratories) will act as the analytical laboratory PM. In this role, Ms. Gordon will oversee laboratory analytical performance and ensure all laboratory protocols are followed to ensure analytical results meet QA requirements. Ms. Gordon, as the analytical laboratory PM, will manage all subcontract laboratories (TestAmerica – Sacramento and TestAmerica – Pittsburg).

Intermountain GeoEnvironmental Services, Inc. has been tentatively identified as the laboratory for geotechnical analyses for soil samples collected from well borings. Dan Seely, PE, would serve as the laboratory PM. Mr. Seely would ensure that the geotechnical testing follows the procedures and QC criteria in the applicable ASTM standards.

The Stable Isotope Ratio Facility for Environmental Research (SIRFER) laboratory at the University of Utah will perform stable isotope analysis for water samples. Dr. Suvankar Chakraborty is the laboratory manager and will oversee laboratory work, verify that all laboratory protocols are followed, and ensure that the results meet QA requirements.

The PM and project chemist will communicate with the laboratory PMs to resolve problems or issues related to the laboratory analyses.

2.2 Problem Definition/Background (A5)

2.2.1 Background

Detailed information regarding site background can be found in Section 2 of the RIWP (CH2M, 2018).

2.2.2 Problem Definition

The RIWP provides an evaluation of DQOs including a problem definition. The problem definition is as follows:

> Tetrachloroethene (PCE)-contaminated groundwater is presumed to be present beneath VHA property and is present in areas hydraulically downgradient of VHA property. PCE-contaminated groundwater was also drawn toward SLC-18 when the well was in operation. People could be exposed to the PCE-contaminated groundwater through two avenues:
>
> - Existing or new water-supply wells including SLC-18, if it is brought back online
> - The currently available information supports the conclusion that exposures to PCE-contaminated groundwater through the use of water-supply wells is controlled by Salt Lake City temporarily removing SLC-18 from service.
> - Vapor-intrusion and direct-contact pathways associated with shallow-groundwater and springs in the East Side Springs areas (currently managed as part of Accelerated Operable Unit 1 [AOU-1])

- Investigations and remedies associated with shallow groundwater within AOU-1 are being addressed separately under the Federal Facilities Agreement and will be incorporated by reference into OU-2 CERCLA documents.
- A time-critical removal action associated with shallow groundwater within AOU-1 is being addressed under VHA removal action authority for now and will be incorporated, at a later time, into the OU-2 RI.

Additional data are needed to characterize the hydrogeology and nature and extent of PCE contamination, to assess potential exposure pathways and risks, and allow development and detailed analysis of remedial alternatives during the subsequent feasibility study (CH2M, 2018).

Section 2.4 provides information on DQOs.

2.3 Project Description (A6)

The tasks described in the RIWP (CH2M, 2018) reflect work that VHA will do as part of Phase 1 of the RI; Phase 2, follow-on work, will be developed after the draft interim RI Report is submitted to EPA and Utah Department of Environmental Quality (UDEQ).

2.3.1 Description of Work Tasks

The activities to be completed as part of the Phase 1 RI include the following:

- Collection and analysis of soil gas samples, with onsite screening-level analysis of the soil gas samples using the HAPSITE gas chromatograph/mass spectrometer (GC/MS) and analysis of a subset of samples at the offsite laboratory for verification
- Collection and offsite analysis of soil samples, depending on the results from the soil gas samples
- Installation of shallow and deep groundwater wells
- Push-ahead groundwater sampling during deep monitoring well drilling, with onsite screening-level analysis of the groundwater samples using the HAPSITE GC/MS
- Collection of soil samples during well installation for geotechnical testing at an offsite laboratory
- Aquifer slug testing on shallow wells
- Aquifer pumping tests on deep monitoring wells.
- Geophysical and groundwater flow logging within wells
- Groundwater monitoring of new and existing monitoring wells, with collection of field parameters and analysis at offsite laboratories
- Surface water sampling, with collection of field parameters and analysis at offsite laboratories

At the completion of Phase 1 fieldwork, the data will be analyzed and remaining data gaps will be identified (if any). Phase 2 of the RI will be implemented at that time to fill these data gaps. Additional field methods described in this QAPP that may be used in subsequent phases (Phase 2 and potential follow-on phases) of the investigation include the following:

- Source investigation including soil gas testing, surface and near-surface soil sampling, and subsurface soil sampling (using direct-push drilling)
- Additional groundwater sampling including ongoing monitoring of existing monitoring wells and the installation and sampling of temporary groundwater sampling points installed through direct-push methods
- An aquifer pump test at the drinking water supply well SLC-18

- Cone-penetration testing
- Additional surface water sampling
- Ecological site reconnaissance
- Well maintenance as necessary

This QAPP addresses analyses that could be performed during both Phase 1 and Phase 2 of the fieldwork. In contrast, the FSP (Appendix A-1 of the RIWP [CH2M, 2018]) addresses Phase 1 only, and will be updated as needed to address additional sampling after Phase 1 is complete. Analyses that could potentially be performed during Phase 2 are described in the RIWP as toolbox items in Section 5.3 of the RIWP.

2.3.2 Project Schedule

The proposed schedule for the RI field activities and associated deliverables is summarized in Figure 6-1 of the RIWP (CH2M, 2018). The project schedule will be updated by the PM on a monthly basis, or more frequently, as needed. It should be noted that after the completion of the Phase 1 fieldwork, a Groundwater Modeling Quality Assurance Project Plan will be developed. Once the Groundwater Modeling QAPP is approved by the regulators, a detailed Groundwater Flow Model and Solute Transport Model will be developed using data from the Phase 1 fieldwork, supplemented with historical data as appropriate. Visualization output will be generated to capture the results of these models.

2.4 Quality Objectives and Criteria (A7)

2.4.1 Project Quality Objectives

Refer to Sections 2.4 and 5.1 of the RIWP for the overall project approach. Specific DQOs were considered independently through the EPA seven-step DQO process (EPA, 2006) to meet the data user's needs for each activity. Table 2-2 summarizes the OU-2 DQOs. The RIWP contains additional information regarding work activities and site details referenced in the DQOs (CH2M, 2018).

Reporting limits must be sufficient to achieve the objectives shown in Table 2-2. Reporting detection limits, regulatory limits, and other uses of the data were taken into consideration in selecting appropriate methods and laboratory reporting limits (RLs). Volatile organic compounds (VOCs), specifically PCE, trichloroethene, cis-1,2-dichloroethene, and vinyl chloride are the currently known constituents of potential concern (COPCs). 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. Semivolatile organic compounds (SVOCs), organochlorine pesticides, metals, and various general water quality and natural attenuation parameters are included in the sampling plans to provide additional data for the RI. The preliminary COPCs and characterization constituents determined from the DQO process with their RL requirements are listed in Tables 2-3 through 2-13. The selected methods are state-of-the-art and appropriate for this study. For most analytes, laboratory-specific method detection limits (MDLs) are expected to be below needed detection levels listed in Tables 2-3 through 2-13. Where sample-specific reporting limits are higher than needed limits, the project team will use MDLs, as needed and available, for project decisions.

Validated laboratory chemical data will be used. This applies to any laboratory contracted by either the U.S. Army Corps of Engineers (USACE) or the CH2M team to perform analyses. Laboratories must have an established program for data reduction, review, approval, and reporting as discussed following subsections.

Table 2-2. Data Quality Objectives
Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Principal Study Questions	Information Inputs	Spatial Study Boundaries	Analytical Approach	Performance or Acceptance Criteria	Plan for Obtaining Data
Overall Problem Statement: PCE-contaminated groundwater is presumed present beneath VHA property and is present in areas hydraulically downgradient of VHA property. PCE-contaminated groundwater was also drawn toward SLC-18 when the well was in operation. People could be exposed to the PCE-contaminated groundwater through (1) existing or new water supply wells including SLC-18 if it is brought back online,[a] or (2) vapor intrusion and direct-contact pathways associated with shallow-groundwater and springs in the ESS area (currently being managed as part of AOU-1).[b] Additional data are needed to characterize the hydrogeology and nature and extent of PCE contamination, to assess potential transport and exposure pathways and risks, and to allow development and detailed analysis of remedial alternatives during the subsequent feasibility study.					
Question 1: What is the source(s) of PCE plume identified by the 1998 EPA monitoring wells? Is there still sufficient mass of PCE in the vadose zone to act as an ongoing source of PCE in groundwater?	Historical documents and aerial photography for potential source areas Soil gas, soil, and groundwater VOC data Soil and soil gas screening levels applicable to the migration-to-groundwater pathway If more site-specific evaluation is needed, additional inputs could include: • Vadose zone lithological data • Vadose zone soil hydraulic properties data • Saturated zone hydraulic properties and groundwater flow data • Precipitation data	Vicinity of identified possible source areas including: • VHA Medical Center Building 7 and Sunnyside Sewer Line • Former Fort Douglas • Former Utah National Guard Vehicle Maintenance Facility • Former U.S. Forest Service Helicopter Pad	If soil gas data suggest the presence of PCE in vadose zone soil above soil gas screening levels, collect vadose zone soil samples. If vadose zone soil samples exceed migration-to-groundwater screening levels, evaluate whether more site-specific analysis, such as one-dimensional, numerical, unsaturated flow and transport modeling would support remedial decision making. If source area soil gas or soil data suggest that other pathways (soil direct exposure or vapor intrusion) could be significant, assess whether additional data are necessary to support human health risk assessment or remedial decision making.	Chemical analyses need to be sensitive enough for comparison to EPA soil screening levels for migration to groundwater for PCE and daughter products.	Identify potential source areas based on historic records, analytical data, and aerial photos. Design source investigations to target potential source areas or areas where data are inadequate to define the nature and extent of contamination. Collect sufficient data to delineate the nature and extent of source; conduct a baseline risk assessment and develop and evaluate remedial alternatives. Methods may include: • Soil gas sampling • Soil sampling via GeoProbe • Groundwater sampling via HydroPunch or monitoring wells
Question 2: What is the lateral and vertical extent of the PCE plume identified by the 1998 EPA monitoring wells? How far downgradient does the PCE plume extend?	• Groundwater sampling results from existing and new wells • MCLs or applicable screening levels for defining the extent of contamination • Hydrogeological data including lithology, samples from unsaturated and saturated intervals, and water levels	Proposed OU-2 RI field investigation area	Based on Phase 1 groundwater monitoring results and professional judgment, consider additional groundwater sampling locations if the interpolated contours of groundwater PCE above MCLs show data gaps and too much uncertainty.	Adequately spaced data (roughly 1,000 feet horizontally and 50 feet vertically) are required to delineate plume to MCLs. A network of monitoring wells is needed including: centerline wells to understand plume behavior along primary flow paths, perimeter wells to delineate and monitor plume stability, and sentinel wells to monitor for future plume expansion both horizontally and vertically. The installation of additional wells will be phased depending on available funds. Chemical analyses need to be sensitive enough for comparison to MCLs or applicable screening levels (Table 5-3 in the RIWP).	Develop a sufficient monitoring network to delineate plume horizontally and vertically, and monitor plume behavior and stability. Methods will include: • Shallow and deep multi-level monitoring wells installed with sonic drilling methods • Groundwater sampling via low-flow sampling • Hydrogeological data collection including soil lithology, depths of saturated intervals, and water levels
Question 3: Is the PCE and daughter products measured in the ESS related to the PCE contamination plume identified by the 1998 EPA monitoring wells?	• Groundwater sampling results from new wells upgradient of 1300 East • MCLs or applicable screening levels for defining the extent of contamination • Hydrogeological data including lithology, water levels, and sampling results	Area east of 1300 East, spanning from approximately Michigan Avenue to the south and 700 South to the north	Based on Phase 1 groundwater monitoring results, determine whether groundwater monitoring wells in this area contain PCE concentrations greater than the MCL or greater than the previously observed PCE concentrations in shallow groundwater west of 1300 East.	Adequately spaced data are required on the east side of 1300 East, perpendicular to the assumed direction of groundwater flow. The spacing should be generally proportional to the north-south extent of groundwater contamination west of 1300 East. Chemical analyses need to be sensitive enough for comparison to MCLs or applicable screening levels (Table 5-3 in the RIWP).	Develop a sufficient monitoring network across the north-south extent of the area upgradient of the known contamination on the west side of 1300 East. Methods will include: • Monitoring wellinstallation via sonic drilling methods • Groundwater sampling via low-flow sampling methods Collect hydrogeological data including soil lithology, depths of saturated intervals, and water levels

Table 2-2. Data Quality Objectives

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Principal Study Questions	Information Inputs	Spatial Study Boundaries	Analytical Approach	Performance or Acceptance Criteria	Plan for Obtaining Data
Overall Problem Statement: PCE-contaminated groundwater is presumed present beneath VHA property and is present in areas hydraulically downgradient of VHA property. PCE-contaminated groundwater was also drawn toward SLC-18 when the well was in operation. People could be exposed to the PCE-contaminated groundwater through (1) existing or new water supply wells including SLC-18 if it is brought back online,[a] or (2) vapor intrusion and direct-contact pathways associated with shallow-groundwater and springs in the ESS area (currently being managed as part of AOU-1).[b] Additional data are needed to characterize the hydrogeology and nature and extent of PCE contamination, to assess potential transport and exposure pathways and risks, and to allow development and detailed analysis of remedial alternatives during the subsequent feasibility study.					
Question 4: What hydrogeological features control PCE fate and transport? If the PCE plume identified by the 1998 EPA monitoring wells extends to ESS (refer to Question 3), what factors control the plume in fault zone/hillside? Does the entire plume discharge to the hillside or does some component continue deeper to the west?	• Lithology and hydrostratigraphy data (grain size distribution, fraction organic carbon, and extent and thickness of perching layers) • Structural geology (fault trace and orientation) • Interpretation of borehole geophysical data from new monitoring wells • Water levels and horizontal and vertical hydraulic gradients • Recharge and pumping history • Aquifer test results and estimated hydrogeological properties including transmissivity, hydraulic conductivity, storativity, and vertical and horizontal hydraulic gradients • Geochemistry data including major ion chemistry and stable isotopes • Previous groundwater modeling and new Phase 1 modeling	Proposed OU-2 RI field investigation area	Integrate information inputs into a sitewide CSM. If professional judgment shows too much uncertainty in specific locations or regarding specific factors, additional data collection will be considered.	Hydrogeological data collection and groundwater modeling conducted under the supervision of a qualified Utah Professional Geologist will be acceptable.	• Collect lithology and hydrostratigraphy data from new monitoring wells and assimilate information from existing wells • Aggregate pumping history information from major Salt Lake City and University of Utah production wells • Obtain available water level data from wells with pressure transducers. • Collect water level data from new and existing wells • Perform aquifer tests on selected wells to characterize hydraulic connectivity in area impacted by the PCE plume • Collect VOC, major ion, and stable isotope data from selected wells and surface water
Question 5: What is the nature of the hydraulic connection between the PCE plume and production wells (SLC-18, University of Utah wells, and Mount Olivet well)?	• Groundwater sampling results from existing and new wells • Lithology and hydrostratigraphy data (grain size distribution, fraction organic carbon, and extent and thickness of perching layers) • Structural geology (fault trace and orientation) • Interpretation of borehole geophysical data from new monitoring wells • Water levels and horizontal and vertical hydraulic gradients • Recharge and pumping history • Aquifer test results and estimated hydrogeological properties including transmissivity, hydraulic conductivity, storativity, and vertical and horizontal hydraulic gradients • Previous groundwater modeling and new Phase 1 modeling	Area between plume and production wells	If professional judgment shows gaps in understanding the connection between the plume and production wells, additional data collection will be considered. Upon development of the new groundwater flow model, calibrated to aquifer property data, evaluate whether operation of nearby production wells at rates consistent with historical operations could reasonably influence groundwater flow conditions at the site.	See Performance or Acceptance Criteria for Questions #2, #3, and #4.	See Plan for Obtaining Data for Questions #2, #3, and #4. In addition, pump testing using new monitoring wells may provide more information regarding the zone of influence of SLC-18.

Table 2-2. Data Quality Objectives

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Principal Study Questions	Information Inputs	Spatial Study Boundaries	Analytical Approach	Performance or Acceptance Criteria	Plan for Obtaining Data
Overall Problem Statement: PCE-contaminated groundwater is presumed present beneath VHA property and is present in areas hydraulically downgradient of VHA property. PCE-contaminated groundwater was also drawn toward SLC-18 when the well was in operation. People could be exposed to the PCE-contaminated groundwater through (1) existing or new water supply wells including SLC-18 if it is brought back online,[a] or (2) vapor intrusion and direct-contact pathways associated with shallow-groundwater and springs in the ESS area (currently being managed as part of AOU-1).[b] Additional data are needed to characterize the hydrogeology and nature and extent of PCE contamination, to assess potential transport and exposure pathways and risks, and to allow development and detailed analysis of remedial alternatives during the subsequent feasibility study.					
Question 6: Besides vapor intrusion in AOU-1, drinking water wells, or potential source-area soil and soil gas, are there other potential human or ecological exposure pathways?	Water sampling results from selected areas of Red Butte Creek	Red Butte Creek sampling locations, in the area where surface water has previously shown to be impacted by PCE, springs are present, and where Red Butte Creek is accessible at ground surface	Evaluate the overall site CSM to assess whether surface water exposure pathways could exist, and, if so, consider additional surface water data collection.	Chemical analyses need to be sensitive enough to determine whether surface water in Red Butte Creek has been impacted by the PCE identified by the EPA monitoring wells. For these purposes, chemical analyses need to be sensitive enough for comparison to MCLs or applicable screening levels (Table 5-3 in the RIWP).	Collect sufficient data to support human-health risk assessment related to OU-2 surface water. Methods could include: • Collection of surface water samples in Red Butte Creek
Question 7: Collect data to support possible remedial technologies, including MNA, hydraulic containment, and bioremediation. Determine which natural attenuation processes are operating, and estimate the rate of degradation of PCE and daughter products formed.	• See Information Inputs for source area(s) and nature and extent of contamination in Questions #1, #2, and #3 • See Information Inputs for geotechnical and hydrogeological data in Questions #2, #3, and #4 • PCE daughter product concentrations and concentration time-series • Reduction/oxidation geochemical data • Data supporting evaluation of natural attenuation • Biological data supporting the assessment of reductive dechlorination • Data supporting evaluation of potential remedial technologies	Entire project area	Evaluate data to assess potential remedial technologies, including the occurrence of natural attenuation processes and estimated degradation rates.	See Performance or Acceptance Criteria for Questions #1 through #5. In addition, a wide variety of definitive quantitative data and qualitative data are required to build lines of evidence for natural attenuation, as required by EPA MNA guidance.	Collect data regarding source area(s) and nature and extent of contamination, as summarized in Questions #1, #2, and #3 Collect geotechnical and geophysical data, as summarized in Questions #4 and #5 Collect data supporting evaluation of biological and abiotic attenuation processes. Methods may include: • PCE daughter product concentrations • Dissolved oxygen measurements • Oxidation-reduction potential measurements • Sulfate/sulfide, ferrous iron, concentrations of dissolved gases • Compound specific isotope analyses • Magnetic susceptibility tests

[a] The currently available information supports the conclusion that exposures to PCE-contaminated groundwater through the use of water-supply wells is controlled by Salt Lake City temporarily removing SLC-18 from service.
[b] Investigations, removal actions, and remedies associated with shallow groundwater within AOU-1 are being addressed separately under the FFA and will be incorporated by reference into OU-2 CERCLA documents.

Notes:
AOU = Accelerated Operable Unit
CERCLA = Comprehensive Environmental Response Compensation and Liability Act
CSM = conceptual site model
EPA = U.S. Environmental Protection Agency
ESS = East Side Springs
FFA = Federal Facilities Agreement
MCL = maximum contaminant level
MNA = monitored natural attenuation
OU = operable unit
PCE = tetrachloroethene
RI = remedial investigation
RIWP = remedial investigation work plan
VHA = Veterans Health Administration
VOC = volatile organic compound

Table 2-3. Project Laboratory – Target Analytes and Reporting Limits – Volatile Organic Compounds in Soil
Quality Assurance Project Plan, OU-2 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]	Lab RL (mg/kg)
1,1,1-Trichloroethane	71-55-6	SW8260C	Protection of Groundwater MCL-SSL (DAF=20)	1.4	0.005
1,1,2,2-Tetrachloroethane	79-34-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0006[b]	0.005
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	SW8260C	Protection of Groundwater SSL (DAF=20)	52	0.01
1,1,2-Trichloroethane	79-00-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.00026[b]	0.005
1,1-Dichloroethane	75-34-3	SW8260C	Protection of Groundwater SSL (DAF=20)	0.016	0.005
1,1-Dichloroethene	75-35-4	SW8260C	Protection of Groundwater MCL-SSL (DAF=20)	0.05	0.005
1,2,3-Trichlorobenzene	87-61-6	SW8260C	Protection of Groundwater SSL (DAF=20)	0.042	0.005
1,2,4-Trichlorobenzene	120-82-1	SW8260C	Protection of Groundwater SSL (DAF=20)	0.024	0.005
1,2,4-Trimethylbenzene	95-63-6	SW8260C	Protection of Groundwater SSL (DAF=20)	0.042	0.0025
1,3,5-Trimethylbenzene	108-67-8	SW8260C	Protection of Groundwater SSL (DAF=20)	0.34	0.0025
1,2-Dibromo-3-Chloropropane	96-12-8	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0000028[b]	0.01
1,2-Dibromoethane (EDB)	106-93-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.000042[b]	0.005
1,2-Dichlorobenzene	95-50-1	SW8260C	Protection of Groundwater SSL (DAF=20)	0.6	0.005
1,2-Dichloroethane	107-06-2	SW8260C	Protection of Groundwater SSL (DAF=20)	0.00096[b]	0.005
1,2-Dichloropropane	78-87-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0054	0.005
1,3-Dichlorobenzene	541-73-1	SW8260C	--	NA	0.005
1,4-Dichlorobenzene	106-46-7	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0092	0.005
2-Butanone (Methyl Ethyl Ketone)	78-93-3	SW8260C	Protection of Groundwater SSL (DAF=20)	2.4	0.01
2-Hexanone	591-78-6	SW8260C	Protection of Groundwater SSL (DAF=20)	0.018	0.01
4-Methyl-2-pentanone	108-10-1	SW8260C	Protection of Groundwater SSL (DAF=20)	2.8	0.01
Acetone	67-64-1	SW8260C	Protection of Groundwater SSL (DAF=20)	5.8	0.02
Benzene	71-43-2	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0046[b]	0.005

Table 2-3. Project Laboratory – Target Analytes and Reporting Limits – Volatile Organic Compounds in Soil
Quality Assurance Project Plan, OU-2 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]	Lab RL (mg/kg)
Bromochloromethane	74-97-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.042	0.005
Bromodichloromethane	75-27-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.00072[b]	0.005
Bromoform	75-25-2	SW8260C	Protection of Groundwater SSL (DAF=20)	0.017	0.005
Bromomethane	74-83-9	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0038[b]	0.01
Carbon Disulfide	75-15-0	SW8260C	Protection of Groundwater SSL (DAF=20)	0.48	0.005
Carbon Tetrachloride	56-23-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0036[b]	0.005
Chlorobenzene	108-90-7	SW8260C	Protection of Groundwater SSL (DAF=20)	0.11	0.005
Chloroethane	75-00-3	SW8260C	Protection of Groundwater SSL (DAF=20)	12	0.01
Chloroform	67-66-3	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0012[b]	0.005
Chloromethane	74-87-3	SW8260C	Protection of Groundwater SSL (DAF=20)	0.098	0.01
cis-1,2-Dichloroethene	156-59-2	SW8260C	Protection of Groundwater SSL (DAF=20)	0.022	0.005
Dibromochloromethane	124-48-1	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0046[b]	0.005
Dichlorodifluoromethane (Freon 12)	75-71-8	SW8260C	Protection of Groundwater SSL (DAF=20)	0.6	0.01
Ethylbenzene	100-41-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.034	0.005
Isopropylbenzene (Cumene)	98-82-8	SW8260C	Protection of Groundwater SSL (DAF=20)	1.5	0.005
Methyl acetate	79-20-9	SW8260C	Protection of Groundwater SSL (DAF=20)	8.2	0.01
Methyl Tert-Butyl Ether	1634-04-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.064	0.005
Methylene chloride	75-09-2	SW8260C	Protection of Groundwater MCL-SSL (DAF=20)	0.026	0.01
m,p-Xylene	108-38-3 and 106-42-3	SW8260C	Protection of Groundwater SSL (DAF=20)	0.38	0.01
o-Xylene	95-47-6	SW8260C	Protection of Groundwater SSL (DAF=20)	0.38	0.005
Styrene	100-42-5	SW8260C	Protection of Groundwater MCL-SSL (DAF=20)	2.2	0.005
Tetrachloroethene	127-18-4	SW8260C	Protection of Groundwater MCL-SSL (DAF=20)	0.036	0.005

Table 2-3. Project Laboratory – Target Analytes and Reporting Limits – Volatile Organic Compounds in Soil
Quality Assurance Project Plan, OU-2 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]	Lab RL (mg/kg)
Toluene	108-88-3	SW8260C	Protection of Groundwater SSL (DAF=20)	1.5	0.005
Total 1,3-dichloro propene (cis- & trans-)	542-75-6	SW8260C	Protection of Groundwater SSL (DAF=20)	0.0034[b]	0.01
trans-1,2-Dichloroethene	156-60-5	SW8260C	Protection of Groundwater SSL (DAF=20)	0.22	0.005
Trichloroethene	79-01-6	SW8260C	Protection of Groundwater SSL (DAF=20)	0.002[b]	0.005
Trichlorofluoromethane (Freon 11)	75-69-4	SW8260C	Protection of Groundwater SSL (DAF=20)	6.6	0.01
Vinyl Acetate	108-05-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.17	0.005
Vinyl Chloride	75-01-4	SW8260C	Protection of Groundwater SSL (DAF=20)	0.00013 [b]	0.005

[a] Lowest of: (1) RSLs for residential exposure, (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 0.1 were used (November 2017).
[b] Due to 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.

Notes:
CAS = Chemical Abstracts Service
DAF = dilution attenuation factor
MCL = maximum contaminant level
mg/kg = milligram(s) per kilogram
RL = reporting limit
RSL = regional screening level
SSL = soil screening level

Table 2-4. Project Laboratory – Target Analytes and Reporting Limits – Semivolatile Organic Compounds in Soil
Quality Assurance Project Plan, OU-2 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]	Lab RL (mg/kg)
1,1'-Biphenyl	92-52-4	SW8270D	Protection of Groundwater SSL (DAF=20)	0.017[b]	0.333
1,2,4,5-Tetrachlorobenzene	95-94-3	SW8270D	Protection of Groundwater SSL (DAF=20)	0.016[b]	0.333
1,4-Dioxane	123-91-1	SW8270D	Protection of Groundwater SSL (DAF=20)	0.0019[b]	0.0333
2,3,4,6-Tetrachlorophenol	58-90-2	SW8270D	Protection of Groundwater SSL (DAF=20)	0.36	0.333
2,4,5-Trichlorophenol	95-95-4	SW8270D	Protection of Groundwater SSL (DAF=20)	8	0.333
2,4,6-Trichlorophenol	88-06-2	SW8270D	Protection of Groundwater SSL (DAF=20)	0.024[b]	0.333
2,4-Dichlorophenol	120-83-2	SW8270D	Protection of Groundwater SSL (DAF=20)	0.046[b]	0.333
2,4-Dimethylphenol	105-67-9	SW8270D	Protection of Groundwater SSL (DAF=20)	0.84[b]	1.33
2,4-Dinitrophenol	51-28-5	SW8270D	Protection of Groundwater SSL (DAF=20)	0.088[b]	3.33
2,4-Dinitrotoluene	121-14-2	SW8270D	Protection of Groundwater SSL (DAF=20)	0.0064[b]	0.333
2,6-Dinitrotoluene	606-20-2	SW8270D	Protection of Groundwater SSL (DAF=20)	0.0013[b]	0.333
2-Chloronaphthalene	91-58-7	SW8270D	Protection of Groundwater SSL (DAF=20)	7.8	0.333
2-Chlorophenol	95-57-8	SW8270D	Protection of Groundwater SSL (DAF=20)	0.18	0.333
2-Methylnaphthalene	91-57-6	SW8270D	Protection of Groundwater SSL (DAF=20)	0.38	0.00667
2-Methylphenol	95-48-7	SW8270D	Protection of Groundwater SSL (DAF=20)	1.5	0.333
2-Nitroaniline	88-74-4	SW8270D	Protection of Groundwater SSL (DAF=20)	0.16[b]	1.33
3-Nitroaniline	99-09-2	SW8270D	--	NA	1.33
2-Nitrophenol	88-75-5	SW8270D	--	NA	0.333
3,3'-Dichlorobenzidine	91-94-1	SW8270D	Protection of Groundwater SSL (DAF=20)	0.016[b]	0.333
4,6-Dinitro-2-methylphenol	534-52-1	SW8270D	Protection of Groundwater SSL (DAF=20)	0.0052[b]	3.33
4-Bromophenyl Phenyl Ether	101-55-3	SW8270D	--	NA	0.333
4-Chloroaniline	106-47-8	SW8270D	EPA RSL	0.0032[b]	0.333
4-Chloro-3-methylphenol	59-50-7	SW8270D	Protection of Groundwater SSL (DAF=20)	3.4	0.333
4-Chlorophenyl Phenyl Ether	7005-72-3	SW8270D	--	NA	0.333

Table 2-4. Project Laboratory – Target Analytes and Reporting Limits – Semivolatile Organic Compounds in Soil
Quality Assurance Project Plan, OU-2 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]	Lab RL (mg/kg)
4-Methylphenol (3/4-Methylphenol)	106-44-5	SW8270D	Protection of Groundwater SSL (DAF=20)	3	0.333
4-Nitroaniline	100-01-6	SW8270D	Protection of Groundwater SSL (DAF=20)	0.032[b]	1.33
4-Nitrophenol	100-02-7	SW8270D	--	NA	1.33
Acenaphthene	83-32-9	SW8270D	Protection of Groundwater SSL (DAF=20)	11	0.00667
Acenaphthylene	208-96-8	SW8270D	--	NA	0.00667
Acetophenone	98-86-2	SW8270D	Protection of Groundwater SSL (DAF=20)	1.2	0.333
Anthracene	120-12-7	SW8270D	Protection of Groundwater SSL (DAF=20)	120	0.00667
Atrazine	1912-24-9	SW8270D	Protection of Groundwater SSL (DAF=20)	0.004[b]	0.333
Benzaldehyde	100-52-7	SW8270D	Protection of Groundwater SSL (DAF=20)	0.082[b]	0.333
Benzo(a)anthracene	56-55-3	SW8270D	Protection of Groundwater SSL (DAF=20)	0.22	0.00667
Benzo(a)pyrene	50-32-8	SW8270D	EPA RSL	0.11	0.00667
Benzo(b)fluoranthene	205-99-2	SW8270D	EPA RSL	1.1	0.00667
Benzo(g,h,i)perylene	191-24-2	SW8270D	--	NA	0.00667
Benzo(k)fluoranthene	207-08-9	SW8270D	EPA RSL	11	0.00667
Bis(2-chloroethoxy)methane	111-91-1	SW8270D	Protection of Groundwater SSL (DAF=20)	0.026[b]	0.333
Bis(2-chloroethyl)ether	111-44-4	SW8270D	Protection of Groundwater SSL (DAF=20)	0.000072[b]	0.333
Bis(2-chloroisopropyl)ether	108-60-1	SW8270D	Protection of Groundwater SSL (DAF=20)	0.52	0.333
Bis(2-Ethylhexyl)Phthalate	117-81-7	SW8270D	Protection of Groundwater SSL (DAF=20)	26	0.333
Butyl Benzyl Phthalate	85-68-7	SW8270D	Protection of Groundwater SSL (DAF=20)	4.8	0.333
Caprolactam	105-60-2	SW8270D	Protection of Groundwater SSL (DAF=20)	5	0.333
Carbazole	86-74-8	SW8270D	--	NA	0.333
Chrysene	218-01-9	SW8270D	EPA RSL	110	0.00667
Dibenz(a,h)anthracene	53-70-3	SW8270D	EPA RSL	0.11	0.00667
Dibenzofuran	132-64-9	SW8270D	Protection of Groundwater SSL (DAF=20)	0.3	0.333
Diethyl Phthalate	84-66-2	SW8270D	Protection of Groundwater SSL (DAF=20)	12	0.333
Dimethyl Phthalate	131-11-3	SW8270D	--	NA	0.333

Table 2-4. Project Laboratory – Target Analytes and Reporting Limits – Semivolatile Organic Compounds in Soil
Quality Assurance Project Plan, OU-2 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]	Lab RL (mg/kg)
Di-n-Butyl Phthalate	84-74-2	SW8270D	Protection of Groundwater SSL (DAF=20)	4.6	0.333
Di-n-octyl Phthalate	117-84-0	SW8270D	EPA RSL	63	0.333
Fluoranthene	206-44-0	SW8270D	Protection of Groundwater SSL (DAF=20)	180	0.00667
Fluorene	86-73-7	SW8270D	Protection of Groundwater SSL (DAF=20)	11	0.00667
Hexachlorobenzene	118-74-1	SW8270D	Protection of Groundwater SSL (DAF=20)	0.0024[b]	0.333
Hexachlorobutadiene	87-68-3	SW8270D	Protection of Groundwater SSL (DAF=20)	0.0054[b]	0.333
Hexachlorocyclopentadiene	77-47-4	SW8270D	Protection of Groundwater SSL (DAF=20)	0.0026[b]	0.333
Hexachloroethane	67-72-1	SW8270D	Protection of Groundwater SSL (DAF=20)	0.004[b]	0.333
Indeno(1,2,3-cd)pyrene	193-39-5	SW8270D	EPA RSL	1.1	0.00667
Isophorone	78-59-1	SW8270D	Protection of Groundwater SSL (DAF=20)	0.52	0.333
Naphthalene	91-20-3	SW8270D	Protection of Groundwater SSL (DAF=20)	0.011	0.00667
Nitrobenzene	98-95-3	SW8270D	Protection of Groundwater SSL (DAF=20)	0.0018[b]	0.333
N-Nitroso-di-n-propylamine	621-64-7	SW8270D	Protection of Groundwater SSL (DAF=20)	0.00016[b]	0.333
N-Nitrosodiphenylamine	86-30-6	SW8270D	Protection of Groundwater SSL (DAF=20)	1.3	0.333
Pentachlorophenol	87-86-5	SW8270D	Protection of Groundwater SSL (DAF=20)	0.0011[b]	1.33
Phenanthrene	85-01-8	SW8270D	--	NA	0.00667
Phenol	108-95-2	SW8270D	Protection of Groundwater SSL (DAF=20)	6.6	0.333
Pyrene	129-00-0	SW8270D	Protection of Groundwater SSL (DAF=20)	26	0.00667

[a] Lowest of: (1) RSLs for residential exposure, and (2) SSLs for groundwater protection using and DAF of 20. RSLs corresponding to an excessive lifetime cancer risk of 1×10^{-6} and a hazard quotient of 0.1 were used (November 2017).
[b] Due to 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
mg/kg = milligram(s) per kilogram
RL = reporting limit
RSL = regional screening level
SSL = soil screening level

Table 2-5. Project Laboratory – Target Analytes and Reporting Limits – Metals in Soil

Quality Assurance Project Plan, OU-2 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]	Lab RL (mg/kg)
Aluminum	7429-90-5	SW6010C	EPA RSL	7,700	10
Antimony	7440-36-0	SW6010C	Protection of Groundwater SSL (DAF=20)	0.7	0.5
Arsenic	7440-38-2	SW6010C	Protection of Groundwater SSL (DAF=20)	0.03[b]	0.5
Barium	7440-39-3	SW6010C	EPA RSL	320	2
Beryllium	7440-41-7	SW6010C	EPA RSL	16	0.25
Cadmium	7440-43-9	SW6010C	Protection of Groundwater SSL (DAF=20)	1.4	0.25
Calcium	7440-70-2	SW6010C	--	NA	250
Chromium	7440-47-3	SW6010C	Protection of Groundwater MCL-SSL (DAF=20)	3,600,000	0.5
Cobalt	7440-48-4	SW6010C	EPA RSL	0.54[b]	0.625
Copper	7440-50-8	SW6010C	Protection of Groundwater SSL (DAF=20)	56	0.5
Iron	7439-89-6	SW6010C	Protection of Groundwater SSL (DAF=20)	700	7.5
Lead	7439-92-1	SW6010C	Protection of Groundwater MCL-SSL (DAF=20)	280	0.25
Magnesium	7439-95-4	SW6010C	--	NA	250
Manganese	7439-96-5	SW6010C	Protection of Groundwater SSL (DAF=20)	56	0.75
Mercury	7487-94-7	SW7471B	Protection of Groundwater SSL (DAF=20)	2.3	0.033
Nickel	7440-02-0	SW6010C	Protection of Groundwater SSL (DAF=20)	52	0.5
Potassium	7440-09-7	SW6010C	--	NA	250
Selenium	7782-49-2	SW6010C	Protection of Groundwater MCL-SSL (DAF=20)	1.0	0.5
Silver	7440-22-4	SW6010C	Protection of Groundwater SSL (DAF=20)	1.6	0.5
Sodium	7440-23-5	SW6010C	--	NA	250
Thallium	7440-28-0	SW6010C	Protection of Groundwater SSL (DAF=20)	0.028[b]	0.4
Vanadium	7440-62-2	SW6010C	EPA RSL	39	0.625
Zinc	7440-66-6	SW6010C	Protection of Groundwater SSL (DAF=20)	740	1

[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 0.1 were used (November 2017).

[b] Due to 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
mg/kg = milligram(s) per kilogram
NA = not applicable
RL = reporting limit
RSL = regional screening level
SSL = soil screening level

Table 2-6. Project Laboratory – Target Analytes and Reporting Limits – Organochlorine Pesticides in Soil
Quality Assurance Project Plan, OU-2 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]	Lab RL (mg/kg)
4,4'-DDD	72-54-8	SW8081B	Protection of Groundwater MCL-SSL (DAF=20)	0.03	0.00067
4,4'-DDE	72-55-9	SW8081B	Protection of Groundwater SSL (DAF=20)	0.22	0.00067
4,4'-DDT	50-29-3	SW8081B	Protection of Groundwater SSL (DAF=20)	1.5	0.00067
Aldrin	309-00-2	SW8081B	Protection of Groundwater MCL-SSL (DAF=20)	0.003	0.00067
BHC, alpha-	319-84-6	SW8081B	Protection of Groundwater SSL (DAF=20)	0.00084	0.00067
BHC, beta-	319-85-7	SW8081B	Protection of Groundwater SSL (DAF=20)	0.003	0.00067
BHC, delta-	319-86-8	SW8081B	--	NA	0.00067
BHC, gamma- (Lindane)	58-89-9	SW8081B	Protection of Groundwater SSL (DAF=20)	0.0048	0.00067
Chlordane, cis-	5103-71-9	SW8081B	Protection of Groundwater SSL (DAF=20)	0.054[b]	0.00067
Chlordane, trans-	5103-74-2	SW8081B	Protection of Groundwater SSL (DAF=20)	0.054[b]	0.00067
Dieldrin	60-57-1	SW8081B	Protection of Groundwater SSL (DAF=20)	0.0014	0.00067
Endosulfan I	959-98-8	SW8081B	Protection of Groundwater SSL (DAF=20)	2.8[c]	0.00067
Endosulfan II	33213-65-9	SW8081B	Protection of Groundwater SSL (DAF=20)	2.8[c]	0.00067
Endosulfan sulfate	1031-07-8	SW8081B	Protection of Groundwater SSL (DAF=20)	2.8[c]	0.00067
Endrin	72-20-8	SW8081B	Protection of Groundwater SSL (DAF=20)	0.18	0.00067
Endrin aldehyde	7421-93-4	SW8081B	--	NA	0.00067
Endrin ketone	53494-70-5	SW8081B	--	NA	0.00067
Heptachlor	76-44-8	SW8081B	Protection of Groundwater SSL (DAF=20)	0.0024	0.00067
Heptachlor epoxide	1024-57-3	SW8081B	Protection of Groundwater MCL-SSL (DAF=20)	0.00056[d]	0.00067
Methoxychlor	72-43-5	SW8081B	Protection of Groundwater SSL (DAF=20)	4.0	0.00067
Toxaphene	8001-35-2	SW8081B	Protection of Groundwater SSL (DAF=20)	0.22	0.033

[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 0.1 were used (November 2017).

[b] Screening level is equivalent to value listed for Technical Chlordane (CAS#12789-03-6)

[c] Screening level is equivalent to value listed for Endosulfan (CAS# 115-29-7)

[d] Due to 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:
BHC = benzene hexachloride
CAS = Chemical Abstracts Service
DAF = dilution attenuation factor
DDD = dichlorodiphenyldichloroethane
DDE = dichlorodiphenyldichloroethene
DDT = dichlorodiphenyltrichloroethane
MCL = maximum contaminant level
mg/kg = milligram(s) per kilogram
NA = not applicable
RL = reporting limit
RSL = regional screening level
SSL = soil screening level

Table 2-7. Project Laboratory – Target Analytes and Reporting Limits – Geotechnical Parameters in Soil
Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Lowest Screening Level Value (mg/kg)	Lab RL
FOC	NA	ASTM D2974	--	NA	0.500 mg/kg
Magnetic Susceptibility	NA	Microbial Insights Laboratory Method	--	NA	1×10^{-9} m^3/kg
Sieve Analysis	NA	ASTM D6913	--	NA	NA
Dry Bulk Soil Density	NA	ASTM D2937	--	NA	NA
Hydrometer	NA	ASTM D422a	--	NA	NA
USCS Soil Classification	NA	ASTM D2487	--	NA	NA
Atterberg Limits	NA	ASTM D4318	--	NA	NA
Gradation	NA	ASTM D1140	--	NA	NA
Vertical Permeability	NA	ASTM D2434	--	NA	NA
Moisture Content	NA	ASTM D2216	--	NA	NA

Notes:
CAS = Chemical Abstracts Service
FOC = fraction of organic carbon
m^3/kg = cubic meter(s) per kilogram
mg/kg = milligram(s) per kilogram
NA = not applicable
RL = reporting limit
USCS = Unified Soil Classification System

Table 2-8. Project Laboratory – Target Analytes and Reporting Limits – Volatile Organic Compounds in Soil Gas
Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Soil Gas Method	Screening Level[a]	Lowest Screening Level Value (µg/m³)[a]	Lab RL (µg/m³)
Laboratory Analytical Parameters					
1,1,1-Trichloroethane	71-55-6	TO-15	VISL	17,000	1.64
1,1,2,2-Tetrachloroethane	79-34-5	TO-15	VISL	1.6	2.75
1,1,2-Trichloroethane	79-00-5	TO-15	VISL	0.7[b]	2.18
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	TO-15	VISL	10,000	3.07
1,1-Dichloroethane	75-34-3	TO-15	VISL	60	1.21
1,1-Dichloroethene	75-35-4	TO-15	VISL	700	3.17
1,2,4-Trimethylbenzene	95-63-6	TO-15	VISL	24	3.93
1,2-Dibromoethane	106-93-4	TO-15	VISL	0.16[b]	6.15
1,2-Dichlorobenzene	95-50-1	TO-15	VISL	700	2.40
1,2-Dichloroethane	107-06-2	TO-15	VISL	3.7	3.24
1,2-Dichloropropane	78-87-5	TO-15	VISL	9.3	1.85
1,2-Dichlorotetrafluoethane (Freon 114)	76-14-2	TO-15	--	NA	0.400
1,3,5-Trimethylbenzene	108-67-8	TO-15	VISL	85,000	1.97
1,3-Butadiene	106-99-0	TO-15	VISL	3.1[b]	21.3
1,3-Dichlorobenzene	541-73-1	TO-15	--	NA	2.40
1,4-Dichlorobenzene	106-46-7	TO-15	VISL	8.7	2.40
1,4-Dioxane	123-91-1	TO-15	VISL	1.8[c]	2.88
2-Butanone (Methyl Ethyl Ketone)	78-93-3	TO-15	VISL	17,000	2.36
2-Hexanone	591-78-6	TO-15	VISL	100	1.64
4-Ethyltoluene	622-96-8	TO-15	VISL	--	1.97
4-Methyl-2-pentanone	108-10-1	TO-15	VISL	10,000	1.64
Acetone	67-64-1	TO-15	VISL	41,000	11.9
Benzene	71-43-2	TO-15	VISL	12	1.28
Bromodichloromethane	75-27-4	TO-15	VISL	2.5	2.01
Bromoform	75-25-2	TO-15	VISL	87	4.13
Bromomethane	74-83-9	TO-15	VISL	17	3.11
Carbon disulfide	75-15-0	TO-15	VISL	2400	2.49
Carbon tetrachloride	56-23-5	TO-15	VISL	16	5.03
Chlorobenzene	108-90-7	TO-15	VISL	170	1.38
Chloroethane	75-00-3	TO-15	VISL	33,000	2.11
Chloroform	67-66-3	TO-15	VISL	4	1.46
Chloromethane	74-87-3	TO-15	VISL	310	1.65
cis-1,2-Dichloroethene	156-59-2	TO-15	VISL	12,000	1.59
cis-1,3-Dichloropropene	542-75-6	TO-15	VISL	23	1.82
Cyclohexane	110-82-7	TO-15	VISL	21,000	1.38
Dibromochloromethane	124-48-1	TO-15	VISL	550	3.41
Dichlorodifluoromethane (Freon 12)	75-71-8	TO-15	VISL	330	1.98

Table 2-8. Project Laboratory – Target Analytes and Reporting Limits – Volatile Organic Compounds in Soil Gas
Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Soil Gas Method	Screening Level[a]	Lowest Screening Level Value (µg/m³)[a]	Lab RL (µg/m³)
Ethyl Acetate	141-78-6	TO-15	VISL	240	1.08
Ethylbenzene	100-41-4	TO-15	VISL	37	1.74
n-Heptane	142-82-5	TO-15	VISL	--	3.28
n-Hexane	110-54-3	TO-15	VISL	2,400	2.82
m,p-Xylene	108-38-3 and 106-42-3	TO-15	VISL	330	3.47
Methylene Chloride	75-09-2	TO-15	VISL	2,100	1.39
Methyl tert-butyl ether	1634-04-4	TO-15	VISL	370	2.88
o-Xylene	95-47-6	TO-15	VISL	330	1.74
Styrene	100-42-5	TO-15	VISL	33,000	1.70
Tetrachloroethene	127-18-4	TO-15	VISL	140	2.71
Tetrahydrofuran	109-99-9	TO-15	VISL	7,000	2.36
Toluene	108-88-3	TO-15	VISL	17,000	1.51
trans-1,2-Dichloroethene	156-60-5	TO-15	VISL	270,000	1.59
trans-1,3-Dichloropropene	542-75-6	TO-15	VISL	23	1.82
Trichloroethene	79-01-6	TO-15	VISL	7	2.15
Trichlorofluoromethane (Freon 11)	75-69-4	TO-15	VISL	41,000,000	2.25
Vinyl Acetate	108-05-4	TO-15	VISL	700	2.82
Vinyl Chloride	75-01-4	TO-15	VISL	5.7	1.02
Field Screening Parameters					
cis-1,2-Dichloroethene	156-59-2	HAPSITE	$SGSL_{pgw}$	120,000	2.0
Tetrachloroethene	127-18-4	HAPSITE	VISL	140	0.69
Trichloroethene	79-01-6	HAPSITE	VISL	7	0.55

[a] Residential VISLs using an attenuation factor of 0.1. VISLs corresponding to an excessive lifetime cancer risk of 1E-06 and a hazard quotient of 0.1 were used.
[b] Due to 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.
[c] Due to 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. The low VISL for 1,4-dioxane is based on protection of groundwater quality and was derived using conservative, default, simplifying assumptions, as described in Section 5.2.1 of the remedial investigation work plan. Soil gas screening would be used in a source investigation in which the RL would be an acceptable limit.

Notes:
µg/m³ = microgram(s) per cubic meter
COPC = constituent of potential concern
RL= reporting limit
$SGSL_{pgw}$ = soil gas equilibrium concentration - protection of groundwater
VISL = vapor intrusion screening level

Table 2-9. Project Laboratory – Target Analytes and Reporting Limits – Volatile Organic Compounds in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (µg/L)[a]	Lab RL (µg/L)
Laboratory Analytical Parameters					
1,1,1-Trichloroethane	71-55-6	SW8260C	EPA MCL	200	1.00
1,1,2,2-Tetrachloroethane	79-34-5	SW8260C	EPA RSL	0.076[b]	1.00
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	SW8260C	EPA RSL	1,000	2.00
1,1,2-Trichloroethane	79-00-5	SW8260C	EPA MCL	5	1.00
1,1-Dichloroethane	75-34-3	SW8260C	EPA RSL	2.8	1.00
1,1-Dichloroethene	75-35-4	SW8260C	EPA MCL	7	1.00
1,2,3-Trichlorobenzene	87-61-6	SW8260C	EPA RSL	0.7[b]	2.00
1,2,4-Trichlorobenzene	120-82-1	SW8260C	EPA MCL	70	2.00
1,2,4-Trimethylbenzene	95-63-6	SW8260C	EPA RSL	1.5	0.5
1,3,5-Trimethylbenzene	108-67-8	SW8260C	EPA RSL	12	0.5
1,2-Dibromo-3-Chloropropane	96-12-8	SW8260C	EPA MCL	0.2[b]	2.00
1,2-Dibromoethane	106-93-4	SW8260C	EPA MCL	0.05[b]	1.00
1,2-Dichlorobenzene	95-50-1	SW8260C	EPA MCL	600	1.00
1,2-Dichloroethane	107-06-2	SW8260C	EPA MCL	5	1.00
1,2-Dichloropropane	78-87-5	SW8260C	EPA MCL	5	1.00
1,3-Dichlorobenzene	541-73-1	SW8260C	--	NA	1.00
1,4-Dichlorobenzene	106-46-7	SW8260C	EPA MCL	75	1.00
2-Butanone (Methyl Ethyl Ketone)	78-93-3	SW8260C	EPA RSL	560	10.0
2-Hexanone	591-78-6	SW8260C	EPA RSL	3.8[b]	5.00
4-Methyl-2-pentanone	108-10-1	SW8260C	EPA RSL	630	5.00
Acetone	67-64-1	SW8260C	EPA RSL	1,400	10.0
Benzene	71-43-2	SW8260C	EPA MCL	5	1.00
Bromochloromethane	74-97-5	SW8260C	EPA RSL	8.3	1.00
Bromodichloromethane	75-27-4	SW8260C	EPA MCL	80	1.00
Bromoform	75-25-2	SW8260C	EPA MCL	80	1.00
Bromomethane	74-83-9	SW8260C	EPA RSL	0.75	2.00
Carbon Disulfide	75-15-0	SW8260C	EPA RSL	81	1.00
Carbon Tetrachloride	56-23-5	SW8260C	EPA MCL	5	1.00
Chlorobenzene	108-90-7	SW8260C	EPA MCL	100	1.00
Chloroethane	75-00-3	SW8260C	EPA RSL	2,100	2.00
Chloroform	67-66-3	SW8260C	EPA MCL	80	1.00
Chloromethane	74-87-3	SW8260C	EPA RSL	19	1.00
cis-1,2-Dichloroethene	156-59-2	SW8260C	EPA MCL	70	1.00
Dibromochloromethane	124-48-1	SW8260C	EPA MCL	80	1.00
Dichlorodifluoromethane (Freon 12)	75-71-8	SW8260C	EPA RSL	20	2.00
Ethylbenzene	100-41-4	SW8260C	EPA MCL	700	1.00

Table 2-9. Project Laboratory – Target Analytes and Reporting Limits – Volatile Organic Compounds in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (µg/L)[a]	Lab RL (µg/L)
Isopropylbenzene (Cumene)	98-82-8	SW8260C	EPA RSL	45	1.00
Methyl acetate	79-20-9	SW8260C	EPA RSL	2,000	2.00
Methyl Tert-Butyl Ether	1634-04-4	SW8260C	EPA RSL	14	1.00
Methylene chloride	75-09-2	SW8260C	EPA MCL	5	2.00
m,p-Xylene	108-38-3 and 106-42-3	SW8260C	EPA RSL	19	2.00
o-Xylene	95-47-6	SW8260C	EPA RSL	19	1.00
Styrene	100-42-5	SW8260C	EPA MCL	100	1.00
Tetrachloroethene (PCE)	127-18-4	SW8260C	EPA MCL	5	1.00
Toluene	108-88-3	SW8260C	EPA MCL	1,000	1.00
Total 1,3-dichloro propene (cis- & trans-)	542-75-6	SW8260C	EPA RSL	0.47[b]	2.00
trans-1,2-Dichloroethene	156-60-5	SW8260C	EPA MCL	100	1.00
Trichloroethene	79-01-6	SW8260C	EPA MCL	5	1.00
Trichlorofluoromethane (Freon 11)	75-69-4	SW8260C	EPA RSL	520	2.00
Vinyl Acetate	108-05-4	SW8260C	EPA RSL	41	2.5
Vinyl Chloride	75-01-4	SW8260C	EPA MCL	2	1.00
Field Screening Parameters					
cis-1,2-Dichloroethene	156-59-2	HAPSITE	EPA MCL	70	5.0
Tetrachloroethene	127-18-4	HAPSITE	EPA MCL	5	5.0
Trichloroethene	79-01-6	HAPSITE	EPA MCL	5	5.0

[a] If a 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 0.1 were used (November 2017).

[b] Due to 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 = microgram(s) per liter
CAS = Chemical Abstracts Service
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level
RL = reporting limit
RSL = regional screening level

Table 2-10. Project Laboratory – Target Analytes and Reporting Limits – Semivolatile Organic Compounds in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (µg/L)[a]	Lab RL (µg/L)
1,1'-Biphenyl	92-52-4	SW8270D	EPA RSL	0.083[b]	5.0
1,2,4,5-Tetrachlorobenzene	95-94-3	SW8270D	EPA RSL	0.17[b]	5.0
1,4-Dioxane	123-91-1	SW8270D	EPA RSL	0.46	0.2
2,3,4,6-Tetrachlorophenol	58-90-2	SW8270D	EPA RSL	24	10
2,4,5-Trichlorophenol	95-95-4	SW8270D	EPA RSL	120	5.0
2,4,6-Trichlorophenol	88-06-2	SW8270D	EPA RSL	1.2[b]	5.0
2,4-Dichlorophenol	120-83-2	SW8270D	EPA RSL	4.6[b]	5.0
2,4-Dimethylphenol	105-67-9	SW8270D	EPA RSL	36	5.0
2,4-Dinitrophenol	51-28-5	SW8270D	EPA RSL	3.9[b]	40
2,4-Dinitrotoluene	121-14-2	SW8270D	EPA RSL	0.24[b]	5.0
2,6-Dinitrotoluene	606-20-2	SW8270D	EPA RSL	0.049[b]	5.0
2-Chloronaphthalene	91-58-7	SW8270D	EPA RSL	75	5.0
2-Chlorophenol	95-57-8	SW8270D	EPA RSL	9.1	5.0
2-Methylnaphthalene	91-57-6	SW8270D	EPA RSL	3.6[b]	5.0
2-Methylphenol	95-48-7	SW8270D	EPA RSL	93	5.0
2-Nitroaniline	88-74-4	SW8270D	EPA RSL	19	20
3-Nitroaniline	99-09-2	SW8270D	--	NA	20
2-Nitrophenol	88-75-5	SW8270D	--	NA	5.0
3,3'-Dichlorobenzidine	91-94-1	SW8270D	EPA RSL	0.13[b]	5.0
4,6-Dinitro-2-methylphenol	534-52-1	SW8270D	EPA RSL	0.15[b]	40
4-Bromophenyl Phenyl Ether	101-55-3	SW8270D	--	NA	5.0
4-Chloroaniline	106-47-8	SW8270D	EPA RSL	0.37[b]	5.0
4-Chloro-3-methylphenol	59-50-7	SW8270D	EPA RSL	140	5.0
4-Chlorophenyl Phenyl Ether	7005-72-3	SW8270D	--	NA	5.0
4-Methylphenol (3/4-Methylphenol)	106-44-5	SW8270D	EPA RSL	190	5.0
4-Nitroaniline	100-01-6	SW8270D	EPA RSL	3.8[b]	20
4-Nitrophenol	100-02-7	SW8270D	--	NA	20
Acenaphthene	83-32-9	SW8270D	EPA RSL	53	5.0
Acenaphthylene	208-96-8	SW8270D	--	NA	5.0
Acetophenone	98-86-2	SW8270D	EPA RSL	190	5.0
Anthracene	120-12-7	SW8270D	EPA RSL	180	5.0
Atrazine	1912-24-9	SW8270D	EPA MCL	3[b]	5.0
Benzaldehyde	100-52-7	SW8270D	EPA RSL	19	5.0
Benzo(a)anthracene	56-55-3	SW8270D	EPA RSL	0.03[b]	5.0
Benzo(a)pyrene	50-32-8	SW8270D	EPA MCL	0.2[b]	5.0
Benzo(b)fluoranthene	205-99-2	SW8270D	EPA RSL	0.25[b]	5.0
Benzo(g,h,i)perylene	191-24-2	SW8270D	--	NA	5.0
Benzo(k)fluoranthene	207-08-9	SW8270D	EPA RSL	2.5[b]	5.0

Table 2-10. Project Laboratory – Target Analytes and Reporting Limits – Semivolatile Organic Compounds in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (µg/L)[a]	Lab RL (µg/L)
Bis(2-chloroethoxy)methane	111-91-1	SW8270D	EPA RSL	5.9	5.0
Bis(2-chloroethyl)ether	111-44-4	SW8270D	EPA RSL	0.014[b]	5.0
Bis(2-chloroisopropyl)ether	108-60-1	SW8270D	EPA RSL	71	5.0
Bis(2-Ethylhexyl)Phthalate	117-81-7	SW8270D	EPA MCL	6	5.0
Butyl Benzyl Phthalate	85-68-7	SW8270D	EPA RSL	16	5.0
Caprolactam	105-60-2	SW8270D	EPA RSL	990	5.0
Carbazole	86-74-8	SW8270D	--	NA	5.0
Chrysene	218-01-9	SW8270D	EPA RSL	25	5.0
Dibenz(a,h)anthracene	53-70-3	SW8270D	EPA RSL	0.025[b]	5.0
Dibenzofuran	132-64-9	SW8270D	EPA RSL	0.79[b]	5.0
Diethyl Phthalate	84-66-2	SW8270D	EPA RSL	1,500	5.0
Dimethyl Phthalate	131-11-3	SW8270D	--	NA	5.0
Di-n-Butyl Phthalate	84-74-2	SW8270D	EPA RSL	90	5.0
Di-n-octyl Phthalate	117-84-0	SW8270D	EPA RSL	20	5.0
Fluoranthene	206-44-0	SW8270D	EPA RSL	80	5.0
Fluorene	86-73-7	SW8270D	EPA RSL	29	5.0
Hexachlorobenzene	118-74-1	SW8270D	EPA MCL	1[b]	5.0
Hexachlorobutadiene	87-68-3	SW8270D	EPA RSL	0.14[b]	5.0
Hexachlorocyclopentadiene	77-47-4	SW8270D	EPA MCL	50	10
Hexachloroethane	67-72-1	SW8270D	EPA RSL	0.33[b]	5.0
Indeno(1,2,3-cd)pyrene	193-39-5	SW8270D	EPA RSL	0.25[b]	5.0
Isophorone	78-59-1	SW8270D	EPA RSL	78	5.0
Naphthalene	91-20-3	SW8270D	EPA RSL	0.17[b]	5.0
Nitrobenzene	98-95-3	SW8270D	EPA RSL	0.14[b]	5.0
N-Nitroso-di-n-propylamine	621-64-7	SW8270D	EPA RSL	0.011[b]	5.0
N-Nitrosodiphenylamine	86-30-6	SW8270D	EPA RSL	12	5.0
Pentachlorophenol	87-86-5	SW8270D	EPA MCL	1[b]	20
Phenanthrene	85-01-8	SW8270D	--	NA	5.0
Phenol	108-95-2	SW8270D	EPA RSL	580	5.0
Pyrene	129-00-0	SW8270D	EPA RSL	12	5.0

[a] If a 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 0.1 were used (November 2017).

[b] Due to 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 = microgram(s) per liter
CAS = Chemical Abstracts Service
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level
NA = not applicable
RL = reporting limit
RSL = regional screening level

Table 2-11. Project Laboratory – Target Analytes and Reporting Limits – Metals in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (µg/L)[a]	Lab RL (µg/L)
Aluminum	7429-90-5	SW6010C	EPA RSL	2000	50
Antimony	7440-36-0	SW6010C	EPA MCL	6	2.5
Arsenic	7440-38-2	SW6010C	EPA MCL	10	2.5
Barium	7440-39-3	SW6010C	EPA MCL	2000	10
Beryllium	7440-41-7	SW6010C	EPA MCL	4	1.25
Cadmium	7440-43-9	SW6010C	EPA MCL	5	1.25
Calcium	7440-70-2	SW6010C	--	NA	1,250
Chromium	7440-47-3	SW6010C	EPA MCL	100	2.5
Cobalt	7440-48-4	SW6010C	EPA RSL	0.6[b]	3.125
Copper	7440-50-8	SW6010C	EPA MCL	1,300	2.5
Iron	7439-89-6	SW6010C	EPA RSL	1,400	25
Lead	7439-92-1	SW6010C	EPA MCL	15	1.25
Magnesium	7439-95-4	SW6010C	--	N/A	1,250
Manganese	7439-96-5	SW6010C	EPA RSL	43	3.75
Mercury	7487-94-7	SW7470A	EPA MCL	2	0.20
Nickel	7440-02-0	SW6010C	EPA RSL	39	2.5
Potassium	7440-09-7	SW6010C	--	NA	1,250
Selenium	7782-49-2	SW6010C	EPA MCL	50	2.5
Silver	7440-22-4	SW6010C	EPA RSL	9.4	2.5
Sodium	7440-23-5	SW6010C	--	NA	1,250
Thallium	7440-28-0	SW6010C	EPA MCL	2	2
Vanadium	7440-62-2	SW6010C	EPA RSL	8.6	3.125
Zinc	7440-66-6	SW6010C	EPA RSL	600	5

[a] If a 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 0.1 were used (November 2017).
[b] Due to 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 = microgram(s) per liter
CAS = Chemical Abstracts Service
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level
NA = not applicable
RL = reporting limit
RSL = regional screening level

Table 2-12. Project Laboratory – Target Analytes and Reporting Limits – Organochlorine Pesticides in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (µg/L)[a]	Lab RL (µg/L)
4,4'-DDD	72-54-8	SW8081B	EPA RSL	0.0063[b]	0.0200
4,4'-DDE	72-55-9	SW8081B	EPA RSL	0.046	0.0200
4,4'-DDT	50-29-3	SW8081B	EPA RSL	0.23	0.2
Aldrin	309-00-2	SW8081B	EPA RSL	0.00092[b]	0.0200
BHC, alpha-	319-84-6	SW8081B	EPA RSL	0.0072[b]	0.0200
BHC, beta-	319-85-7	SW8081B	EPA RSL	0.025	0.0200
BHC, delta-	319-86-8	SW8081B	--	NA	0.0200
BHC, gamma- (Lindane)	58-89-9	SW8081B	EPA MCL	0.2	0.0200
Chlordane, cis-	5103-71-9	SW8081B	EPA RSL	0.02[c]	0.0200
Chlordane, trans-	5103-74-2	SW8081B	EPA RSL	0.02[c]	0.0200
Dieldrin	60-57-1	SW8081B	EPA RSL	0.0018[b]	0.0200
Endosulfan I	959-98-8	SW8081B	EPA RSL	10[d]	0.0200
Endosulfan II	33213-65-9	SW8081B	EPA RSL	10[d]	0.0200
Endosulfan sulfate	1031-07-8	SW8081B	EPA RSL	10[d]	0.0200
Endrin	72-20-8	SW8081B	EPA MCL	2	0.0200
Endrin aldehyde	7421-93-4	SW8081B	--	NA	0.0200
Endrin ketone	53494-70-5	SW8081B	--	NA	0.0200
Heptachlor	76-44-8	SW8081B	EPA MCL	0.4	0.0200
Heptachlor epoxide	1024-57-3	SW8081B	EPA MCL	0.2	0.0200
Methoxychlor	72-43-5	SW8081B	EPA MCL	40	0.0200
Toxaphene	8001-35-2	SW8081B	EPA MCL	3	1.00

[a] If a 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 0.1 were used (November 2017).

[b] Due to 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.

[c] Screening level is equivalent to value listed for Technical Chlordane (CAS#12789-03-6)

[d] Screening level is equivalent to value listed for Endosulfan (CAS# 115-29-7)

Notes:
µg/L = microgram(s) per liter
BHC = benzene hexachloride
CAS = Chemical Abstracts Service
DDD = dichlorodiphenyldichloroethane
DDE = dichlorodiphenyldichloroethene
DDT = dichlorodiphenyltrichloroethane
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level
NA = not applicable
RL = reporting limit
RSL = regional screening level

Table 2-13. Project Laboratory – Target Analytes and Reporting Limits – General Water Quality/Natural Attenuation Parameters in Water (Groundwater/Surface Water)

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	CAS Number	Method	Screening Level	Screening Level Value (mg/L)[a]	Lab RL (mg/L)
TOC	TOC	SW9060	--	NA	3
TDS	TDS	SM2540C	--	NA	20
Ethane (Dissolved Gas)	74-84-0	RSK-175	--	NA	0.004
Ethene (Dissolved Gas)	74-85-1	RSK-175	--	NA	0.004
Methane (Dissolved Gas)	74-82-8	RSK-175	--	NA	0.004
Chloride	16887-00-6	E300.0	--	NA	0.500
Nitrate/Nitrite	14797-55-8/14797-65-0	E300.0	EPA MCL	10/1.0	0.25/0.25
Sulfate	14808-79-8	E300.0	--	NA	2.5
Sulfide	18496-25-8	SM4500S2-CF	--	NA	4.0
Alkalinity	ALK	SM2320B	--	NA	1.00
Stable Oxygen and Hydrogen Isotopes	NA	Picarro Cavity Ring-Down Spectroscopy		NA	NA

[a] If a MCL is set for the analyte the screening level is the MCL. Otherwise the screening level is the RSL for tap water or, if no RSL is set, the soil screening level for groundwater protection using a dilution attenuation factor of 20, and soil saturation level. RSLs corresponding to an excessive lifetime cancer risk of 1×10^{-6} and a hazard quotient of 0.1 were used.

Notes:
CAS = Chemical Abstracts Service
EPA = U.S. Environmental Protection Agency
MCL = maximum contaminant level
mg/L = milligram(s) per liter
MS = mass spectrometer
NA = not applicable
qPCR = quantitative polymerase chain reaction
RL = reporting limit
RSL = regional screening level
TDS = total dissolved solids
TOC = total organic carbon

2.4.2 Data Quality Indicators and Quality Control Sample/Measurement Performance

The QA objective of this plan is to identify procedures and criteria that will provide data of known and appropriate quality for the needs identified in Section 2.4.1. Data quality is assessed by representativeness, comparability, accuracy, precision, and completeness. These parameters, the applicable procedures, and level of effort are described in the following paragraphs.

The applicable QC procedures, quantitative target limits, and level of effort for assessing data quality are dictated by the intended use of the data and nature of the analytical methods. Analytical precision, accuracy, and completeness align with the needs identified in Section 2.4.1. Analytical methods and quality control procedures are further detailed in Section 3.

To assess the quality of the data generated during this site investigation, the data quality indicators of precision, accuracy, representativeness, comparability, completeness, and sensitivity 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/matrix spike duplicate (MS/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:

$$RPD = \frac{(C_1 - C_2)}{(C_1 + C_2) / 2} x100\% \quad (1)$$

where:
RPD = relative percent difference
C1 = larger of the two observed values
C2 = smaller of the two observed values

If calculated from three or more replicates, use relative standard deviation (RSD) rather than RPD:

$$RSD = (s / \overline{y})\, x\, 100\% \quad (2)$$

where:
RSD = relative standard deviation
S = standard deviation
$\overline{y}$ = mean of replicate analyses

Standard deviation, s, is defined as follows:

$$S = \sqrt{\sum_{i=1}^{n} \frac{(yi - \overline{y})^2}{n-1}} \quad (3)$$

where:
S = standard deviation
yi = measured value of the ith replicate
$\overline{y}$ = mean of replicate analyses
N = number of replicates

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 (LCS). 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:

$$\%R = 100\% \; x \left[\frac{S - U}{C_{sa}} \right] \quad (4)$$

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:

$$\%R = 100\% \; x \left[\frac{C_m}{C_{sm}} \right] \quad (5)$$

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 (for example, for storage, preservation, and transportation) were developed and are discussed in Section 3. The associated documentation will establish 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:

$$\%C = 100\% \; x \left[\frac{V}{T} \right] \quad (6)$$

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

Assessment of data for representativeness and comparability will comply with the following qualitative definitions:

Sensitivity of the data is evaluated by comparing the laboratory MDL study results to values reported for the project data. The lowest calibration standard is used to determine the reported laboratory limits. The reported laboratory limits are compared to the project action limits and screening criteria to assess whether sensitivity was adequate for project needs.

2.5 Special Training/Certification (A8)

All project staff working onsite must be health and safety trained and must follow requirements specified in the health and safety plan including the 40-hour Hazardous Waste Operations Training and annual 8-hour refresher training. In addition, the FTL will have completed the 8-hour Hazardous Waste Operations Supervisor Training. The health and safety plan describes the specialized training, including tracking and documentation, required for personnel on the project. The PM will ensure that all field staff have the appropriate training and certifications (if required) for their assigned tasks. Training records are maintained in CH2M's health and safety database. Certifications are verified as part of a readiness review before initiation of work. Table 2-14 provides a summary of the training requirements for the project.

As summarized in Table 2-1, the project staff includes one professional engineer, two professional geologists, one chemist, one construction quality manager, and one project management professional.

2.6 Documents and Records (A9)

Field documentation and records are described in Section 5.11 of the FSP (Appendix A-1 of the RIWP) and standard operating procedures (SOPs) B.3, *Field Documentation*, and B.13, *Sample Handling and Shipping* (Appendix B of the RIWP [CH2M, 2018]). Field documentation records and electronic files that will be generated during the RI include the following:

- Field logbooks
- Field forms
- Photographs (electronic files)
- Chain-of-custody forms
- GPS data (electronic files)

Field logbooks, field forms, and chain-of-custody forms will be handwritten in the field, reviewed by the PM or designated project reviewer 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 B.3, *Field Documentation.* Chain-of-custody documentation is discussed in SOP B.13, *Sample Handling and Shipping.*

Table 2-14. Specialized Training Requirements

Quality Assurance Project Plan, OU-2 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
Field Team Leader	• 40-Hour Hazardous Waste Operations • 8-Hour Hazardous Waste Site Supervisor Training • 8-Hour Hazardous Waste Operations Refresher	• 2013 • 2013 • Current	Jasin Olsen	Field Team Leader	Certificates available upon request and documentation files are maintained onsite.
Field Staff	• 40-Hour Hazardous Waste Site Worker • 8-Hour Refresher	• Current	All field staff	Field technicians, geologist, equipment operators, various	Certificates available upon request and documentation files are maintained onsite.
HAPSITE Gas Chromatograph/ Mass Spectrometer Operator	• INFICON/KD Analytical Training Certificate • 40-Hour Hazardous Waste Site Worker • 8-Hour Refresher	• 2014 • 2013 • 2013/2014	Quin Bingham CH2M Technician	HAPSITE Operator	Certificates available upon request and documentation files are maintained onsite.

Laboratory documentation will follow method requirements and QA protocols listed in Section 3.
Laboratory documentation will be retained for a minimum of 3 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.

Data management procedures are described in Section 5.4.1 of the RIWP (CH2M, 2018).

2.6.1 Analytical Data Package

An EPA Level IV Contract Laboratory Program equivalent data package will be provided by the project laboratory with exception of the 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 analytical methods
- Result for each analyte (dry weight basis for soils)
- Percent solids results for soil samples
- Dilution factor (provide 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 non-conformances 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
- Method blank results
- Analytical batch reference number that cross references samples to QC sample analyses
- Chain-of-custody 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 (required in hard copy format only)
- Interference check sample true and measured concentrations and percent recoveries (required in hard copy format only)
- Method of standard addition results (if applicable; required in hard copy format only)
- Post-digestion spike recoveries (if applicable; required in hard copy format only)
- Internal standard recovery and retention time information, as applicable
- Initial calibration summary, including standard concentrations, response factors, average response factors, RSDs or correlation coefficients, and calibration plots or equations, if applicable (required in hard copy format only)
- Continuing calibration verification summary, including expected and recovered concentrations and percent differences (required in hard copy format only)
- 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, a full EPA Level IV data deliverable equivalent

2.6.2 Electronic Data Deliverable Format

Electronic data deliverables (EDD) 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. The subcontractor laboratories will provide the data in accordance with the specifications in Appendix A as well as the EDD specifications for EQuIS at earthsoft.com/support/edd-formats/EQEDD. All project analytical data will be uploaded to the EQuIS project database for reporting and archiving.

Per Federal Acquisition Regulation (FAR) 52.215-2 (Audit and Records), electronic and hard copy laboratory data must be retained by CH2M for a minimum of 3 years after contract closeout. The laboratory will use a storage device that is capable of recording data for long-term, offline storage. The project consultant team will also retain backup hard copies of field and laboratory data in project files and electronic copies on an offsite file sharing network.

2.6.3 Project Information/Records Storage and Retention

CH2M will retain the entire project file as hard copy and/or electronic data on CH2M project servers until the completion of all project activities or sent to VHA when requested. Thereafter, all data will be transferred to VHA, and CH2M will archive and maintain the data in a secure and protected facility for a minimum of 3 years after contract closeout, per FAR 52.215-2 (Audit and Records). Analytical results and sample location data collected during the RI will be entered into a geographic information system (GIS) database for mapping and publication, and the GIS metadata and files will be transferred to VHA and archived by CH2M in electronic format for transmittal to EPA and UDEQ, if requested.

Backup copies of electronic data, including PDF files of documents will be stored offsite on a SharePoint server maintained by CH2M during the term of the contract, and at the end of the contract, transferred to an offsite data archive site.

The official administrative record for the site is located at Salt Lake City Public Library – Foothill-Anderson Branch, in Salt Lake City, Utah. 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. Hard copies of project reports, including the RIWP, FSP, 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, lab 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 for and the sampling design is described in step seven of the DQO process for each problem statement associated with the RI field work is summarized in Table 2-2. Details are provided in Section 5 of the RIWP and Section 3 of the FSP (Appendix A-1 to the RIWP [CH2M, 2018]). Analytical methods include the following:

- Contaminant analyses (VOCs, SVOCs, total metals, and pesticides)
- General water quality and natural attenuation parameters (stable isotopes of hydrogen and oxygen, total organic carbon, total dissolved solids, chloride, sulfate, nitrate/nitrite, and alkalinity)
- Geotechnical parameters for soil
- Water quality field parameters [pH, oxidation-reduction potential, 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 samples collected during well installation or during source area investigation
- Soil gas samples collected during source area investigation
- Groundwater samples collected by:
  - Passive diffusion sampling
  - Low-flow groundwater sampling
  - Low-yield well sampling
  - Zone Isolation Sampling Technology wells

Methods and protocols are described in further detail in Section 5 of the RIWP and Section 5 of the FSP (Appendix A-1 of the RIWP [CH2M, 2018]). Sampling SOPs are itemized and provided in Appendix B of the RIWP (CH2M, 2018).

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 introduced as evidence.

In addition to field notebooks, VHA requires CH2M to use a number of documents for tracking sample custody. Field documents include sample custody seals and chain-of-custody records. CH2M 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
- Site name and location
- Project number
- Sample type and matrix
- Container
- Preservative
- Analysis method

The process for labelling samples, as well as the sample nomenclature, is provided in further detail in Section 5.11.2 of the FSP (Appendix A-1 of the RIWP [CH2M, 2018]).

3.3.2 Packaging and Shipping

Samples that require cooling to 4 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, one-gallon zip-lock bags with a minimum amount of air. The chain-of-custody record will be placed inside a zip-lock plastic 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 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 following collection. The chain-or-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. Soil gas samples for EPA Method TO-15 analysis will be collected in clean, certified Summa canisters (individually-certified) with clean flow controllers. 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, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Parameter	Method	Matrix	Holding Time (From Date Sampled)	Container	Preservative
Organics					
Dissolved Gases	RSK-175	Water	14 days	3 × 40 mL glass VOA vial[a,b]	4°C (± 2°C), HCl to pH < 2
Organochlorine Pesticides	SW8081B	Soil	14 days to extraction, 40 days to analysis	50 grams minimum in one 8-ounce wide mouth glass jar[b]	4°C (± 2°C)
Organochlorine Pesticides	SW8081B	Water	7 days to extraction, 40 days to analysis	1 × 1 L wide mouth glass bottle	4°C (± 2°C)
VOCs	SW8260C	Soil	48 hours to preservation, Encore samplers 14 days from sample collection	4 × 5 grams Encore[b] 1 × 4-ounce glass jar for moisture content	4°C (± 2°C)
VOCs	SW8260C	Water	14 days	3 × 40 mL glass VOA vial[a,b]	4°C (± 2°C), HCl to pH < 2
SVOCs	SW8270D	Soil	14 days to extraction, 40 days to analysis	50 grams minimum in one 8-ounce wide mouth glass jar[b]	4°C (± 2°C)
SVOCs	SW8270D	Water	7 days to extraction, 40 days to analysis	1 × 1 L wide mouth glass bottle	4°C (± 2°C)
VOCs	TO-15	Soil Gas	30 days	1 L or 6 L Summa Canister	NA
Inorganic					
Metals (except Mercury)	SW6010C	Water	180 days	1 L polyethylene bottle	4°C (± 2°C), HNO_3 to pH < 2
Metals (except Mercury)	SW6010C	Soil	180 days	Fill one 8-ounce wide mouth glass jar	4°C (± 2°C)
Mercury	SW7470A	Water	28 days	1 L polyethylene bottle	4°C (± 2°C), HNO_3 to pH < 2
Mercury	SW7471B	Soil	28 days	Fill one 8-ounce wide mouth glass jar	4°C (± 2°C)
General Chemistry					
Chloride, Nitrate/Nitrite, Sulfate	E300.0	Water	28 days, Nitrate/Nitrite 48 hours	1L polyethylene bottle	4°C (± 2°C)
Sulfide	SM4500S2-CF	Water	7 days	250 mL polyethylene bottle	4°C (± 2°C), NaOH to pH>10; ZnAc
TDS	SM2540C	Water	7 days	1 L polyethylene bottle	4°C (± 2°C)
TOC	SW9060	Water	28 days	250 mL wide mouth glass bottle	4°C (± 2°C), H_2SO_4 to pH < 2

Table 3-1. Recommended Sample Container, Preservation, and Holding Times
Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Parameter	Method	Matrix	Holding Time (From Date Sampled)	Container	Preservative
Alkalinity	SM2320B	Water	14 days	1 L polyethylene bottle	4°C (± 2°C)
Stable Oxygen and Hydrogen Isotopes	Isotope Ratio Mass Spectroscopy	Water	180 days	2 × 20 mL glass vial	4°C (± 2°C)
Geotechnical					
FOC	ASTM D2974	Soil	28 days	Fill one 4-ounce wide mouth glass jar	4°C (± 2°C)
Magnetic Susceptibility	Microbial Insights Laboratory Method	Soil	NA	Fill one 250-mL polyethylene jar	None
Sieve Analysis	ASTM D6913	Soil	NA	5-gallon bucket	None
Dry Bulk Soil Density	ASTM D2937	Soil	NA	Shelby Tube	None
Hydrometer	ASTM D422a	Soil	NA	1 gallon bag	None
USCS Soil Classification	ASTM D2487	Soil	NA	1 gallon bag	None
Atterberg Limits	ASTM D4318	Soil	NA	From USCS bag	None
Gradation	ASTM D1140	Soil	NA	5-gallon 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 required for selective ion matrix analysis.
[b] Additional sample volume required for matrix spike/matrix spike duplicate analysis

Notes:
°C = degrees Celsius
FOC = fraction of organic carbon
H_2SO_4 = sulfuric acid
HCl = hydrochloric acid
HNO_3 = nitric acid
L = liter
MS = mass spectrometer
mL= milliliter(s)
NA = not applicable
NaOH = sodium hydroxide
SVOC = semivolatile organic compound
TDS = total dissolved solid
TOC = total organic carbon
VOA = volatile organic analytic
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. An example chain-of-custody form is included as Appendix B of this QAPP. The sampling team members will fill out the date and time of sample collection 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 assure 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. The laboratory sample custodian will document any anomalies and report these to the laboratory PM. Anomalies will be resolved with the project chemist. 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 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 sample analyses and necessary QA checks have been completed at the laboratory, the unused portion of the sample will be disposed of properly 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 are 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 ensure that the samples remain sealed under custody until arrival at the lab, an additional large liner bag (drum liner type) will be placed inside around the entire contents of cooler, tied tightly closed, and secured with additional custody seal.

3.3.5 Field Notebooks and Field Documentation

Field notebooks/logbooks are hardbound notebooks with pre-printed sequentially numbered pages in which all activities associated with the field investigation will be thoroughly described. Field notebooks are intended to provide sufficient data to reconstruct events occurring during the field project. SOP B.03, *Field Documentation,* and the FSP (Appendixes B and A-1 of the RIWP, respectively [CH2M, 2018]) contain further information. General information regarding sampling activities will be recorded and includes, at a minimum, the following:

- Summary of daily activities, including information presented at the daily safety meeting
- Equipment onsite
- Descriptions of deviations from the FSP or QAPP
- Name and affiliation of all personnel or visitors onsite
- Weather conditions during the field activity
- Stop and start times for sampling activities at each location
- 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 or project documents (for example, work plans and 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 above 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 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, soil gas, 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, oxidation-reduction potential, and temperature. In addition, field screening for VOCs using a portable GC/MS (HAPSITE) will be used to measure concentrations of individual VOCs in soil gas and groundwater. Field analytical methods will be performed in accordance with the SOPs provided in Appendix B of the RIWP (CH2M, 2018). The HAPSITE analyses will be performed according to the manufacturer's operating instructions provided in SOPs B.17 and B.18 (Appendix B of the RIWP [CH2M, 2018]).

3.4.1.1 Specific Conductance

The specific conductance of a sample will be measured by using a calibrated meter equipped with a flow-through cell, weighted screen, or sample cup.

3.4.1.2 Turbidity

Routine calibration of the turbidity instrument will be performed according to the recommended manufacturer's calibration instructions.

3.4.1.3 pH

Commercially prepared buffer solutions will be used for calibration. These solutions are certified with an accuracy of plus or minus 0.01 pH unit at a specific temperature, usually 25°C. Buffer solutions will not be used past the expiration date printed on the label. Buffer solutions used for instrument calibration will be stored in a separate container and replaced with new buffer solutions at least once per week.

3.4.1.4 Dissolved Oxygen

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

Oxidation-reduction potential meters will be calibrated and used according to the manufacturer's guidelines.

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 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 soil gas or headspace for purged groundwater samples. The portable GC/MS will be calibrated daily in accordance with the manufacturer's operating instructions provided in SOP B.17, *Analytical Method for the Determination of Volatile Organics in Air Using the HAPSITE Field GC/MS* (Appendix B of the RIWP [CH2M, 2018]). Use of the headspace sampling system for screening-level groundwater analysis using the HAPSITE is described in SOP B.18 in Appendix B of the RIWP (CH2M, 2018). HAPSITE method files and data files will be retained on the project drive. Project analytes, methods, and target detection limits are provided in Tables 2-8 and 2-9. 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 Appendixes C and D, respectively. Laboratory manuals and SOPs are referenced, but not provided for geotechnical analyses as they are standard ASTM methods. A laboratory QA manual and SOP are not provided for the magnetic susceptibility analysis because that is a proprietary method. An SOP is provided for the stable isotope method, which will be performed at the SIRFER laboratory at the University of Utah. With the exception of stable isotope analyses for water samples and geotechnical analyses for soil samples (which are not chemical analyses regulated under the CERCLA program), analyses will be performed by laboratories that can demonstrate method proficiency and are certified in the State of Utah (if the method is included in the state's certification program).

The turnaround time for laboratories will be 30 calendar days from sample receipt.

Project analytes, methods, and target laboratory detection limits are listed in Tables 2-3 through 2-13.

The following methods will be used for the project:

- **Metals – SW-846 Method 6010C, Inductively Coupled Plasma.** 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/7471B, Cold Vapor Atomic Absorption.** This technique is based on the absorption by mercury vapor. The 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.
- **Organochlorine Pesticides – SW-846 Method 8081B, Gas Chromatography/Electron Capture Detection.** This technique is used to quantitate organochlorine pesticides 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.
- **Volatile Organic Analytic (VOA) – SW-846 Method 8260C, 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.
- **Semivolatile VOA – SW-846 Method 8270D, GC/MS.** The SVOC GC/MS technique is used to quantitate most neutral, acidic, and basic 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. Such compounds include

polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols including nitrophenols.

- **VOA – EPA Method TO-15, GC/MS.** Volatile organics in soil gas are collected in an evacuated Summa canister and analyzed using the GC/MS technique.
- **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.
- **Dissolved Gases (Methane, Ethane, Ethene) – Method RSK-175, GC/Flame Ionization Detector.** Dissolved gases in water are sampled and analyzed uses GC coupled to a flame ionization detector and is able to detect methane, ethane, and ethene at low parts per billion levels.
- **Total Organic Carbon – 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.
- **Alkalinity – Standard Methods 2320B, Titrimetric.** Alkalinity is determined by titrating a portion of the sample to an endpoint of pH 4.5 using hydrochloric acid.
- **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-CF, 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.
- **Magnetic Susceptibility – Microbial Insights Laboratory Method.** A proprietary method utilizing a Bartington magnetic susceptibility system to assess the magnetic susceptibility of soil samples.
- **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 D422a.** Measurement of particle size of fine-grained (silt/clay) particles.
- **Unified Soil Classification System Soil Classification – ASTM D2487.** Standard soil classification system.
- **Atterberg Limits – ASTM D4318.** Measurement of plasticity of the 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 (wt/wt) 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 (for example, sample collection and shipping) and checks controlling laboratory activities (for example, digestion and analysis) are used. These checks are discussed in the following subsections. The numbers of QC samples associated with each sample medium, and details regarding their collection, are provided in Section 5.12 of the FSP (Appendix A-1 of the RIWP [CH2M, 2018]).

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 fraction of organic carbon and magnetic susceptibility, field QC samples will not be collected for any geotechnical 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 media. The 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 analysis. Field blanks will be collected at a minimum frequency of 1 per 20 samples collected or 1 per day when fewer than 20 samples are collected. No field blanks will be collected for soil gas samples (EPA Method TO-15).

3.5.1.2 Equipment Rinsate Blanks

Equipment rinsate blanks are used to evaluate sampling device cleanliness. Rinsate blanks are collected after a 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 minimum frequency of 1 per 20 samples collected or 1 per day when fewer than 20 samples are collected. No equipment rinsate blanks will be collected for soil gas samples (EPA Method TO-15).

3.5.1.3 Trip Blanks

The trip blank consists of a VOC 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. Trip blanks are prepared only when VOC samples are collected and analyzed for VOC analytes. No trip blanks will be collected for soil gas samples (EPA Method TO-15).

Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. Each cooler of samples sent to the laboratory for analysis of VOCs will contain one trip blank. When an analyte is detected in the trip blank, the appropriate flag will be applied to all VOC sample results from samples in the cooler with the affected trip blank.

3.5.1.4 Field Duplicates

A FD sample is collected at the same time and from the same source as the original sample, but submitted to the laboratory as a separate sample to assess the consistency of the overall sampling and analytical system. Water, soil, and soil gas FDs will be collected and analyzed on a 10 percent basis for all analytical laboratory methods or a minimum of 1 per sampling event (defined as a shipment of

samples to the lab under one chain-of-custody) if fewer than 10 samples are collected. Field duplicate samples will be collected, numbered, packaged, 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 4°C will contain at least one temperature blank. The temperature blank should be a 40-milliliter glass vial 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 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 method blanks, LCSs, MSs, MSDs, and laboratory duplicates. In addition, post digestion spike, serial dilution, and interference check samples will be evaluated for metals analysis only, and surrogates and internal standards will be evaluated for those methods where applicable (for example, organic methods only). The laboratory will also complete initial calibrations 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 3-3 through 3-12.

3.5.2.1 Method Blanks

Method blanks are designed to detect contamination of the field samples in the laboratory environment. Method blanks verify that interferences caused by contaminants in solvents, reagents, glassware, or in other sample processing hardware are known and minimized. The method blank 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 re-analysis. 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 method blank. Method blanks are performed per laboratory batch per analytical method.

3.5.2.2 Matrix Spikes/Matrix Spike Duplicates

MSs and MSDs are aliquots of a sample spiked with known concentrations of all target analytes. The spiking occurs during sample preparation and before analysis. Each analyte in the MS and 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 and one MSD will be designated by the PM for each site and included for every 20 field samples, for applicable methods. MS/MSDs are not performed for EPA Method TO-15.

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 sample, depending on the laboratory SOP.

3.5.2.4 Post Digestion Spike

A post-digestion spike is a portion of the sample digestate that is fortified with known quantities of compounds of interest. The post-digestion spike 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. Post-digestion spikes are only evaluated for metals analyses.

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 percent difference (%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 (for example, metals analyses) to verify background and inter-element correction factors. They consist of two solutions: A and B. Solution A contains the interfering analytes, and Solution B contains both the analytes of interest and the interfering analytes. Both solutions 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

The 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. Failure of the LCS to meet %R criteria for any compound requires corrective action specified in Tables 3-3 through 3-12. If corrective action specifies re-extraction and re-analysis, all samples associated with the out-of-control LCS must be re-extracted and re-analyzed after control is reestablished. All re-extraction and re-analysis 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 analyte(s) 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. Whenever a surrogate recovery is outside the acceptance limit, corrective action as outlined in Tables 3-3 through 3-12, where applicable, must be performed.

3.5.2.9 Internal Standards

Internal standards are compounds added to every GC/MS standard, method blank, LCS/LCS duplicate, MS/MSD, and sample extract at a known concentration before instrument analysis for organic methods only. They are used to quantify the target analytes. Internal standards provide for stable sensitivity and response during every analytical run. An internal standard 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 Soil Gas/Water (HAPSITE)
Quality Assurance Project Plan, OU-2 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	% Recovery = 70-130%	Rerun, investigate source of problems. Recalibrate instrustment if rerun does not meet QC acceptance limits	Field Technician	Accuracy	% Recovery = 70-130%
System Blank	Daily, before sample analysis	Target analytes: < RL	Rerun, maintenance.	Field Technician	Accuracy – Contamination	Target analytes: < RL
Internal Standards	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

Table 3-3. Quality Control Samples (Field and Lab) for Volatile Organic Compounds in Soil and Water
Quality Assurance Project Plan, OU-2 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
Method Blank	One per prep batch of 20 or fewer samples of similar matrix	Target analytes < 50% RL except for common lab contaminants < RL	Investigate source of contamination. Rerun method blank. No samples may be run until an acceptable method blank has been run	Laboratory Supervisor	Accuracy	Target analytes < 50% RL except for common lab contaminants < RL
Laboratory Control Sample	One per batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-4	Re-prepare sample and re-analyze, if recovery is high and sample is < RL narrate	Laboratory Supervisor / Data Validator	Accuracy	Recovery within QC limits as defined in Table 3-4
Matrix Spike	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, re-prepare the samples and QC. Check standard prep. Flag data	Laboratory Supervisor / Data Validator	Accuracy	Recovery within QC limits as defined in Table 3-4
Matrix Spike Duplicate	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 / Data Validator	Accuracy and Precision	Recovery within QC limits as defined in Table 3-4
Internal Standards	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 / Data Validator	Accuracy	IS area -50% to +100% compared to IS from CCV IS RT window ±0.5 minutes compared to CCV RT

Table 3-3. Quality Control Samples (Field and Lab) for Volatile Organic Compounds in Soil and Water
Quality Assurance Project Plan, OU-2 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
Surrogate Standard	Every sample	All surrogate recoveries within laboratory specified QC limits	If sample volume available, re-analyze. Report both if second successful analysis is outside hold time or both fail QC criteria. Flag data	Laboratory Supervisor / Data Validator	Accuracy	All surrogate recoveries within laboratory specified QC limits
Field Duplicates	One per 10 per matrix	RPD < 30% (aq), < 50% (solids) for compounds w/ concentration > RL	Evaluate batch precision, other FD results Note in validation report	Data Validator	Precision	RPD < 30% (aq), < 50% (solids) for compounds w/ concentration > RL
Equipment Blank	Collected 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
Trip Blank	One per shipment aqueous VOCs	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:
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, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD % Recovery[a]	Soil LCS % Recovery[a]	RPD[b]	Water MS/MSD % Recovery[a]	Water LCS % Recovery[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

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

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD % Recovery[a]	Soil LCS % Recovery[a]	RPD[b]	Water MS/MSD % Recovery[a]	Water LCS % Recovery[a]	RPD[b]
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
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
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 (Cumene)	SW8260C	68 - 134	68 - 134	30	72-131	72-131	20
Methyl acetate	SW8260C	53 - 144	53 - 144	30	56-136	56-136	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

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

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD % Recovery[a]	Soil LCS % Recovery[a]	RPD[b]	Water MS/MSD % Recovery[a]	Water LCS % Recovery[a]	RPD[b]
Toluene	SW8260C	77 - 121	77 - 121	30	80-121	80-121	20
Total 1,3-dichloro propene (cis- & trans-)	SW8260C	71 - 130	71 - 130	30	77-123	77-123	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	65-141	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 based on DoD QSM Version 5.1, Appendix C (DoD, 2017)
[b] RPDs are applicable to both MS/MSD and LCS

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

Table 3-5. Quality Control Samples (Field and Lab) for Semivolatile Organic Compounds in Soil and Water

Quality Assurance Project Plan, OU-2 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
Method Blank	One per prep batch of 20 or fewer samples of similar matrix	Target analytes < 50% RL except for common lab contaminants < RL	Investigate source of contamination. Rerun method blank. If MB still fails acceptance limits, re-prepare/reanalyze method blank and associated samples. No samples may be run until an acceptable method blank has been run	Laboratory Supervisor	Accuracy	Target analytes < 50% RL Except for common lab contaminants < RL unless target analytes in field samples are > 10× those in method blank
Laboratory Control Sample	One per batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-6	Re-prepare sample and re-analyze, if recovery is high and sample is < RL narrate	Laboratory Supervisor / Data Validator	Accuracy	Recovery within QC limits as defined in Table 3-6
Matrix Spike	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, re-prepare the samples and QC. Check standard prep. Flag data	Laboratory Supervisor / Data Validator	Accuracy	Recovery within QC limits as defined in Table 3-6
Matrix Spike Duplicate	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 / Data Validator	Accuracy and Precision	Recovery within QC limits as defined in Table 3-6
Internal Standards	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 / Data Validator	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 Lab) for Semivolatile Organic Compounds in Soil and Water
Quality Assurance Project Plan, OU-2 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%
Field Duplicates	One per 10 per matrix; minimum of one per matrix	RPD < 30% (aq), <50% (solid) for compounds w/ concentration > RL	Evaluate batch precision, other FD results Note in validation report	Data Validator	Precision	RPD < 30% (aq), <50% (solid) for compounds w/ concentration > RL
Equipment Blank	Collected 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

Notes:
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 Semivolatile Organic Compounds in Soil and Water

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD % Recovery[a]	Soil LCS % Recovery[a]	RPD[b]	Water MS/MSD % Recovery[a]	Water LCS % Recovery[a]	RPD[b]
1,1-'-Biphenyl	SW8270D	40-117	40-117	30	49-115	49-115	20
1,2,4,5-Tetrachlorobenzene	SW8270D	37-119	37-119	30	35-121	35-121	20
1,4-Dioxane	SW8270D	[c]	[c]	30	[c]	[c]	20
2,3,4,6-Tetrachlorophenol	SW8270D	44-125	44-125	30	50-128	50-128	20
2,4,5-Trichlorophenol	SW8270D	41-124	41-124	30	53-123	53-123	20
2,4,6-Trichlorophenol	SW8270D	39-126	39-126	30	50-125	50-125	20
2,4-Dichlorophenol	SW8270D	40-122	40-122	30	47-121	47-121	20
2,4-Dimethylphenol	SW8270D	30-127	30-127	30	31-124	31-124	20
2,4-Dinitrophenol	SW8270D	[c]	[c]	30	23-143	23-143	20
2,4-Dinitrotoluene	SW8270D	48-126	48-126	30	57-128	27-128	20
2,6-Dinitrotoluene	SW8270D	46-124	46-124	30	57-124	27-124	20
2-Chloronaphthalene	SW8270D	41-114	41-114	30	40-116	40-116	20
2-Chlorophenol	SW8270D	34-121	34-121	30	38-117	38-117	20
2-Methylnaphthalene	SW8270D	38-122	38-122	30	40-121	40-121	20
2-Methylphenol	SW8270D	32-122	32-122	30	30-117	30-117	20
2-Nitroaniline	SW8270D	44-127	44-127	30	55-127	55-127	20
3-Nitroaniline	SW8270D	33-119	33-119	30	41-128	41-128	20
2-Nitrophenol	SW8270D	36-123	36-123	30	47-123	47-123	20
3,3'-Dichlorobenzidine	SW8270D	22-121	22-121	30	27-129	27-129	20
4,6-Dinitro-2-methylphenol	SW8270D	29-132	29-132	30	44-137	44-137	20
4-Bromophenyl Phenyl Ether	SW8270D	46-124	46-124	30	55-124	55-124	20
4-Chloroaniline	SW8270D	17-106	17-106	30	33-117	33-117	20
4-Chloro-3-methylphenol	SW8270D	45-122	45-122	30	52-119	52-119	20
4-Chlorophenyl Phenyl Ether	SW8270D	45-121	45-121	30	53-121	53-121	20
4-Methylphenol	SW8270D	42-126	42-126	30	25-120	25-120	20
4-Nitroaniline	SW8270D	[c]	[c]	30	[c]	[c]	20

Table 3-6. Precision and Accuracy for Semivolatile Organic Compounds in Soil and Water

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD % Recovery[a]	Soil LCS % Recovery[a]	RPD[b]	Water MS/MSD % Recovery[a]	Water LCS % Recovery[a]	RPD[b]
4-Nitrophenol	SW8270D	30-132	30-132	30	[c]	[c]	20
Acenaphthene	SW8270D	40-123	40-123	30	47-122	47-122	20
Acenaphthylene	SW8270D	32-132	32-132	30	41-130	41-130	20
Acetophenone	SW8270D	33-115	33-115	30	46-118	46-118	20
Anthracene	SW8270D	47-123	47-123	30	57-123	57-123	20
Atrazine	SW8270D	47-127	47-127	30	44-142	44-142	20
Benzaldehyde	SW8270D	[c]	[c]	30	[c]	[c]	20
Benzo(a)anthracene	SW8270D	49-126	49-126	30	58-125	58-125	20
Benzo(a)pyrene	SW8270D	45-129	45-129	30	54-128	54-128	20
Benzo(b)fluoranthene	SW8270D	45-132	45-132	30	53-131	53-131	20
Benzo(g,h,i)perylene	SW8270D	43-134	43-134	30	50-134	50-134	20
Benzo(k)fluoranthene	SW8270D	47-132	47-132	30	57-129	57-129	20
Bis(2-chloroethoxy)methane	SW8270D	36-121	36-121	30	48-120	48-120	20
Bis(2-chloroethyl)ether	SW8270D	31-120	31-120	30	43-118	43-118	20
Bis(2-chloroisopropyl)ether	SW8270D	33-131	33-131	30	37-130	37-130	20
Bis(2-Ethylhexyl)Phthalate	SW8270D	51-133	51-133	30	55-135	55-135	20
Butyl Benzyl Phthalate	SW8270D	48-132	48-132	30	53-134	53-134	20
Caprolactam	SW8270D	46-117	46-117	30	[c]	[c]	20
Carbazole	SW8270D	50-123	50-123	30	60-122	60-122	20
Chrysene	SW8270D	50-124	50-124	30	59-123	59-123	20
Dibenz(a,h)anthracene	SW8270D	45-134	45-134	30	51-134	51-134	20
Dibenzofuran	SW8270D	44-120	44-120	30	53-118	53-118	20
Diethyl Phthalate	SW8270D	50-124	50-124	30	56-125	56-125	20
Dimethyl Phthalate	SW8270D	48-124	48-134	30	45-127	45-127	20
Di-n-Butyl Phthalate	SW8270D	51-128	51-128	30	59-127	59-127	20
Di-n-octyl Phthalate	SW8270D	45-140	45-140	30	51-140	51-140	20

Table 3-6. Precision and Accuracy for Semivolatile Organic Compounds in Soil and Water

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD % Recovery[a]	Soil LCS % Recovery[a]	RPD[b]	Water MS/MSD % Recovery[a]	Water LCS % Recovery[a]	RPD[b]
Fluoranthene	SW8270D	50-127	50-127	30	57-128	57-128	20
Fluorene	SW8270D	43-125	43-125	30	52-124	52-124	20
Hexachlorobenzene	SW8270D	45-122	45-122	30	53-125	53-125	20
Hexachlorobutadiene	SW8270D	32-123	32-123	30	22-124	22-124	20
Hexachlorocyclopentadiene	SW8270D	[c]	[c]	30	[c]	[c]	20
Hexachloroethane	SW8270D	28-117	28-117	30	21-115	21-115	20
Indeno(1,2,3-cd)pyrene	SW8270D	45-133	45-133	30	52-134	52-134	20
Isophorone	SW8270D	30-122	30-122	30	42-124	42-124	20
Naphthalene	SW8270D	35-123	35-123	30	40-121	40-121	20
Nitrobenzene	SW8270D	34-122	34-122	30	45-121	45-121	20
N-Nitroso-di-n-propylamine	SW8270D	36-120	36-120	30	49-119	49-119	20
N-Nitrosodiphenylamine	SW8270D	38-127	38-127	30	51-123	51-123	20
Pentachlorophenol	SW8270D	25-133	25-133	30	35-138	35-138	20
Phenanthrene	SW8270D	50-121	50-121	30	59-120	59-120	20
Phenol	SW8270D	34-121	34-121	30	–		20
Pyrene	SW8270D	47-127	47-127	30	57-126	57-126	20

[a] LCS and MS/MSD limits based on DoD QSM Version 5.1, Appendix C (DoD, 2017)
[b] RPDs are applicable to both MS/MSD and LCS/LCSD
[c] Will use laboratory limits

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

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

Quality Assurance Project Plan, OU-2 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 re-analyze, 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 method blank
Interference Check Solutions (A and B)	At beginning and end of instrument run and after every 20 samples	ICS-A: Unspiked analytes < RL ICS-AB: % Recovery 80% -120%	Reanalyze samples analyzed after last acceptable ICS-A/ICS-AB	Laboratory Supervisor	Accuracy	ICS-A: Unspiked analytes < RL ICS-AB: % Recovery 80% - 120%
Serial Dilution	One per prep batch of 20 or fewer samples of similar matrix	% Difference 10% if sample concentration > 50× MDL	Perform PDS, Qualify data	Laboratory Supervisor / Data Validator	Accuracy	% Difference 10% if sample concentration > 10× MDL
Laboratory Control Sample	One per prep batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-8	Redigest and re-analyze, if recovery is high and sample is < RL narrate	Laboratory Supervisor	Accuracy	Defined in Table 3-8
Laboratory Duplicate	One per prep batch of 20 or fewer samples of similar matrix	RPD $\leq$ 20% if sample concentration > 5×RL, if < 5×RL	Check instrument performance, qualify data	Laboratory Supervisor / Data Validator	Precision	Values ≥5×RL: RPD ±20% Values < 5×RL: ±5×RL
Matrix Spike Sample	One per prep batch of 20 or fewer samples of similar matrix	% Recovery 75% - 125%	Perform post-digestion spike analysis, qualify data	Laboratory Supervisor / Data Validator	Accuracy	Recovery ±25% of true value if sample < 4× spike value
Post-Digestion Spike	For elements outside of QC limits in Matrix Spike	% Recovery 75% - 125%	Check instrument performance, qualify data	Laboratory Supervisor / Data Validator	Accuracy	Recovery ±25% of true value
Internal Standards[a]	Each field and QC sample[a]	Percent relative intensity of each IS standard within 60 – 125%	Reanalyze samples after 2-fold dilution	Laboratory Supervisor	Accuracy	Percent relative intensity of each IS standard within 60 – 125%

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

Quality Assurance Project Plan, OU-2 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
Field Duplicates	One per 10 per matrix	RPD < 30% (aq), <50% (solid) for compounds w/ concentration > RL	Evaluate batch precision, other FD results Note in validation report	Data Validator	Precision	RPD < 30% (aq), <50% (solid) for compounds w/ concentration > RL
Equipment Blank	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] Internal standards are not required, but are optional for Method SW6010C. Empirical Laboratories does employ the use of internal standards for SW6010C.

Notes:
aq = aqueous
EB = equipment blank
FD = field duplicate
ICS = interference check solutions
IS = internal standards
MDL = method detection limit
PDS = post digestion 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, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD % Recovery	Soil LCS % Recovery	RPD[a]	Water MS/MSD % Recovery	Water LCS % Recovery	RPD[a]
Aluminum	SW6010C	75-125	70-130	20	75-125	70-130	20
Antimony	SW6010C	75-125	50-150	20	75-125	50-150	20
Arsenic	SW6010C	75-125	70-130	20	75-125	70-130	20
Barium	SW6010C	75-125	70-130	20	75-125	70-130	20
Beryllium	SW6010C	75-125	70-130	20	75-125	70-130	20
Cadmium	SW6010C	75-125	70-130	20	75-125	70-130	20
Calcium	SW6010C	75-125	70-130	20	75-125	70-130	20
Chromium	SW6010C	75-125	70-130	20	75-125	70-130	20
Cobalt	SW6010C	75-125	70-130	20	75-125	70-130	20
Copper	SW6010C	75-125	70-130	20	75-125	70-130	20
Iron	SW6010C	75-125	70-130	20	75-125	70-130	20
Lead	SW6010C	75-125	70-130	20	75-125	70-130	20
Magnesium	SW6010C	75-125	70-130	20	75-125	70-130	20
Manganese	SW6010C	75-125	70-130	20	75-125	70-130	20
Mercury	SW7470A/7471B	75-125	70-130	20	75-125	70-130	20
Nickel	SW6010C	75-125	70-130	20	75-125	70-130	20
Potassium	SW6010C	75-125	70-130	20	75-125	70-130	20
Selenium	SW6010C	75-125	70-130	20	75-125	70-130	20
Silver	SW6010C	75-125	50-150	20	75-125	50-150	20
Sodium	SW6010C	75-125	70-130	20	75-125	70-130	20
Thallium	SW6010C	75-125	70-130	20	75-125	70-130	20
Vanadium	SW6010C	75-125	70-130	20	75-125	70-130	20
Zinc	SW6010C	75-125	70-130	20	75-125	70-130	20

[a] RPDs are applicable to MS/MSD only

Notes:
LCS = laboratory control samples
MS/MSD = matrix spike/matrix spike duplicate
RPD = relative percent difference

Table 3-9. Quality Control Samples (Field and Lab) for Organochlorine Pesticides in Soil and Water

Quality Assurance Project Plan, OU-2 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
Method Blank	One per prep batch of 20 or fewer samples of similar matrix	Target analytes < 50% RL except for common lab contaminants < RL	Investigate source of contamination. Rerun method blank. If MB still fails acceptance limits, re-prepare/ reanalyze method blank and associated samples. No samples may be run until an acceptable method blank has been run.	Laboratory Supervisor	Accuracy	Target analytes < 50% RL except for common lab contaminants < RL unless target analytes in field samples are > 10× those in method blank
Laboratory Control Sample	One per batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-10	Re-prepare and re-analyze, if recovery is high and sample is < RL narrate	Laboratory Supervisor / Data Validator	Accuracy	Recovery within QC limits as defined in Table 3-10
Matrix Spike	One per batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-10	If recoveries are outside limits and surrogate criteria are met, note in narrative. If both the surrogate and MS/MSD are unacceptable, re-prepare the samples and QC. Check standard prep. Flag data	Laboratory Supervisor / Data Validator	Accuracy/	Recovery within QC limits as defined in Table 3-10
Matrix Spike Duplicate	One per batch of 20 or fewer samples of similar matrix	Recovery within QC limits as defined in Table 3-10	Same as MS	Laboratory Supervisor / Data Validator	Accuracy and Precision	Recovery within QC limits as defined in Table 3-10
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 Quality Assurance Manager	Accuracy	Recoveries within laboratory specified QC control limits
Field Duplicates	One per 10 per matrix	RPD < 30% (aq), < 50% (solid) for compounds w/ concentration > RL	Evaluate batch precision, other FD results Note in validation report	Data Validator	Precision	RPD < 30% (aq), < 50% (solid) for compounds w/ concentration > RL
Equipment Blank	Collected 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

Notes:
aq = aqueous
EB = equipment blank
FD = field duplicate
MB = method blank
MS = matrix spike
MSD = matrix spike duplicate
QC = quality control
RL = reporting limit
RPD = relative percent difference

Table 3-10. Precision and Accuracy for Organochlorine Pesticides in Soil and Water

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Soil MS/MSD % Recovery[a]	Soil LCS % Recovery[a]	RPD[b]	Water MS/MSD % Recovery[a]	Water LCS % Recovery[a]	RPD[b]
4,4'-dichlorodiphenyldichloroethane	SW8081B	56-139	56-139	30	56-143	56-143	20
4,4'-dichlorodiphenyldichloroethene	SW8081B	56-134	56-134	30	57-135	57-135	20
4,4'-dichlorodiphenyltrichloroethane	SW8081B	50-141	50-141	30	51-143	51-143	20
Aldrin	SW8081B	45-136	45-136	30	45-134	45-134	20
benzene hexachloride, alpha-	SW8081B	45-137	45-137	30	54-138	54-138	20
benzene hexachloride, beta-	SW8081B	50-136	50-136	30	56-136	56-136	20
benzene hexachloride, delta-	SW8081B	47-139	47-139	30	52-142	52-142	20
benzene hexachloride, gamma (Lindane)	SW8081B	49-135	49-135	30	59-134	59-134	20
Chlordane, cis-	SW8081B	54-133	54-133	30	60-129	60-129	20
Chlordane, trans-	SW8081B	53-135	53-135	30	56-136	56-136	20
Dieldrin	SW8081B	56-136	56-136	30	60-136	60-136	20
Endosulfan I	SW8081B	53-132	53-132	30	62-126	62-126	20
Endosulfan II	SW8081B	53-134	53-134	30	52-135	52-135	20
Endosulfan sulfate	SW8081B	55-136	55-136	30	62-133	62-133	20
Endrin	SW8081B	57-140	57-140	30	60-138	60-138	20
Endrin aldehyde	SW8081B	35-137	35-137	30	51-132	51-132	20
Endrin ketone	SW8081B	55-136	55-136	30	58-134	58-134	20
Heptachlor	SW8081B	47-136	47-136	30	54-130	54-130	20
Heptachlor epoxide	SW8081B	52-136	52-136	30	61-133	61-133	20
Methoxychlor	SW8081B	52-143	52-143	30	54-145	54-145	20
Toxaphene	SW8081B	33-141	33-141	30	33-134	33-134	20

[a] LCS and MS/MSD limits based on DoD QSM Version 5.1, Appendix C (DoD, 2017)
[b] RPDs are applicable to both MS/MSD and LCS/LCSD

Notes:
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 3-11. Quality Control Samples (Field and Lab) for Volatile Organic Compounds in Soil Gas

Quality Assurance Project Plan, OU-2 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
Method Blank	Daily	Target analytes: < 50% RL; common contaminants <2.5× RL	Rerun, maintenance. Flag data	Lab Chemist	Accuracy – Contamination	Target Analytes < 50% RL; common contaminants < 2.5× RL unless results in field samples > 10× that in method blank
LCS/LCS Duplicate	Daily	% Recovery = 70-130% RPD < 30%	Rerun, investigate source of problems. Flag data	Lab Chemist	Accuracy Precision	% Recovery 70-130% RPD <30%
Field duplicate	One per 10 samples	RPD < 40%	Flag data	Data Validator	Precision	RPD <40%
Internal Standards	Every sample	Area ±40% DCV	Rerun, flag data	Lab Chemist	Accuracy	Area ±40% DCV
Surrogate spike	Every sample	% Recovery 70-130% or as per lab limits	Rerun, flag data	Lab Chemist	Accuracy	% Recovery 70-130% or as per lab limits
Reporting Limit LCS	Daily	Results ±40% true for 90% of compounds	Flag data that fall outside control limits	Lab 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	Lab Chemist	Accuracy – Contamination	After cleaning, pressurized canister (using zero humid air) should not contain Target Analytes greater than 0.2 ppbV

Notes:
DCV = daily calibration verification
LCS = laboratory control sample
ppbV = part(s) per billion by volume
QC = quality control
RL = reporting limit
RPD = relative percent difference
SOP = standard operating procedure

Table 3-12. Quality Control Samples (Field and Lab) for General Chemistry Parameters in Water

Quality Assurance Project Plan, OU-2 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	Re-prepare and re-analyze, 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 method blank.
Laboratory Control Sample[b]	One per prep batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits as defined in Table 3-13	Re-prepare and re-analyze, if recovery is high and sample is < RL narrate	Laboratory Supervisor	Accuracy	Recovery within lab statistical QC limits as defined in Table 3-13
Laboratory Duplicate	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 / Data Validator	Precision	Values ≥5×RL: RPD ±20% Values < 5×RL: ±5× RL
Matrix Spike Sample[c]	One per prep batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits as defined in Table 3-13	Re-prepare and re-analyze, if recovery is high and sample is < RL narrate	Laboratory Supervisor / Data Validator	Accuracy	Recovery within lab statistical QC limits (< 4× spike value) as defined in Table 3-13
Field Duplicates	One per 10 per matrix	RPD <30% for compounds w/ concentration > RL	Evaluate batch precision, other FD results. Note in validation report	Data Validator	Precision	RPD < 30% for compounds w/ concentration > RL
Equipment Blank[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:
EB = equipment blank
FD = field duplicate
QC = quality control
RL = reporting limit
RPD = relative percent difference
SOP = standard operating procedure

Table 3-13. Precision and Accuracy for General Chemistry Parameters in Water

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Analyte	Method	Water MS/MSD % Recovery	Water LCS % Recovery	RPD[c]
Chloride	EPA 300.0[a]	87 – 111	87 – 111	20
Nitrate/Nitrite	EPA 300.0[a]	88 – 111	88 – 111	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

[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/MSD only

Notes:
DoD = U.S. Department of Defense
LCS = laboratory control sample
MS/MSD = matrix spike/matrix spike duplicate
NA = Not applicable
RPD = relative percent difference
TDS = total dissolved solids
TOC = total organic carbon

Table 3-14. Quality Control Samples (Field and Lab) for Stable Oxygen and Hydrogen Isotope Parameters in Water
Quality Assurance Project Plan, OU-2 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 8 samples	Not vary by more than 0.9‰ for δ2H and 0.12‰ for δ18O (1 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 δ2H and 0.12‰ for δ18O (1 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
Field Duplicates	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‰ (δ18O) and 0.75‰ (δ2H)	Evaluate batch precision, other FD results. Note in validation report	Data Validator	Precision	RPD < 50% for compounds w/ 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 quality control standards to monitor method performance.

Notes:
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.

3.6.1 Field Equipment

Field personnel will record service and maintenance information of all field equipment in the project field logbook(s). 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 B.1, *Equipment Calibration* (Appendix B of the RIWP [CH2M, 2018]). A calibration will be verified at any time during the day when measurements are suspected to be erroneous. Calibration solutions will be available onsite 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 onsite. 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 SOP B.17 (Appendix B of the RIWP [CH2M, 2018]).

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 FTL and the project data manager. If the activity results are adversely affected, the results of the evaluation will be documented and the PM and (other data users) will be notified. The 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.

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 non-routine. 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.

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

The ICAL curves must be verified daily before sample analysis using a continuing calibration verification (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 ensure 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 of OU-2 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 B of the RIWP (CH2M, 2018).

3.8 Data Acquisition Requirements (Non-direct Measurements – B9)

Non-direct measurements including historical data (for example, water quality data and, groundwater elevation measurements) were used in the development of the conceptual site model and the OU-2 project objectives. Relevant historical data will be stored in a project-specific database built in the EarthSoft EQuIS system described in the data management plan (CH2M, 2017). All manually generated information (for example, field test results and GIS sample coordinates) will undergo QC verification as required by the project data manager. The acceptance criteria for existing data are included in relevant SOPs (Appendix B of the RIWP [CH2M 2018]), as applicable. If specific criteria are not available, professional judgment will be applied. Data that may not or do not meet current DQO 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 QAPP. 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 information recording or transcription; data reduction and manipulation for evaluation, review, approval, and reporting; and the process of document tracking, storage, and retrieval of all information obtained during sample collection and analysis. The project data manager will oversee the data handling and management processes for all forms of data collected. The project team will provide the processes and guidelines for sample tracking, storage, access, delivery, and reporting of new chemical analytical, geologic, and spatial data generated by investigation operations. Key data management objectives include the following:

- Apply well-documented data validation modifications to the electronic data
- Manage sample data using a unique sample identification number that conforms to the sample labeling specified in Section 5.11.2 of the FSP (Appendix A-1 of the RIWP [CH2M, 2018])
- Establish a sample inventory of data collected and provide methods of sample inventory reconciliation
- Store and provide sample-specific attributes, including location identifier, sample type, sample media, depth, date, and target study area in a manner compatible with ArcGIS format
- Provide reporting and delivery formats to support data analysis, site characterization, risk assessment, modeling, and spatial analysis
- Review laboratory data and verification are performed by a qualified laboratory analyst at the contracted laboratory before releasing data electronically to the consultant

Field personnel will record all raw field data and measurements into an appropriate format for reporting purposes. The records of all data reduction calculations must be kept on the project-specific field forms or field logbook. Data recorded on equipment memory or removable memory cards such as photographs or GIS data will be downloaded at the end of the work day onto a laptop computer, and then uploaded to the CH2M server to create a backup of the electronic field data. The FTLs will ensure the field personnel appropriately and correctly enter field data into the project-specific field forms and field logbooks as well as internal project database, which is compatible with ArcGIS.

All data entered into any database system will be verified by the FTL or their designee by printing a hard copy of all field information entered (for example, water level, purge volume, and field water-quality constituents, and portable GC/MS measurements) and comparing against raw data values contained on the field form.

The laboratory analyst will convert all raw values produced in the laboratory into reportable values. The records of all data reduction calculations must be kept on the appropriate laboratory worksheet. If the final values are not generated by direct-reading instruments or if a computer analyst performs all necessary data reduction of the raw data, the laboratory analyst will record the final values on computer-generated laboratory worksheets. All strip charts and chromatograms must be labeled, dated, and initialed by the analyst performing the analysis. Each laboratory worksheet bears a unique run-ID number. This run-ID number is part of a multiple index system used by the laboratory information management system (LIMS) to identify the samples and constituents performed for an individual worksheet. The analyst will also verify that reagent spikes, blanks, check standards, and duplicates are within acceptable limits. If all QC samples are within acceptable limits, the analyst will submit the worksheets for processing into the LIMS. The LIMS stores the data until all requested analyses are complete; the data are then transferred to the final electronic deliverable (Labspec 7 and EQuIS EDD file formats) for transmittal to CH2M. An electronic deliverable will be provided by the contracted laboratory in a Microsoft Excel format following the guidelines/example for Labspec 7 (Appendix A). All laboratory EDDs will be uploaded into the EQuIS database system for maintaining, reporting, and archiving.

On receipt, all field data are printed and checked for anomalies by the project data manager and QA/QC manager. The project data manager and QA/QC manager will also review the laboratory data to identify any anomalies requiring follow-up with field team members or the laboratory.

All test runs (standards, blanks, and samples) made on the field portable GC/MS are stored in digital form on internal memory cards. The test runs are identified by type (such as standard, blank, sample, and duplicate), matrix (such as soil, water, soil gas, and indoor air), sample name, date, time, injection volume, and analysis number (sequential number assigned daily by the GC/MS). At the conclusion of each day, data will be transferred from the GC/MS to a personal computer for backup, and a hard copy of each analysis will be printed for retention in a backup file.

The CH2M team will retain the entire project file as hard copy and/or electronic data on CH2M project servers until the completion of all project activities. Thereafter, all data will be provided to USACE/VHA and archived by CH2M 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). Analytical results and sample location data collected during the RI will be entered into a GIS database for mapping and publication, and the GIS metadata and files will be provided to USACE/VHA and archived by CH2M in electronic format for transmittal to EPA and UDEQ, if requested.

The official administrative record file for the site (hardcopy records) is located at the Salt Lake City Public Library – Anderson-Foothill Branch, in Salt Lake City, Utah. Compact disc 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, lab data, and other published project information for their records.

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 onsite technical system audit (TSA) of each laboratory will be performed, 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 onsite evaluation of a measurement or data collection system. TSAs for project activities are performed to verify that requirements (that is, 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. Each item is defined in the approved QAPP, which provides the basis for the TSA. Any undocumented or unauthorized deviations from the QAPP and corrective action are noted in the written lab 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 CH2M project personnel, subcontractors, consultants, VHA, and USACE project personnel will be responsible for identifying project deficiencies and notifying the QCSM. Any defects and deficiencies identified will be documented in the daily report and added to a tracking system (for example, electronic spreadsheet). The QC officer will describe any deficient items and the change in status of any deficiencies in the daily report and in a non-conformance 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, but not limited to, 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 ensure that the following corrective actions for the following non-conformances are implemented:

- Evaluate all reported non-conformances
- Control additional work on non-conforming items
- Determine disposition or action to be taken
- Maintain a log of non-conformances
- Review non-conformance reports
- Evaluate disposition or action taken
- Monitor inclusion of non-conformance reports in final site documentation and document control
- Notify the PM, who will notify USACE of non-conformances and corrective actions. USACE will then notify VHA.

The FTL will guarantee that no additional work dependent on the non-conforming activity is performed until the non-conformance is corrected. Also, the FTL will implement corrective action as initiated by the QCSM. Each non-conformance 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 PM by the laboratory QC officer. 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, but are not limited to, the following instances:

- 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 intra-laboratory or inter-laboratory 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 non-compliant 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 non-conformance with QA protocols. The report will summarize each non-conformance condition. A copy of the reports will be submitted with the final data package. The QA/QC manager in consultation with the program manager 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 of OU-2. These reports will be written by the PM or designee and submitted to USACE and VHA.
- **Data Summary Reports** – Communicate the conclusions of individual investigation events for OU-2 and any laboratory analysis associated with the event. These reports will be written by the PM or designee and submitted to USACE and VHA.
- **Data Validation and Usability 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 of OU-2. Reports may pertain to field data collection and health and safety events. These reports will be written by the PM or QCSM and submitted to USACE and VHA.
- **Laboratory Audit Reports/Corrective Action Reports** – 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 PM, USACE, and VHA.
- **Remedial Project Manager 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 OU-2 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 for OU-2 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 EPA/540/G-89/004-OSWER Directive 9355.3-01 *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, identified vertical and horizontal extent of contamination, geologic data interpretations, and ecological and baseline risk assessment results. CH2M 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 CH2M to USACE and VHA, with distribution by VHA to EPA and UDEQ Remedial Project Managers.

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, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Reports	Frequency	Provided to	Format	Prepared by
Progress reports	Monthly	USACE	Written	PM
Site investigation reports, field summary reports (that is, data summary reports), data validation and usability reports, interim RI Report, and RI Report	As needed	USACE and VHA, with distribution by VHA to EPA and UDEQ	Written	CH2M
Nonconformance reports	As needed	USACE and VHA, with distribution as needed by VHA to EPA and UDEQ	Written	PM, QCSM
Laboratory audit (TSA)/CARs	As scheduled	USACE and VHA, with distribution by VHA as needed to EPA and UDEQ	Written	Project chemist or designee
Field (QAPP) implementation audit / CARs	As scheduled, at least once during field work	USACE and VHA, with distribution by VHA as needed to EPA and UDEQ	Written	QCSM
Remedial Project Manager Meetings	Quarterly	USACE and VHA, with distribution by VHA to EPA and UDEQ	Verbal, with minutes	CH2M

Notes:
CAR = corrective action report
CH2M = CH2M HILL, Inc.
EPA = U.S. Environmental Protection Agency
PM = project manager
QAPP = quality assurance project plan
QCSM = quality control systems manager
RI = remedial investigation
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 QA/QC manager, 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 Section 3 and Section 5 of the FSP (Appendix A-1 of the RIWP [CH2M, 2018]) so that defensible and justifiable data are obtained. Integrity of information and field activity data will be the responsibility of the 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 non-conformity reports) will be assembled for review and will be verified by a peer review process. Example field forms are provided in Appendix A of the FSP (Appendix A-1 of the RIWP [CH2M, 2018]). A sample chain-of-custody form is included in Appendix B. If possible, any inconsistencies discovered will be resolved immediately by seeking clarification from the field personnel responsible for data collection. Integrity of the field sample custody is accomplished according to the sample custody procedures previously defined in Section 3.3. The field documentation verification process is summarized in Table 5-1.

5.1.2 Laboratory Data Reduction, Review, and Approval

The laboratory review of Contract Laboratory Program 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 but is not limited to the following:

- Instrument calibrations
- Raw data
- Transcribed data
- Calculations including calculations of precision, accuracy, representativeness, completeness, comparability, and sensitivity

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, corrective action reports, the results of re-analysis, sample holding times, and sample preservations will be evaluated.

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

Quality Assurance Project Plan, OU-2 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	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	I	FTL
	Lab chain of custody indicates any errors, signatures signifying acceptance of custody	E	Lab sample custodian
Sample location data (GPS coordinates)	All sample locations have associated northings, eastings, elevation	I	FTL
Field originated NCRs/CARs	When required, properly completed with appropriate corrective action specifies and signatures where required; properly filed	I	QCSM
Field summary reports	Submitted on weekly basis, properly archived on server, each day of work week accounted for, all information present	I	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	QCSM
Sample receiving document	Lab verified against chain of custody	E	Lab sample custodian
Draft lab results	All samples have results as requested, IDs match chain of custody, all QC present and reported as per QAPP	E	Lab 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	QCSM 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.	I	Project chemist during data validation stage
	Surrogate and internal summary forms (where appropriate), initial and continuing calibration summary and raw data	E	Lab QA manager designee
Lab originated NCRs/CARs	When required, properly completed with appropriate corrective action specifies and signatures; properly filed	E	Lab QA manager or lab PM
Memo to document QAPP modifications or deviations	Verify that all QAPP modifications are documented	I	QCSM
Analytical EDDs	Verify that all SDGs are reported in specified format and have required deliverables	E	Lab QA/QC Manager or lab PM

Notes:
CAR = corrective action report
EDD = electronic data deliverable
FTL = field team leader
GPS = geographic positioning system
ID = identification
LCS = laboratory control samples
MS/MSD = matrix spike/matrix spike duplicate
NCR = non-conforming report
PM = project manager
QA = quality assurance
QAPP = quality assurance project plan
QC = quality control
QCSM = quality control systems manager
SDG = sample delivery group
TSA = technical system audit

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.

Table 5-2. Laboratory Data Qualifiers
Quality Assurance Project Plan, OU-2 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 method detection 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 quality assurance project plan specified reporting limit.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control 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.

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, re-analysis 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.

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 CH2M to ensure that the data collected meet the requirements specified in Section 3.

The *Guidance for Evaluating Performance Based Chemical Data* (PB review) (USACE, 2005) and the EPA Contract Laboratory Program National Function Guidelines (NFGs) for data validation 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 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)" [EPA, 2017a, 2017b]. 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 quality control 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, CH2M will conduct data validation using the calibration and QC requirements specified in Section 3 for those specified in the guidelines. Data validation will not be performed on the geotechnical sample 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 will receive a (EPA Stage 2B) validation review (EPA, 2009), and 10 percent of the samples for each analysis performed will receive a (EPA Stage 4) validation review.

Laboratory data validation will be conducted by a CH2M 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 CH2M, and the software program 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 of changed data qualifiers will be generated from the data validation software program.

Data will be evaluated relative to the criteria established in Section 5.1. The flagging criteria that will be applied is summarized in Tables 5-4 and 5-5. The data validation and usability report identifies any non-conformances and explains any limitations on data use.

5.3 Reconciliation with User Requirements (D3)

The QCSM is ultimately responsible for data quality. All data quality issues concerning field sampling efforts, laboratory analysis, data validation, database management, and data reporting will be referred to the QCSM.

5.3.1 Usability Report

The project chemist will prepare the data validation and usability 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 (for example, 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

The data validation and usability 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 data validation and usability report will be provided as an appendix to the interim RI report.

5.3.2 Project-specific Measurement Performance Criteria

The project-specific MPC are summarized in Tables 3-2 through 3-11.

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

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Validation Step Tier 2B/4	Validation Input	Description	Responsible for Validation (Name, Organization)
2B/4	Field logbook	Sampling protocols followed as per QAPP, appropriate QC samples collected, proper preservation	FTL
2B/4	Field analytical results and logbook	Calibrations, blanks, duplicates all recorded and meet criteria	FTL
2B/4	Sample location data (GPS coordinates)	Northings, eastings and elevations traceable to all sample locations	FTL
2B/4	Field originated NCRs/CARs	All issues properly documented, corrective actions implemented and effective	FTL
2B/4	Field surveillance report	All issues properly documented, corrective actions implemented and effective	FTL
2B/4	Chain-of-custody forms; sample receiving document	Sample IDs for lab data match the reported data, all samples have data reported for requested analysis unless noted in the narrative	Project Chemist/QCSM
2B/4	Analytical data package Lab SOPs/reference methods QAPP MPC	Holding times met all method criteria	Project Chemist/QCSM
2B/4		Review of dilutions and re-analyses results against reported data; when multiple analyses appropriate run was reported, proper units are reported	Project Chemist/QCSM
2B/4		Calibrations analyzed at required frequency and met criteria	Project Chemist/QCSM
2B/4		Comparison of QC sample results (such as surrogate, internal standards, spikes, and blanks) all match criteria in method and QAPP	Project Chemist/QCSM
2B/4		Blanks free of contamination; if analytes present > RDL samples properly qualified if sample concentration < 10× blank concentration	Project Chemist/QCSM
2B/4		Detection limits, project action limits met	Project Chemist/QCSM
4		Recalculation of instrument and sample results from the laboratory instrument responses, and comparison of recalculated results to laboratory reported results. Sample results are evaluated by checking each (or selected) instrument output (for example, chromatograms, mass spectra, atomic emission spectra data, instrument background corrections, interference corrections) for correct identification and quantitation of analytes (for example, peak integrations, use of appropriate internal standards for quantitation, elution order of analytes, and interferences)	Project Chemist/QCSM
2B/4	Laboratory surveillance report	All issues properly documented, corrective actions implemented and effective	Project Chemist/QCSM
2B/4	Lab originated NCRs/CARs	All issues properly documented, corrective actions implemented and effective	Project Chemist/QCSM
2B/4	Memo about QAPP modifications	All issues properly documented, corrective actions implemented and effective	Project Chemist/QCSM
2B/4	Analytical EDDs	All data reported in excel format; EDD verified against hard copy lab report	Project Chemist/QCSM

Notes:
CAR = corrective action report
EDD = electronic data deliverable
GPS = geographic positioning system
FTL = field team leader
ID = identification
MPC = measurement performance criteria
NCR = non-conforming report
QAPP = quality assurance project plan
QCSM = quality control systems manager
RDL = representative detection limit
SOP = standard operating procedure

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

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Quality Control Check	Evaluation	Data Flag	Samples Affected
Holding Time	Holding time exceeded for extraction or analysis	J positive results; UJ non-detects	Sample
	Holding time exceeded by a factor of two	J positive results; R non-detects	
Sample Preservation	Sample not preserved; or not analyzed within 7 days from sample collection (SW8260C VOA)	J positive results; UJ non-detects; R non-detects based on professional judgement	Sample
Sample Integrity	Bubbles (> pea size) in VOA vial used for analysis	J positive hits; UJ non-detects	Sample
Temperature	> 6°C	J positive results; UJ non-detects	All samples in same cooler
Initial Calibration	RRF < 0.050 but > 0.01	J positive results, UJ non-detects	All associated samples in analysis batch
	RRF < 0.01	J positive results, R non-detects	
	Method specified calibration criteria exceeded (for example %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 non-detects	
Calibration Verification (second-source and continuing calibration verification)	RRF < 0.050 but >0.01	J positive results, UJ non-detects	All associated samples in analysis batch
	RRF < 0.01	J positive results, R non-detects	
	%D > 20.0% with high recovery	J positive results	
	%D > 20.0% with low recovery	J positive results, UJ non-detects	
Laboratory Control Sample	%R > UCL	J positive results	All samples in preparation batch
	%R < LCL	J positive results, UJ non-detects	
	%R < 10%	J positive results; R nondetects	
Calibration Blank Method Blank	Convert blank concentration to soil units, if applicable; multiply the highest blank concentration by 5	U positive sample results < 5× highest blank concentration	All samples in preparation batch or analytical batch, whichever one applies, associated with method blank or calibration blank
Equipment Blank Field Blank			All samples, same site, matrix and date (water) or all samples, same site, matrix (soil) associated with equipment blank

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

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Quality Control Check	Evaluation	Data Flag	Samples Affected
Trip Blank			All samples shipped in the same cooler as the trip blank
Matrix Spikes			
% Recoveries	%R > UCL	J positive results	MS analytes in parent sample and field duplicate, if any
	%R < LCL	J positive results, UJ nondetects	
	%R < 10%	J positive results; R nondetects	
RPDs	RPD > UCL	J positive results	
Surrogates			
SW8081B/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	Two or more surrogates in same fraction with %R > UCL	J positive results	All analytes in same fraction in sample
	Two or more surrogates in same fraction with %R < LCL but not < 10%	J positive results; UJ nondetects	
	Two or more surrogates in same fraction with %R < LCL and < 10%	J positive results; R nondetects	
Internal Standards	Area > UCL	J positive results	Associated analytes in sample
	Area < LCL but not < 10%	J positive results; UJ nondetects	
	Area < 10%	J positive results; R nondetects	
Field Duplicates	Concentration of reported analytes are > 5× the reporting limit in either sample and RPD > UCL (30% for water samples; 50% for soil samples)	J positive results	Field duplicate pair
	One or both sample results < 5× the reporting limit and a difference of ±2× the reporting limit for water (±4× 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, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Quality Control Check	Evaluation	Data Flag	Samples Affected

Notes:
All QA/QC criteria are included in Tables 3-2 through 3-13 and will be used for verification criteria.
Organic methods include SW8081B, SW8260C, SW8270D, and TO-15.
Spike recovery limits do not apply when 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.
Qualifier may not apply in cases where a surrogate coelutes with a non-target analyte.
Qualifier may not apply in cases where low surrogate recoveries are because of sample dilution.

%D = percent difference
%R = percent recovery
°C = degree(s) Celsius
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 quality assurance project plan specified reporting limit.
LCL = lower control limit
MB = method blank
MS = matrix spike
QA = quality assurance
QC = quality control
R = The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
RPD = relative percent difference
RRF = relative response factor
RSD = relative standard deviation
U = The compound was analyzed for, but was not detected above, the reported method detection limit.
UCL = upper control limit
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.
VOA = volatile organic analytic

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

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Quality Control Check	Evaluation	Flag	Samples Affected
Holding Time	Holding time exceeded for extraction, digestion, or analysis	J positive results; R non-detects for mercury; UJ non-detects for all other analytes	Sample
	Holding time for digestion or analysis exceeded by a factor of 2	J positive results; R non-detects	
Sample Preservation	Sample preservation requirements not met (If sample preservation was not done in the field, but was performed at the laboratory upon sample receipt, no flagging is required)	J positive results; R non-detects	Sample
Initial Calibration (multi-point only)	Correlation coefficient ≤ 0.995	J positive; UJ non-detects	All associated samples in analytical batch
Calibration Verification (initial calibration verification, continuing calibration verification)	%R > UCL	J positive results	All associated samples in analytical batch
	%R < LCL	J positive results, UJ non-detects	
Interference Check Sample (SW6010C/SW6020 only)	%R > UCL	J positive results	All associated samples in analytical batch
	%R < LCL	J positive results; UJ non-detects	
Laboratory Control Sample	%R > UCL	J positive results	All samples in preparation batch
	%R < LCL	J positive results; UJ non-detects	
	%R < 40% (< 20% for Antimony or Silver)	J positive results; R nondetects	
Calibration Blank Method Blank	Convert blank concentration to soil units, if applicable; multiply the highest blank concentration by 5	U positive sample results < 5× highest blank concentration	All samples in preparation batch or analytical batch, whichever one applies, associated with method blank or calibration blank
Equipment Blank Field Blank Ambient Blank	Convert blank concentration to soil units, if applicable; multiply the highest blank concentration by 5	U positive sample results < 5× highest blank concentration	All samples, same site, matrix and date (water) or all samples, same site, matrix (soil) associated with equipment blank
Matrix Spikes			
% Recoveries	%R > UCL	J positive results	MS analytes in parent sample and field duplicate, if any
	%R < LCL	J positive results, UJ nondetects	
	%R < 30%	J positive results; R nondetects	
RPDs	RPD > UCL	J positive results	

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

Quality Assurance Project Plan, OU-2 Remedial Investigation 700 South 1600 East PCE Plume, Salt Lake City, Utah

Quality Control Check	Evaluation	Flag	Samples Affected
Dilution Test	If concentration is > 50 times the MDL and percent difference > UCL	J positive results, UJ non-detects	Parent sample result(s)
Post-digestion Spikes/Recovery Test (metals only)	Spike results indicate performance of MSA required, but MSA not done		All samples in digestion batch if MSA not performed
	%R > UCL	J positive	
	%R < LCL	J positive results, UJ nondetects	
Field Duplicates	Concentration of reported analytes are > 5× RL in either sample and RPD > UCL (30% for water samples; 50% for soil samples)	J positive results	Field duplicate pair
	One or both sample results < 5× RL and a difference of ±2× the reporting limit for water (±4× for soil).	J positive; UJ nondetect	
Laboratory Sample Duplicates	Concentration of reported analytes are > 5× RL in either sample and RPD > 20%	J positive results	Parent sample result(s)
	One or both sample results < 5× RL and a difference of ±2× the reporting limit	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 SW6010C, SW6020, SW7470A, SW7471B, and General Chemistry Parameters.
Spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more.

%R = percent recovery
°C = degree(s) Celsius
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 quality assurance project plan specified reporting limit.
LCL = lower control limit
MS = matrix spike
MSA = method of standard addition
QA = quality assurance
QC = quality control
R = The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
RPD = relative percent difference
U = The compound was analyzed for, but was not detected above, the reported method detection limit.
UCL = upper control limit
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.

SECTION 6

References

CH2M HILL, Inc. (CH2M). 2017. *Data Management Plan, OU-2 Remedial Investigation, 700 South 1600 East PCE Plume, Salt Lake City, Utah.* Final. May.

CH2M HILL, Inc. (CH2M). 2018. *Remedial Investigation Work Plan, OU-2 Remedial Investigation, 700 South 1600 East PCE Plume, Salt Lake City, Utah.* Final. February.

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). 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). 2002. *EPA Guidance for Quality Assurance Project Plans*. EPA QA/G-5. EPA/600/R-02/009. December.

U.S. Environmental Protection Agency (EPA). 2005. *Uniform Federal Policy for Quality Assurance Project Plans*. EPA/505/B-04/900A. March.

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.

Appendix A
Electronic Data Deliverable Specification (LabSpec7)

February 2011 Revision
Laboratory Electronic Deliverable Format forCH2M HILL, version 4.31
Sources: Vito D'Aurora/RDD, Ed Svastits/GNV

Electronic Data Deliverable Format for CH2M HILL

The electronic data deliverable (EDD) file from the laboratory will be a comma-delimited ASCII (CDA) file in the format listed below. There will be one file per hard copy report and the filename of the EDD file will be in the format REPORTID.txt or REPORTID.csv, where REPORTID is the hard copy report identifier of sample delivery group.

The first row of the EDD will contain the 48 field name values as listed in the EDD Specification Table

The EDD Specification Table lists the attributes of the columns for each row of the CDA file. The fields should be reported in the order indicated.

The **Data Type** column describes the value in the field as either text (alphanumeric), number (numeric only), date (format: mm/dd/yyyy), or time (24-hour format hh:mm). If the field is conditional or optional and there is no value to be reported, report a null (i.e., no) value. For a text field, do not report a zero-length string (i.e., "").

The **Data Length** column contains the maximum length of a text value for the particular data field.

The **Rqmt** column contains a code indicating whether the value is required (R) for all rows, optional (O) for all rows, or conditional (C) and depends on the type of result reported.

The VVL (Valid Value List) column contains a flag to indicate whether the data field has (Y) or does not have (N) a valid value list provided by CH2M HILL associated with it.

February 2011 Revision
Laboratory Electronic Deliverable Format for CH2M HILL, version 4.31
Sources: Vito D'Aurora/RDD, Ed Svastits/GNV

Modification Notes:

Changes as of September 2006 Revision:

1. Change the Requirement for CAS to R (Field No. 21).

Changes as of February 2011 Revision:

1. Add new field to the end of labspec named "Spike_Added" (Field No. 48).
2. Add new field to end of labspec named 'Surr_Spike_Units' (Field No. 49).
3. Add new field to end of labspec named "LOD" (Field No. 50).
4. Add new field to end of labspec named "LODAdjusted (Field No. 51).
5. FieldID expanded to 30 characters.

EDD Specification Table						
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**VVL**	**Description and Comments**
1	VersionCode	text	15	R	Y	Code identifying the version of the EDD deliverable.
2	LabName	text	10	R	Y	Identification code for the laboratory performing the work. This value is used to distinguish among different facilities.
3	SDG	text	15	R	N	Sample delivery group designation. Always populated for all samples, including QC.
4	FieldID	text	30	R	N	Client sample ID as appears on COC with lab-assigned suffixes to make it unique. Suffixes to add: "DL" (dilution), "RE" (reanalysis), "DUP" (laboratory duplicate), and "CF" (confirmation). For multiple dilutions or re-analyses of the same sample append the replicate number after the suffix (i.e "RE", "RE1", "RE2", etc.) If the sample identifier on the COC and the prefix/suffix is greater than 20 characters, abbreviate the value but make it unique. For laboratory QC samples (i.e., method blanks, lab control samples), use a unique lab sample identifier.
5	NativeID	text	30	R	N	Client sample ID, exactly as on the COC. No prefix or suffix allowed on client sample IDs. Used to identify the native sample from which other samples are derived (e.g., QAQCType = "LR", "MS", or "SD"). For laboratory QC samples (i.e., method blanks, lab control samples), use the FieldID value that was assigned. However, for lab blank spike duplicate samples, use the FieldID value that was assigned to the associated lab blank spike sample.

EDD Specification Table						
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**VVL**	**Description and Comments**
6	QAQCType	text	2	R	Y	This is the code for the sample type. Any field sample that is not used as lab QC and is not otherwise marked on the COC should have the designation of "N" (normal field sample). No suffix allowed (i.e., do not add numbers as suffixes to the QAQCType values as is called for in the ERPIMS guidelines). Note that if all analyses for a given sample are diluted, then the first dilution should be designated as the normal sample.without LRType of DL .Also note for a laboratory duplicate the QA/QCType should be LR with a LRType of "D". (see LRType, below).
7	LRType	text	3	C	Y	This is the code for laboratory replicate sample type. Values are: blank (if QAQCType value is not "LR" and sample is not a dilution), "DL" (dilution), "RE" (re-analysis), "D" (inorganic duplicate), "CF" (confirmation). For multiple dilutions or re-analyses of the same sample, append the replicate number after the LRType value (i.e., "RE", "RE2", "RE3", etc.).
8	Matrix	text	5	R	Y	Sample matrix code. Valid values are as follows: "AIR", "WATER", "SOIL", unless otherwise provided by the project data manager and marked on the COC. The use of "liquid", "solid", etc. for lab QC is not allowed.

EDD Specification Table						
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**VVL**	**Description and Comments**
9	LabSampleID	text	17	R	N	Laboratory sample ID that is assigned by the laboratory. For dilution, reextractions and confirmation results a suffix will be assigned as follows: "DL" (dilution), "RE" (reanalysis), "D" (laboratory duplicate), and "CF" (confirmation). For multiple dilutions or re-analyses of the same sample append the replicate number after the suffix (i.e "DL", "DL1", "DL2", etc.). Ex: "D97-11111RE" is acceptable.
10	AnalysisMethod	text	20	R	Y	Analysis method code. This is the identifier of the analytical method that was performed on the sample. Example: SW8260B. Generic names such as "EPA" should not be used.
11	ExtractionMethod	text	20	R	Y	Preparation method code. A value in this field is required. If the preparation is described in the method, use "METHOD". If there is no separate preparation required, use "NONE". Note that Total and Dissolved metal analyses are differentiated by the value in this column. Note that Total, TCLP, and SPLP analyses are now differentiated by the value in the LeachMethod column (see below).
12	SampleDate	date		C	N	Date of sample collection. Value is required for all samples sent to the laboratory and samples derived from those samples. Format: mm/dd/yyyy
13	SampleTime	time		C	N	Time of sample collection. Value is required for all samples sent to the laboratory and samples derived from those samples. 24-hour format: hh:mm
14	ReceiveDate	date		C	N	Date of sample receipt in the lab. Value is required for all samples sent to the laboratory and samples derived from those samples. Format: mm/dd/yyyy
15	ExtractDate	date		C	N	Date of sample preparation (extraction or digestion). Value is required if the ExtractionMethod field value is other than "NONE". Format: mm/dd/yyyy

EDD Specification Table						
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**VVL**	**Description and Comments**
16	ExtractTime	time		C	N	Time of sample preparation. Value is required if the ExtractionMethod field value is other than "NONE". 24-hour format: hh:mm
17	AnalysisDate	date		R	N	Date of sample analysis. Value is required for all records. Format: mm/dd/yyyy
18	AnalysisTime	time		R	N	Time of sample analysis. Value is required for all records. 24-hour format: hh:mm
19	PercentSolids	number		R	N	Percent solids within the sample. Should be zero for water samples.
20	LabLotCtlNum	text	10	C	N	Identifier of an autonomous group of environmental samples and associated QC samples **prepared** together. For example, its value can be a digestion or extraction batch ID. If there is no separate extraction or preparation performed, leave this field blank.
21	CAS	text	20	R	N	CAS number of analyte, if available.
22	ParamID	text	12	R	Y	Parameter identifier code for the parameter listed in the Analyte field.
23	Analyte	text	60	R	N	Name of analyte, chemical name.
24	Result	text	10	R	N	Result of the analysis. Surrogate analytes will be reported in units of percent. All others will be reported in sample concentration units. If undetected, report the MDLadjusted, LODadjusted or RLadjusted, depending on the project. (Reported as a text field to preserve significant figures.)
25	ExpectedValue	number		C	N	"100" for surrogates; "0" (zero) for blanks; spike level plus parent result for LCS, and MS/MSD; parent value for lab duplicate; etc.
26	Units	text	10	R	Y	Units of measure used in the analysis. Report "PERCENT" for surrogate analytes and concentration units for all others.

EDD Specification Table						
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**VVL**	**Description and Comments**
27	Dilution	number		R	N	Total dilution reported in the analysis. Default value should be 1 (one). This value should reflect changes to sample preparation amounts as defined by the method (e.g., less sample used for standard VOC analysis).
28	MDL	number		C	N	Minimum detection limit adjusted for preparation and dilution. Note that this value may be the method detection limit or the instrument detection limit, depending on the method and the project requirements. This value is **not** adjusted for percent moisture.
29	RL	number		C	N	Reporting limit adjusted for preparation and dilution. Value is **not** adjusted for percent moisture. Equivalent to QSM LOQ.
30	LabQualifier	text	6	R	N	Lab qualifier for the results, as reported on the hard copy. Use "=" as first (or only) qualifier value for detected results if there are no other qualifiers for the result.
31	Surrogate	text	1	R	Y	Is the chemical a surrogate? Report "Y" for yes or "N" for no.
32	Comments	text	240	O	N	Comment field
33	ParValUncert	text	16	C	N	Radiological parameter value uncertainty.
34	Recovery	number		C	N	Percent recovery for MS, SD, LCS, LCSD, and surrogate compounds.
35	LowerControlLimit	number		C	N	Lower control limit value for spiked compounds, expressed in units of Percent. A value in this field is required if there is a value in the Recovery field (Field No. 34).
36	UpperControlLimit	number		C	N	Upper control limit value for spiked compounds, expressed in units of Percent. A value in this field is required if there is a value in the Recovery field (Field No. 34).
37	Basis	text	1	R	Y	Weight basis for soil (or solid) sample analysis. Use "D" for dry-weight basis, "W" for wet-weight basis, or "X" if not applicable.
38	ConcQual	text	1	R	Y	Concentration qualifier. Use "=" for detects, "J" for estimated value (value between detection limit and reporting limit), "U" for a nondetected result, or "E" for a result that has exceed the calibration range.

EDD Specification Table						
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**VVL**	**Description and Comments**
39	MDLAdjusted	number		C	N	Minimum detection limit adjusted for preparation, dilution **and percent moisture**. See the description of the MDL field (Field No. 28) for an explanation of the contents of this field.
40	RLAdjusted	number		C	N	Reporting limit adjusted for preparation, dilution **and percent moisture**. Equivalent to QSM LOQ
41	SampleDescription	text	30	C	N	Full sample identifier value as it appears on the COC. In some cases, this may be the name of the sampling location instead of the sample. Required for all samples that are either collected in the field and specified on the COC, or derived from samples that are collected in the field and specified on the COC.
42	LeachMethod	text	20	R	Y	Analytical method used for leaching the sample. This applies to TCLP, SPLP, or other leaching or pre-extraction leaching procedures. Use "NONE" if the sample was not leached.
43	LeachDate	date		C	N	Date that the leaching method was performed (start date for multi-date leaching procedures). Value is required if the LeachMethod field value is other then "NONE". Format: mm/dd/yyyy.
44	LeachTime	time		C	N	Time that the leaching procedure started. Value is required if the LeachMethod field value is other then "NONE". 24-hour format: hh:mm.
45	LeachLot	text	10	C	N	Identifier of an autonomous group of environmental samples and associated QC samples **leached** at the same time. Value is required if the LeachMethod field value is other then "NONE". If the sample was not leached, leave this field blank.
46	AnalysisLot	text	10	R	N	Identifier of an autonomous group of environmental samples and associated QC samples **analyzed** together. A value in this field is mandatory (i.e., it should not be blank).
47	CalRefID	text	10	C	N	Identifier of a group of environmental and QC samples linked by a common set of calibration records. All results with the same CalRefID value will have had the same initial calibration run.

EDD Specification Table						
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**VVL**	**Description and Comments**
48	Spike_Added	number	18	C	N	Concentration of an analyte spiked into a sample. Populate for MS, SD, BS,BD, and surrogate compounds (maximum 6 decimal places).
49	Surr_Spike_Units	text	10	R	Y	Concentration unit for the surrogate spike added.
50	LOD	number		C	N	Limit of detection (QSM LOD) adjusted for preparation and dilution. Value is **not** adjusted for percent moisture.
51	LODAdjusted	number		C	N	Limit of detection (QSM LOD) adjusted for preparation, dilution **and percent moisture**.

Each row is uniquely identified by the values in the following fields:

- FieldID
- LabSampleID
- AnalysisMethod
- ExtractionMethod
- LeachMethod
- ParamID

If an analytical sample must be diluted or reanalyzed and reported in addition to the original analytical sample, the diluted or reanalyzed sample should have a FieldID value that is different that that of the original sample. This can be accomplished through the addition of a suffix to the original FieldID that establishes a new and unique FieldID for the associated records.

Example Valid Values

The project data manager will provide the laboratory with a list of valid values that the laboratory will use in constructing the EDD. Listed below are some example valid values.

Field Name	Valid Value	Meaning
VersionCode	4.20AFCEE3	Format 4.20, AFCEE data values. LabQualifier field contains

Field Name	Valid Value	Meaning
		the laboratory qualifier values defined in the AFCEE QAPP, version 3.0.
VersionCode	4.20EPACLP	Format 4.20, EPA data values. LabQualifier field contains the standard EPA CLP lab qualifiers.
QAQCType	N	Normal, environmental sample
QAQCType	LB	Laboratory method blank
QAQCType	MS	Laboratory matrix spike sample
QAQCType	SD	Laboratory matrix spike duplicate
QAQCType	LR	Laboratory replicate (, reanalysis, re-extraction and duplicate)
QAQCType	BS	Laboratory method blank spike
QAQCType	BD	Laboratory method blank spike duplicate
LRType	DL	First dilution sample
LRType	DL2	Second dilution sample
LRType	DL3	Third dilution sample
LRType	RE	First reanalysis/re-extraction sample
LRType	RE2	Second reanalysis/re-extraction sample
LRType	RE3	Third reanalysis/re-extraction sample
LRType	D	Inorganic duplicate sample
LRType	CF	First confirmation analysis sample

Field Name	Valid Value	Meaning
LRType	CF2	Second confirmation analysis sample
LRType	CF3	Third confirmation analysis sample
AnalysisMethod	SW8260B	Volatiles by method 8260B in EPA SW846.
AnalysisMethod	SW8270C	Semivolatiles by method 8270C in EPA SW846.
AnalysisMethod	SW6010B	ICP metals by method 6010B in EPA SW846.
AnalysisMethod	SW7060	GFAA Arsenic by method 7060 in EPA SW846.
ExtractionMethod	FLDFLT	Field filtration for dissolved metals analysis
ExtractionMethod	C3050	CLP-modified SW3050 acid digestion for metals analysis in soil samples.
ExtractionMethod	SW1311	TCLP extraction
ExtractionMethod	DISWAT	Distilled water extraction for analytes in soil samples.
ExtractionMethod	SW3510	Separatory funnel extraction
ExtractionMethod	SW3540	Soxhlet extraction
ExtractionMethod	TOTAL	Digestion of unfiltered waters for total metals analysis
ParamID	ACE	Acetone
ParamID	AS	Arsenic
ParamID	BHCGAMMA	gamma-BHC (Lindane)
ParamID	BZ	Benzene
ParamID	CDS	Carbon disulfide

Field Name	Valid Value	Meaning
ParamID	PB	Lead
ParamID	PHENOL	Phenol
ParamID	SE	Selenium
ParamID	TCE	Trichloroethene

Appendix B
Example Chain-of-Custody Documentation

Project/Contact Information		Total Number of Containers	Requested Analysis	THIS AREA FOR LAB USE ONLY		
Project #				**Lab #**	**Pg 1**	**of 1**
Project Name				**Lab PM**	**Custody Review**	
Report Copy to				**Log In**	**LIMS Verification**	
Company Name/Contact				**pH**	**Cust Seals Y N Ice Y N**	
				QC Level 1 **2 3**		

Sampling		Type		Matrix													Preservative	Cooler Temperature		
Date	**Time**	**Comp**	**Grab**	**Water**	**Soil**	**Air**	**Client Sample ID** (9 Characters Max)									**LAB QC**		**Alternate Description**		**Lab ID**

Sampled By	**Relinquished By**	
Received By	**Relinquished By**	
Received By	**Date/Time**	
Special Instructions		

Appendix C
Laboratory Quality Assurance Manuals

Empirical Laboratories, in Nashville, Tennessee, is the primary analytical laboratory. They will perform all analyses, with the exception of the following:

- Volatile organic compounds in soil gas (EPA Method TO-15)
- Fraction of organic carbon in soil (ASTM D2974)

The latter two methods will be subcontracted by Empirical to TestAmerica-Sacramento and Test America-Pittsburgh, respectively.

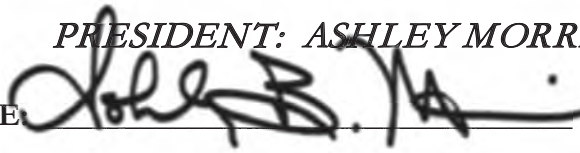
Quality assurance manuals for each laboratory are provided in this appendix.

Appendix C-1
Empirical Laboratories, LLC
Quality Manual

EMPIRICAL LABORATORIES, LLC
QUALITY MANUAL
EFFECTIVE DATE: 20161108
REVISION # 37

Empirical Laboratories, LLC (EL)
621 Mainstream Drive, Suite 270
Nashville, TN 37228
Phone: 615.345.1115
Fax: 866.417.0548

PRESIDENT: ASHLEY MORRIS

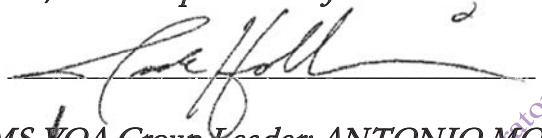
SIGNATURE:  **DATE: 20161108**

LABORATORY TECHNICAL DIRECTORS

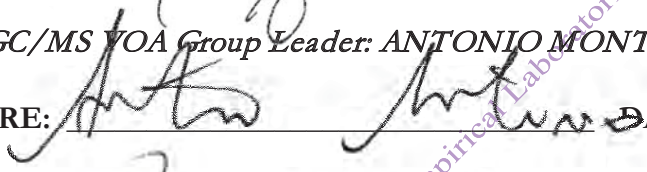
LAB DIRECTOR: RICK DAVIS

SIGNATURE:  **DATE: 20161108**

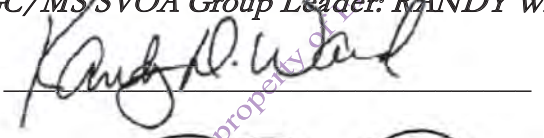
GC/LC Group Leader: JADE HOLLIMAN

SIGNATURE:  **DATE: 20161108**

GC/MS VOA Group Leader: ANTONIO MONTEIRO

SIGNATURE:  **DATE: 20161108**

GC/MS SVOA Group Leader: RANDY WARD

SIGNATURE:  **DATE: 20161108**

Metals/Wet Chemistry Group Leader: RICK DAVIS (Interim)

SIGNATURE:  **DATE: 20161108**

DATA QUALITY MANAGER

MARCIA K. McGINNITY

SIGNATURE:  **DATE: 20161108**

QAM_R37_20161108

- Section 1.2 updated to formally identify responsibility to ensure compliance with NELAC/TNI 2009 standards and DoD QSM 4.2/5.0 standards.
- Table 2.1 updated to clarify sections and SOP references.
- Section 2.5.3 title and contents updated to clarify QS05 includes handling of client complaints, corrective actions and feedback to clients.
- Added LCMS 8060 system to instrument listing.

QAM_R36_20160815

- Organizational chart and key personnel references. Includes Keith Blanchard as Director of Finance and Business Administration. Also revised Client Service Manager to Office Manager and updated responsibilities across upper management.
- Removed address for Florida administrative offices.

QAM_R35_20160630

- Organizational chart updated to include Donald Hensel as Extractions Group Leader and Amy Barnett as Data Review Manager.
- Certification listings updated in section 7.0 including NELAP primary state update to Florida and associated update to table 7.1.
- Section 2.5.5 updated for electronic signature handling.

QAM_R34_20150826

- Organizational chart updated to include Ashley Morris as President.
- Control chart section updated to reference QS15 for specific frequencies/details.
- Section 3.2 updated to reference "sample kits" instead of "bottle kits" and point to QS10 or QS11 for specifics
- Section 4.1 updated to generalized references with laboratory SOPs including specifics.
- Section 4.2 – move equipment maintenance logbook information to QS03 or QS08?
- Section 4.3 – updated to reference details of support equipment calibration within QS08. Table 4.1 removed.
- Section 4.4 – updated equipment listing.
- Section 5.0 – updated to reference QS03 for detailed processes.
- Section 6.0 – reference QS03 and QS02. Reference QS05 and QS15 for handling of client notifications.
- Section 7.0 - Updated for most recent certification listings.

Summary of Changes: Quality Manual
QAM_R33_20150109

- Organizational chart updated to reflect Group Leaders instead of organic and inorganic section supervisors. Removing references to sections supervisors throughout document.
- Adding IC2 instrumentation and Chromeleon software to table 4.2.
- Updated certification summary list.
- Removed references to sulfide by 9030B/9034 and SM4500 CN in list of certifications.

Summary of Changes: Quality Manual
QAM_R32_20140804

- Added 17025 and QSM 5.0 references in opening paragraph.
- Rotated graph 1.1.
- Added Owner/CEO/CFO and OM to section 1.2. Specified technical managers. Also specified AB notification if absence exceeds 35 days.
- Added OM to section 1.3 and updated chart 1.3.
- Section 2.3 Management Review requirements moved to QS15 with reference in QAM.
- Updated table 2.1 to include additional QSM5.0 required element and clarify locations
- Updated section 2.1.2 to indicate master table of SOPs and reference documents at TOC_SOPs_Documents_Cntrolled.xls.
- Updated section 2.1.3 to reference SOPs QS02, QS03 and QS06 for details. Also specified confidentiality and plans in event of sale or closure.
- Updated sections 2.2, 2.3 and 2.4 to reference SOP QS15 for specifications/processes.
- Updated section 2.5.2 to clarify training specifications and reference SOP QS03.
- Updated sections 2.5.4 and 2.5.5 to reference SOP QS06.
- Sections 4.3, 4.4 and Table 4.1 updated to reflect QSM 5.0 requirements and reference QS08.

- Table 4.2 updated to reflect added instrumentation
- Section 5.0 updated to remove detailed specifications and reference SOP QS03.
- Section 6.0 updated to remove detailed specifications and reference SOP QS02.
- Section 7.0 updated to include certification listing with expirations first, NJ NELAP scope in table 7.1 second and DOD ELAP scope in table 7.2 second. Also to indicate that detailed scopes for other state certifications are available upon request.

Changes to this Revision, R31 Dated 20131021

- Management Review requirements clarified in section 2.3.
- Preventive actions added to section 2.4.
- Reference to QS 12 added to section 2.5 Review of New Work.
- Ethics and Data Integrity form updated to include initials and indicate initials/signature to be used for documentation of initials/signature until annual employee signature log generated.
- Tables 2.3 and 3.1 removed and replaced with appropriate QS reference.
- Added BNA5 components to table 4.2 instrument listing.
- Updated certification listing in section 7.2.

Changes to this Revision, R30 Dated 20130715

- Introductory statement updated to indicate commitment to comply TNI 2009 standards.
- NELAC references updated to reflect TNI 2009 references.
- References to toxicology removed and organizational chart updated.
- Updated section 2.1.2 to reference SOP QS01 and rather than include details that are covered in the SOP.
- Updated section 2.2 to indicate timeframe for client notification of issues identified during internal audit.
- Section 2.3: Management Review updated to indicate inclusion of positive feedback and to include section on **Findings and Corrective Actions**.
- Section 2.5.2 Updated reference to DOCs to indicate completion required "prior to" performing, assessing or reporting a particular method or a component of a method.

Changes to this Revision, R29 Dated 20121115

- Section 2.5.5 "Electronic Initials/Signatures" updated.
- IDOC/DOC form 2.3 updated

Changes to this Revision, R28 Dated 20120730

1. Added Administrative Office contact information to cover page.
2. Added list of charts/tables/graphs/forms to table of contents page.
3. Updated section 1.2 to add statement concerning duties outside main responsibilities.
4. Updated quality manual table 2.1 to reference TNI standards with DoD quality systems clarifications/requirements.
5. Update holding time table 3.1 for "Analyze Immediately" parameters and to add missing parameters plus clarify VOA soil preservation options.
6. Updated instrument table 4.2 to include new headspace autosampler and new HPLC setup.
7. Added EDD content match to table 5.2.
8. Updated certification listing in tables 7.1 and 7.2.
9. Removed DL/LOD/LOQ definitions from section 7.0 as appropriately referenced in SOP QS08.

Changes to this Revision, R27 Dated 20110822

1. Add address to title page.
2. Replace DL/LOD/LOQ tables with analyte listing for NJ-NELAP and DoD-ELAP scopes
3. Update DoD references to 4.2
4. Update organization chart
5. Update listing of state certifications.

Changes to this Revision, R26 Dated 20110516

1. Detail is provided on the transmittal and storage of client results and proprietary information.
2. Section 2.2 (Internal Audits) has been edited to include information on how the laboratory handles client notification when when an internal audit casts doubt on the validity of the data produced.
3. Additional details on Internal Audits and timeframes for Corrective Actions have been added to Section 2.2
4. Table 3.1 (Method Preservation Summary), a clarification is added for the Holding Time labeled as "ASAP".
5. Reference to QS05 SOP added for qualifiers applied to data exceeding criteria in section 2.5.3

Changes to this Revision, R25 Dated 20110112

1. Removed old organizational chart from beneath updated organizational chart to allow accurate PDF version.
2. Added MEE/RSK-175 information to table 3.1.
3. Demonstration of Capability Form updated to allow better reflection of preparation and analysis information.

Changes to this Revision, R24 Dated 20101117

1. Added Group Leader Definition, LIMS Manager Definition and add Purchasing Manager to Lab Director responsibilities.
2. Metals - in-house preservation – wait 24 hours prior to digestion/analysis
3. Updated 1% data package review criteria to 10% with checklist added.
4. More detail added to the SOP process to indicate which documents are considered controlled and which are not.

Changes to this Revision, R23

1. Reference to QS02 removed.
2. Annual weight verification corrected to every 5 years.
3. MDL criteria updated to initial setup with annual verification.
4. Data package review changed to 1% annually.
5. Vinyl chloride water DL/LOD/LOQ divided by 2.

Changes to this Revision, R22

1. Section 1.0 has the revised and updated Organizational Chart
2. Section 2.1.2 has been updated to include information on SOP updates when changes are made within the laboratory prior to the SOP hardcopy being finalized
3. The reference for Customer Complaints SOP in section 2.3 has been changed to QS05
4. SOP QS12 in section 3.2 has been updated to SOP QS10 and SOP QS15 has been updated to QS14. There was an error in the original reference of the SOPs.
5. Table 3.1 has been updated and table 4.2 (calibration specifics) has been removed.
6. Tables in section 7 have been updated.
7. The certifications list in section 7.2 has been updated

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1.0 Company and Organization Information

1.1 Introduction

This manual is a compilation of the laboratory's implementation of overall requirements based upon management's commitment to ISO 17025, The NELAC Institutes (TNI) 2009 standards, EPA, DoD QSM 4.2, DoD QSM 5.0, specific State, and other regulatory requirements. The manual references overall quality systems practices and the sources to specific procedures and technical protocols as followed within the laboratory. This section presents information on the Company, its beliefs, mission, vision, and core values, specific laboratory personnel positions and the responsibilities which each provides in the implementation of the Quality Assurance Program and the execution of Quality Control activities. ***Quality Control begins at the lab bench with each analyst knowing the QC criteria for the analytical methods being performed and evaluating the analytical instrument calibrations and resulting data for compliance with these criteria. Each analyst has the responsibility and authority for halting an out-of-control analytical procedure, taking appropriate corrective action or seeking guidance from supervisory personnel. This process stands as the cornerstone of the Quality Assurance Program. The underlying principle at EL is a commitment to professionalism, ethical practices at all levels and in all phases of our business, and an on-going commitment to continuous improvements***.

The COMPANY'S **VISION** is to remain an ethical leader in the Environmental Laboratory Services Industry, striving to provide optimum customer satisfaction and superior data quality.

Our **MISSION** is to deliver the best-applied laboratory services that respond to our client's unique needs with the goal of exceeding their expectations.

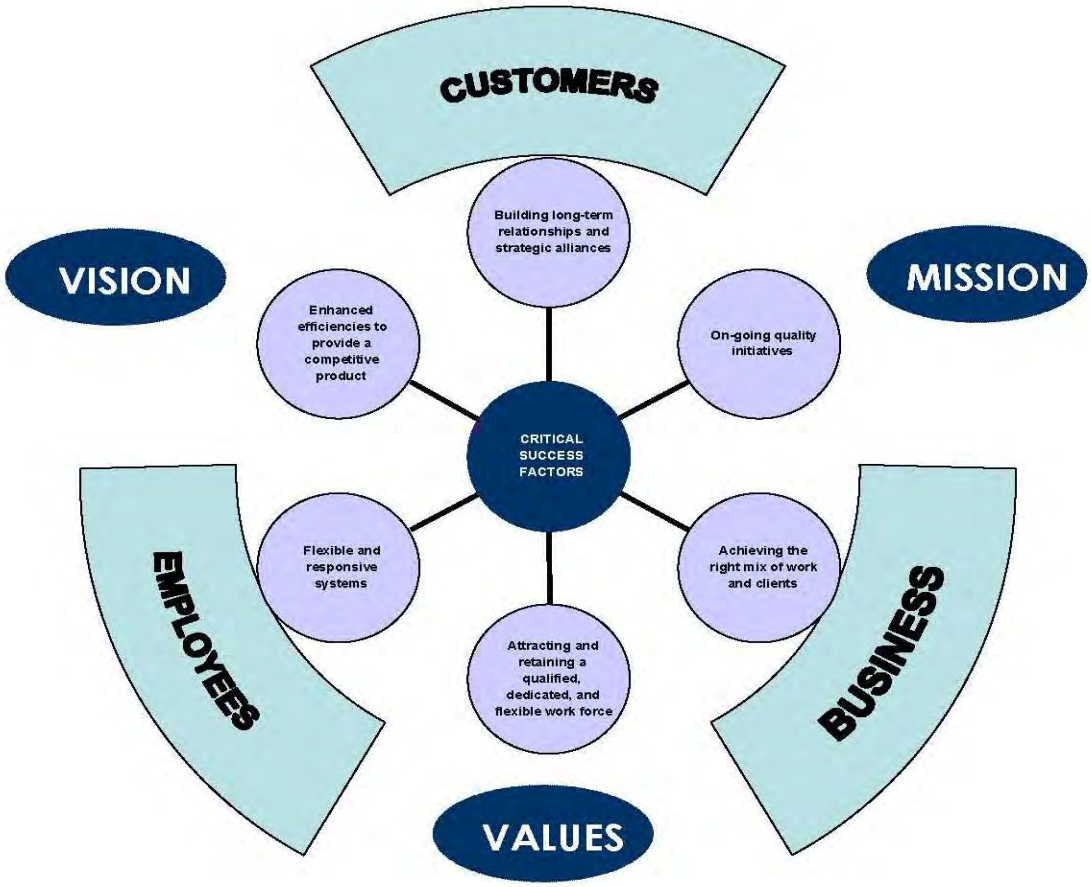
The Company's culture is defined by our fundamental core values which are upheld and maintained by management in much the same manner as is required of staff. These core values underpin the operations of the Company so that both management and staff work in a cohesive manner towards common objectives that translate to organizational and personal success.

These **CORE VALUES** are defined as:

- **Customer Service from the Heart:** At EL, customer service comes from our hearts and we understand that our Clients are our key partners to success. We believe in a customer-centric strategic plan and understand that serving our customers is essential at all levels of our organization.
- **360^0 Level of Integrity**: At the core of our daily business practices is integrity. EL uses integrity as the guiding principle to direct current policy and future policy development in the area of Client Services, Employee Relations, and analytical data production/ delivery.
- **Flexibility in Growth and Performance**: Our goal is to be flexible and open to change in an industry that is often influenced by great ideas and business technologies. EL will continue to provide first class services in an industry that faces challenging economic times. Over the 45+ years of our Company's existence, EL has survived and grown through many changes and our laboratory and its people stand strong and willing to make the necessary infrastructure investments towards our future stability and growth.
- **Team Spirit**: EL encompasses a holistic sense of purpose and goodwill that engenders team spirit and aims to engage our biggest asset, "company employees," in a manner that is productive to the mission of the Company. EL understands that we are only as good as our weakest link and we provide support and training to all of our staff to develop the skills needed to support EL's strategic plan.
- **Management by Fact**: EL will monitor success through accurate data and will institute actions in the interest of positive growth using a factual basis for decisions.

EL has a comprehensive strategic plan for on-going success. The elements of our plan include the laboratory's key stakeholders: Our Clients, Employees, and the Business. The following graphically represents our strategic plan for success:

Graph 1.1 – Strategic Plan for Success



In an effort for continuous improvements, the laboratory evaluates each of its **Critical Success Factors** annually and defines specific tasks that need to be improved or implemented:

- Enhanced efficiencies to provide a competitive product
- Flexible and responsive systems
- Building long-term relationships and strategic alliances
- On-going quality initiatives
- Achieving the right mix of work and clients
- Attracting and retaining a qualified, dedicated, and flexible Workforce

1.2 Organizational Structure, Roles, and Responsibilities

The President, Laboratory Director, Data Quality Manager (Quality Assurance Officer), Data Review Manager and the Project Managers listed below are the official signatories for the Laboratory. The staff technical positions outlined in summary format below are fully implemented within the organization and an organizational chart is included at the end of this section. Some additional administrative positions are also integrated into the overall laboratory operations. Due to the laboratory's small-business status, any of the top management personnel may be called upon to perform duties outside their main responsibilities where they have the necessary qualifications. In the event of a technical managers absence for more than 15 days, an acting technical manager will be named. If the absence exceeds 35 days, primary accrediting bodies (L-A-B and FL NELAP) will be notified. The President, Lab Director, Data Quality Manager, Data Review Manager and Group Leaders, under the guidance of the Data Quality Manager, are committed to and responsible for ensuring compliance with the NELAC/TNI 2009 Standards and DoD QSM 4.2/5.0 standards, as appropriate.

Owner/CEO
As CEO, responsibilities include planning, directing and evaluating the organization's fiscal function and performance. Evaluates and advises on the impact of long range planning and economic decisions about the Company's future. Provides oversight of all Human Resource functions and administration, and participates in oversight and strategic planning.

President
Perform functions to develop and drive the company's strategic planning process, support the management and administrative staff in the operations of the business, develop and maintain strategic alliances to further the mission of EL, and guide the laboratory testing and analysis functions of the organization, formulating and carrying out major policies, programs and objectives covering overall organization activities. Enhances and/or develops, implements and enforce policies and procedures of the organization by way of systems that will improve the overall operation and effectiveness of the company. Evaluate potential alliances acquisitions and/or mergers.

Laboratory Director (LD)
Perform functions to direct and manage the fulfillment process to provide timely and accurate processing of samples and reporting of analytical test results to clients, and participate in the corporate strategic planning processes to enhance operations, improve service, and maximize profits, formulating and carrying out organization policies, objectives and programs for a major function of the organization. The LD also serves as **the overall** Technical Manager for the laboratory. The LD is an approved signatory who can sign and approve Final Reports.

Director of Finance & Administration (DoFA)
The DoFA manages all aspects of the financial department to include Accounts Payable, Accounts Receivable, Benefits/HR Administration, Payroll Processing, Purchasing Management, and Business & Payroll Tax Administration. The DoFA prepares and monitors all financial statements on an ongoing basis. The administrative areas of responsibility for the DoFA include Client Support Services and Information Technology. Client Support Services includes Project Management, Sample Receiving, and Shipping. The Information Technology department includes the Network Administrator and the LIMS Administrator.

Data / Quality Manager (DQM)
Also known as the laboratory's **Quality Assurance Officer**, performs functions to implement, direct and manage the organization's quality systems and programs to ensure continuous and complete compliance with regulatory and certification requirements, guided by precedent and working within the limits of established policies. In this position, the DQM serves as the focal point for all Projects and for Level III and IV delivery requirements, including initial Project specific technical review, and report review. The DQM is an approved signatory who can sign and approve Final Reports.

Data Review Manager (DRM)
Perform functions to manage and direct the activities of report generation. In this position, the DRM serves as the focal point for all Level III and IV delivery requirements, including data compilation, case narrative generation, report review, and final package production and review. The DRM is an approved signatory who can sign and approve Final Reports.

Project Manager (PM)
Perform function to manage and monitor the projects of an assigned group of clients, to meet their specific needs through coordination of laboratory operations and/or external resources, and assist organization management to develop new business, making decisions based on conclusions for which there is little precedent. All Project Managers are approved signatories who can sign and approve Final Reports.

LIMS Administrator (LA)
The LIMS (Laboratory Information Management System) Administrator is responsible for the maintenance of the LIMS, implementing enhanced functionality, and basic programming in support of all LIMS based sample processing, data capture, and output/reporting.

Group Leaders (GL)
Perform functions to direct the day-to-day activities of technicians and analysts in preparation and performance of chemical and physical analyses of client samples, including laboratory maintenance, waste disposal, and making routine decisions for which there is a precedent. Analytical Group Leaders also qualify as technical managers for their sections.

Analyst (Lab Scientist)
Perform duties within area of expertise to conduct chemical and physical laboratory tests and analyses of solids, liquids, and/or gases; define and report on sample content in keeping with client requirements and the organization's quality and operating standards, guided by precedent and working within the limits of established policies.

Technician
Perform duties to receive and segregate samples into batches, prepare for analysis, perform routine test, document data, and maintain the department and instrumentation, following detailed instructions and under standard procedures.

1.3 Key Laboratory Relationships
This section summarizes the relationships between management, technical operations, support services, and the Quality System. The laboratory's Quality Systems overview is provided within section 2.0 of this manual and outlines specific activities within the laboratory's quality program. Quality is essentially an element of the laboratory that must be contained in all aspects of the operations and also must be followed by all levels of personnel. Even though, ultimately, it is the DQM's responsibility to ensure that the laboratory's Quality Systems elements are adhered to and to identify system weaknesses and vulnerabilities, the DQM is closely supported by all key staff such as the President, Laboratory Director, Director of Finance & Administration, Project Managers, and the Group Leaders. Collectively, the laboratory's key staff ensure that the lab's policies and objectives for quality of testing services are documented in the Quality Manual and, furthermore, each person assures that the staff reporting to them is trained in the Quality Manual and appropriate Quality Systems documents.

The **DQM** has the responsibility for the overall implementation of the quality system and the adherence to the standards related to each of the processes and policies within the system. The DQM has direct access to the highest level of management at which decisions are made on lab policy and /or resources, and to the Lab Director. In the absence of the DQM, the President will temporarily serve as the focal quality systems point of contact until an alternative plan of action is worked out. The DQM has the authority to "stop work" should any assessments or findings cast doubt on data integrity. Stop work may include the temporary discontinuation of performing a specific method or all work to be discontinued by a specific employee.

The **Lab Director** works closely with the DQM to ensure the review, implementation, and effectiveness of the systems in place. The Lab Director also serves as a liaison and facilitator between the laboratory operations staff and the DQM. The Lab Director shall assure that the Quality Manual is communicated to, understood by, and implemented by all personnel concerned. The Lab Director is responsible for all laboratory activities and in the absence of the Lab Director, the President will assume the role until an alternate plan of action is put into place.

Each **Group Leader (GL)** works closely with the DQM and the Lab Director to assure that daily workflow is in accordance with overall laboratory quality systems practices. GL's are very familiar with the calibration and test procedures, the objective of the calibration or test and the assessment of results. During GL absences, personnel

within each section will report directly to the Lab Director unless an alternate plan of action is put into place prior to the GL's absence or until an alternate plan of action is put into place.

The DoFA, supported by the Office Manager works closely with the Lab Director and DQM to assure that all solicitations are fully reviewed and response submittals are technically sound and accurate. The Office Manager is also responsible for reviewing and executing all client contracts to perform work. The DoFA works closely with Lab Director, Project Managers and administrative staff to assure that client's turnaround time expectations along with internal policies are met and followed.

The laboratory's **Project Managers** work as the liaison between the Client's and the laboratory operations staff. They are responsible for understanding the Client's regulatory and reporting needs and clearly communicating those needs to the operations staff. When work is received, the Project Managers review the login of the work and review the Final Report prior to delivery to the Client. The Project Managers are critical in identifying both technical and administrative issues and bringing them to the attention of the DQM, President, DoFA, and / or Lab Director so that counter measures are put into place to avoid future failures.

The laboratory's **Technical Directors** are ultimately responsible for the implementation of all technical requirements throughout the laboratory, with the oversight and assistance of the DQM and by training each of their staff adequately. By working closely with the DQM, the Technical Directors provide training to their staff and also provide feedback to the DQM. Besides the laboratory's Lab Director, each Analytical Group Leader is also qualified as a Technical Director for the Laboratory.

All **personnel** within the laboratory have direct access to their Group Leaders, the Lab Director, the President, the Data Quality Manager and the Human Resource Manager/DoFA. Even though the preferred path for trouble shooting, handling complaints, enhancements / or changes / or edits to existing procedures and policies is through the direct Group Leader, any of the laboratory's key personnel are available for open discussions and feedback.

In an effort to continue to maintain an open door policy and to facilitate discussions lab wide, the laboratory attempts to have Company-wide meetings quarterly and Key Staff meetings monthly. This forum allows for the communication of key performance indicators both administratively and those related to quality / technical issues.

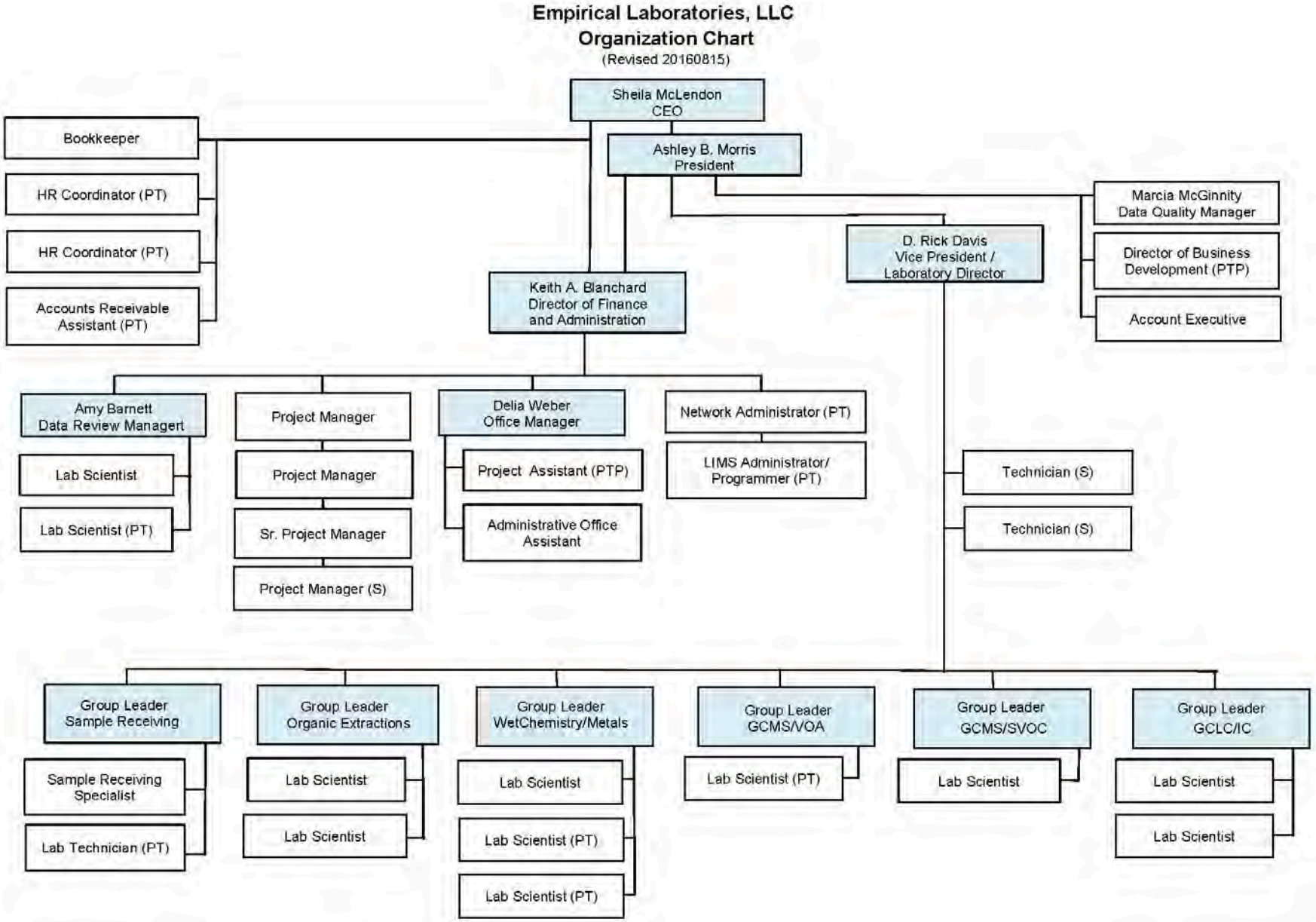
Table 1.2 Responsibility Matrix

Description	Lab Director	DQM	Group Leader	Project Manager	All Personnel
Initiation, Implementation, and Revision of Quality Systems	S	P	S	S	M
Operational Oversight to QS Policies	P	S	S	S	M
Ascertaining Adherence to QS Policies	S	P	S	S	M
Liaison between Regulatory Requirements / Regulators and the Laboratory	S	P	S	S	M
Liaison between the Customer and the Laboratory	S	S	S	P	M

Table Key:

P = Primary Responsibility
S = Support Role to Include Primary Responsibility as Assigned / Discussed
M = Must follow protocols as trained and per laboratory procedures

Chart 1.3 – Organization Chart



2.0 Quality Systems

The management and staff are dedicated and committed to performing its daily activities in accordance with the designed Quality Management System outlined within this document. It is the responsibility of all personnel to cooperate and support the principles, policies, and procedures outlined within the Quality System. In the spirit of good laboratory practices and continuous improvement processes, the laboratory is dedicated to creating operational efficiencies by also continuing to be compliant with the Quality Systems guidelines and mandates.

2.1 Quality Management System

The laboratory's Quality Management System is comprised of 3-essential elements, the Quality Manual, Standard Operating Procedures, and Document and Record Keeping. A brief description of each of these elements is listed below:

2.1.1 Quality Manual

This manual is based upon various State, Federal, and regulatory requirements, including those of the NELAC Institute (TNI) 2009, DoD QSM 4.2 and DoD QSM 5.0 standards. The manual is a general shell document that provides an overview on the laboratory's compliance to the technical and administrative compliance and certification requirements under the scope of the specific programs. The table below provides a reference to the mandated Quality Manual elements for both TNI2009, DoD QSM 4.2 and DoD QSM 5.0 requirements.

Table 2.1 TNI 2009 Requirement (*with DoD Quality System clarifications/requirements in italics)*		**Section Reference**
4.2.8.3	**The quality manual shall contain:**	
a)	document title;	Cover
b)	laboratory's full name and address;	Cover
c)	name, address (if different from above), and telephone number of individual(s) responsible for the laboratory;	Cover
d)	identification of all major organizational units which are to be covered by this quality manual and the effective date of the version;	Table of Contents
e)	identification of the laboratory's approved signatories;	1.2
f)	the signed and dated concurrence (with appropriate names and titles), of all responsible parties including the quality manager(s), technical manager(s), and the agent who is in charge of all laboratory activities, such as the laboratory director or laboratory manager;	Signature Page
g)	the objectives of the quality system and contain or reference the laboratory's policies and procedures;	1.0
h)	the laboratory's official quality policy statement, which shall include quality system objectives and management's commitment to ethical laboratory practices and to upholding the requirements of this Standard; *DoD Quality System: Commitment to Continual Improvement (Requirement)* *The quality policy shall also include a statement of management's commitment to continually improve the quality system. Management shall provide evidence of this commitment, which includes, but is not limited to, communicating to staff at all levels the importance of:* • *Meeting customer requirements;* • *Operating in accordance with statutory and regulatory requirements; and* • *Operating in accordance with the laboratory's documented ethics policy.*	1.1
i)	a table of contents, and applicable lists of references, glossaries and appendices.	Table of Contents
4.2.8.4	The quality manual shall contain or reference:	
a)	all maintenance, calibration and verification procedures used by the laboratory in conducting tests;	4.0, QS09
b)	major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;	4.0
c)	verification practices, which may include inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;	2.4
d)	procedures for reporting analytical results;	6.0, QS02
e)	the organization and management structure of the laboratory, its place in any parent organization, and relevant organizational charts;	1.0

Table 2.1 TNI Requirement (*with DoD Quality System clarifications/requirements in italics)* Continued		Section Reference
f)	procedures to ensure that all records required under this Standard are retained, as well as procedures for control and maintenance of documentation through a document control system that ensures that all standard operating procedures (SOPs), manuals, or documents clearly indicate the time period during which the procedure or document was in force;	2.1, QS01
g)	job descriptions of key staff and reference to the job descriptions of other laboratory staff; *DoD Quality System: Key Staff (Clarification)* *At a minimum, the following laboratory management staff (however named) shall be considered key staff:* *1. Management (e.g., President, Chief Executive Officer, Chief Operating Officer, Laboratory Director);* *2. Technical managers (e.g., Technical Director, Group Leaders);* *3. Quality managers;* *4. Support systems and administrative managers (e.g., LIMS manager, purchasing manager, project managers); and* *5. Client services managers.* *The quality manual shall describe the reporting relationship between key personnel and other staff. Job descriptions of key personnel shall describe their responsibilities.*	1.2
h)	procedures for achieving traceability of measurements;	4.0
i)	a list of all methods under which the laboratory performs its accredited testing;	7.0
j)	procedures for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;	2.5.1, QS12
k)	procedures for handling samples;	3.0, QS10, QS11, QS14
l)	procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;	2.5.3 QS05
m)	policy for permitting departures from documented policies and procedures or from standard specifications;	2.5.3 QS05
n)	procedures for dealing with complaints;	2.5.3 QS05
o)	procedures for protecting confidentiality (including national security concerns), and proprietary rights;	2.1
p)	procedures for audits and data review; *DoD Quality System: Procedures for Audits and Data Review (Requirement)* *The procedures for audits and data review shall specify which records must be included in the review.* *Internal data reviews consist of a tiered or sequential system of verification, consisting of at least three tiers, 100% review by the analyst, 100% verification review by a technically qualified supervisor or data review specialist, and a final administrative review.*	2.2, QS15 5.0, QS03
q)	procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training; and	2.5.2, QS03
r)	policy addressing the use of unique electronic signatures, where applicable.	2.5.5
DOD	***The following shall be implemented in addition to TNI 4.2.8.4 a) through r)***	
s)	*procedures for procurement of standards;*	QS04/QS16
t)	*procedures for data management including validation, verification, and purging of electronic data and data systems;*	QS03, QS06
u)	*procedures for manual entry of raw data from analytical measurements that are not interfaced to LIMS and the verification and records of the accuracy of manually entered data;*	QS03
v)	*procedures for making changes to electronic data (including establishing the requirements for a hardcopy or electronic log to record all changes to electronic data that affect data quality);*	QS03, QS06
w)	*procedures for how electronic data are processed, maintained, and reported;*	QS02, QS03
x)	*procedures for ensuring that data review includes all quality-related steps in the analytical process, including sample preparation, dilution calculations, chromatography evaluation, and spectral interpretations. The SOP shall require that records of data review be maintained and available for external review.*	Technical SOP checklists
y)	*A list of all current certifications and accreditations that the laboratory holds and the scope of certification or accreditation (with expiration date) for each;*	7.0

2.1.2 Standard Operating Procedures (SOPs)

Standard Operating Procedures (SOPs) have been created for each specific function at Empirical Laboratories to insure that proper and appropriate procedures are followed for all functions from preparation of sample kits through sample receipt, sample analysis, data generation, data validation/reporting, raw data records and reports storage, and waste disposal. In order to achieve this purpose, the SOP documents themselves must be carefully managed to insure that all personnel have SOPs which describe the tasks for which they are responsible and that these SOPs are current and appropriate. SOP **QS01** documents the process for maintaining SOPs. In addition, the

laboratory maintains an electronic master directory of all current SOPs and reference documents. This file is located on the V: drive and can be found using the following path:

V:/Standard Operating Procedures / TOC_SOPs_Documents_Controlled.xls

2.1.3 Laboratory Documents and Record Keeping Practices

The laboratory maintains a record system to meet its particular circumstances and comply with any applicable regulations. The system produces accurate records, which document all laboratory activities. **The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the test report required for the historical reconstruction of final results reported to external customers and regulators for a minimum of seven years.**

The laboratory's record keeping practices are addressed on a personnel level in SOP **QS03**. Archival is covered in SOP **QS02** with electronic backup/storage addressed in SOP **QS06**. Laboratory records and documents can fall into the following categories:

- **Administrative Records:** are complete records related to any personnel employed by the laboratory, including qualifications, training records, performance records, etc.
- **Certifications:** is all information related to business and technical certifications.
- **Technical Documents:** are current and retired Standard Operating Procedures, Quality Manual, Training Records, Sample, QA/QC, and Proficiency Testing raw data with appropriate documentation, any valid technical notes that are required to reconstruct final data reported, Electronic Data, Final Reports, Electronic Data Deliverables (EDDs), Chain of Custody documents, logbooks, Sample Receipt and Login forms, etc.

The laboratory maintains complete confidentiality of Client information and, based upon agreements in place, understands that certain information is also potentially proprietary. Data within the laboratory (hard copy) and electronic data is safe through on-site building security and also through network and electronic security systems. This ensures the confidentiality of client data. Most of the laboratory's data is transmitted electronically (secure email or upload to FTP site) to person(s) as designated by the client or via a contract. In order for data to be transmitted or sent to a third party, the client would need to provide written authorization for release of the data and records. In the event that the company is sold, the purchasing entity will assume all responsibility for retention of all documents in accordance with all regulatory and state legal time requirements. In the event that the lab closes permanently, all records will be scanned, verified then shredded and filed electronically with the appropriate regulatory and/or state agency. An escrow account will be set up specifically to pay for the storage of these electronic records for the duration of the required time period.

2.2 Internal Audits

At least annually, the laboratory's DQM conducts internal audits of its activities to ascertain operational compliance to the overall Quality Systems policies and procedures. Internal audits cover both technical methods performed at the laboratory and the overall laboratory quality system. Internal audits are covered in detail in SOP **QS15**.

2.3 Management Reviews

Annually, the management of the laboratory will review its quality systems to ensure the stability and effectiveness of the system. The review will be focused on reports from managerial and supervisory staff. This document will be reviewed by management and distributed to the appropriate managers and supervisory staff. This document will be kept in the laboratory files and available for review. It is the responsibility of the DQM and the Lab Director to coordinate the Management Review and provide a comprehensive report to the President. Upon distribution of the Management Review to all appropriate personnel, the laboratory's key staff will work together to take actions as appropriate to the overall laboratory's strategic plan (business and quality initiatives). SOP **QS15** addresses specific details of the Management Review process..

2.4 Verification/Preventative Action Practices

The laboratory participates in various **Proficiency Testing** (PT) programs, which are administered through independent and approved vendors. Various regulatory programs, such as TNI, DoD, and State specific ones, outline and mandate the frequency, criteria, and appropriate corrective actions for the validation of the PT programs. The DQM is responsible for maintaining the laboratory's compliance to the PT requirements of the various programs under which the laboratory is certified and /or approved.

Blind QC samples (duplicates and blanks) submitted to the laboratory from the field also form an important element of the laboratory's verification practices. Based upon Internal Audits, a Client's request, Project Data Quality Objectives (DQO's), method development and improvement goals, implemented corrective actions, due to consistent method failures, and /or due to inconsistencies in method performance, the DQM can initiate **double blind PT studies**, where pre-spiked samples are logged in to the laboratory's operational sample flow system without any of the staff knowing that the sample is a double blind to assess the effectiveness of the method.

Preventative Action Practices: In order to maintain the laboratory at the best operational capability, actions are taken to prevent down-time and delays. Maintenance contracts are maintained for critical instruments and/or software, routine maintenance is performed to maintain optimal operating conditions for instruments, servers and software, part-time technicians are employed during busy work periods, employee meetings are conducted to discuss current and upcoming work and strategic staff meetings are held to plan for the future.

Control Charts: In-house control limits and control charts are generated/reviewed in order to identify trends and address issues. Details on the processes are included in SOP **QS15**.

2.5 Systems Integrity

The procedures described in section 2.0 all describe the laboratory's efforts to both establish and verify efficient Quality Control and Assurance practices. In order to continue to maintain these systems, the laboratory has additional systems that also verify the integrity of the systems:

2.5.1 New Work Review

All new projects are initiated by the Laboratory Director, or designee, who delegates responsibilities for the new projects according to available resources. Laboratory key staff meets prior to initiation of a new project in order to determine if appropriate facilities and resources are available. The plan for any new testing shall be reviewed and approved by the Laboratory Director before commencing such work. After agreement is reached, facilities and resources are organized to efficiently perform the work. For any new testing requirements, the designated employee shall write a standard operating procedure and demonstrate capability to perform those tests prior to reporting results. The SOP(s) shall be under document control and a Demonstration of Capability Statement(s) must be on file. SOP **QS12** "Requests for Pricing, Subcontracting, Technical Project Information Form and Project Setup" details the process for review of new work.

2.5.2 Training Program

The training program consists of Orientation, Initial and Continuing Technical Training. Each person employed by the laboratory is assessed both initially and on an on-going basis for the required skill set to perform environmental testing and their compliance to the laboratory's overall policies and procedures. Each employee is assessed by their direct Group Leader and, in many cases, input is also received from the Laboratory Director. The following training is provided to each employee and detailed in SOP **QS03** "**Laboratory Personnel Training plus Data Flow, Review, and Integrity**":

- **Orientation:** Upon hire, the HR Manager or designee reviews the Company's overall administrative policies, including those related to work hours, safety, and general workplace rules. During this training, the employee is also trained on the laboratory's **Ethics and Data Integrity policy** and signs an ethics statement (found in QS03). The DQM or designee reviews the Company's QAM and quality systems documents appropriate to their position. All personnel are trained on the DoD mandated Ethics and Data Integrity as included in QS03.

The IT department performs orientation to the laboratory computer system including computer security while the Safety Officer or designee performs orientation related to laboratory safety requirements.

- **Initial Technical Training:** The Group Leader, or designee, is responsible for training each employee on position specific responsibilities and procedures to be followed. Components of the initial training include all the applicable specific up to date **SOPs, reference to quality systems documents** and **Demonstration of Capabilities (DOC)**. A DOC form is included in SOP **QS03**. It is the responsibility of laboratory's management along with all technical personnel to complete all required and applicable training documents and approvals for personnel prior to performing, assessing or reporting a particular method or a component of a method.
- **Continuing Training:** Annual training is performed which includes training on the laboratory's Quality Systems documents, Ethics and Data Integrity, computer safety, and other general training items. External training is also provided such as that for a specific method or instrument operation, etc.

2.5.3 Departures from Documented Policies and Procedures, Feedback, Complaints and Corrective Action

All of the laboratory's personnel have the responsibility of adhering to the policies and procedures; however, it is the DQMs responsibility to ensure that that the Quality Systems processes related to technical compliance are being followed. Quality Systems SOP **QS05** addresses the following:

- **Non-conformance report (NCR)** – Documents minor quality control outliers/ anomalies / deviations / exceedances.
- **Corrective Action Report (CAR)** – Documents major quality control outliers/ anomalies / deviations / exceedances including client complaints, results of internal or external audits, performance testing failures or repeated minor quality control exceedances. The CAR procedure is used to document any major departure identified during internal audits, external audits or routine data review.
- **Evaluation and Client Notification** – QS05 provides the procedure for evaluation and notification if data reported to any Client/Authority is identified as adversely affected.
- **Qualification** of associated data.

2.5.4 Security Levels

The laboratory sets LIMS security levels for data approval, review, and access based upon job descriptions and requirements. The DQM has the highest level of security across all software systems and within the LIMS. The LIMS Administrator has all access rights to LIMS. Security levels for each employee are approved by the DQM. These items are covered in SOP **QS06**.

2.5.5 Electronic Initials/Signatures

Electronic signatures are generated for all management personnel and analysts for use in reporting and PDF data review. They are stored on the lab server under password protection with the Network Administrator password known only to the Network Administrator and Senior Management. Signatures are uploaded to the manager/analyst computer and attached to a stamp within Adobe Acrobat so the signature stamp is only available when the manager/analyst is logged on to their computer. They are also securely stored within the LIMS so the signature stamp is available when the manager/analyst is logged into the LIMS. Signatures are applied by the manager/analyst to reviewed data or by managers to reports/forms/SOPs. Senior Management may apply a signature that is not their own with written or e-mailed documentation of that person's approval.

3.0 Sample Handling Policies

3.1 Field Sampling

When the laboratory is employed to perform field sampling activities, the laboratory utilizes guidance from SOP QS11, which outlines sampling guidelines for projects undertaken by the laboratory. A trained field sampler is sent to the site for sample collection and delivery of samples to the laboratory. Any relevant data such as the identification of the sampler, environmental conditions, field testing results, etc. are documented for the final

interpretation of the sample results and integrity. The laboratory has a recommended Chain of Custody that is provided to the Clients; however, due to the fact that the laboratory performs only a small percentage of sampling events for the samples received, the Chain of Custody documents utilized for the sampling events could potentially be those specific to the sampling organization.

3.2 Sample Receipt and Handling

The laboratory's detailed procedures for sampling handling, which include sample kit preparation, sample receipt; sample acceptance policies, sample login, sample login review, and sample distribution are detailed within SOPs **QS10** and **QS11**.

3.3 Sample Storage and Disposal

As a rule, samples are stored for 45 days following receipt and extracts/digestates are stored for 90 days following sample receipt; however, exceptions to this general rule may apply based upon client/project specific needs. Sample disposal is outlined in SOP **QS14**.

4.0 Procedures and Calibration

4.1 Analytical Procedures

Most of the analytical procedures performed by the laboratory are directed by the regulatory sector. Hence, most of the methodologies used begin with state and federal agencies. The laboratory will generally utilize only those methods that USEPA has recognized as "approved" analytical procedures. The following is a list of analytical references that the laboratory uses routinely. This list is not intended to be all inclusive.

EL utilizes various methods as approved by the EPA and referenced in the SOP's to include:

- US EPA Methods as written in the Code of Federal Regulations
- SW-846, Test Methods for Evaluating Solid Waste (various promulgated updates as required)
- Standard Methods for the Examination of Water and Wastewater, 20th-22nd editions (as requested)
- ASTM Standards
- NIOSH Manual of Analytical Methods, 3rd edition
- Other analytical references and methods may be utilized as required by clients and regulatory agencies.
- See specific methods utilized in the laboratory SOP's.

4.2 Equipment

A list of the laboratory's major equipment is listed in Table 4.2 within this section. Equipment is maintained, inspected, and cleaned according to the manufactures specifications. Any defective equipment is taken out of service until it has been shown to perform satisfactorily. Service of equipment is performed either in-house or by external service organizations. All records and certificates from service calls are retained. Instruments are tagged when they are out of service.

Equipment and reference material records include the following:

- Name of item
- Manufacturer, identification, serial number
- Date received and placed in service
- Copy of manufacturer's instructions or manuals
- Details of maintenance carried out to date
- History of any damage, malfunction, modification, or repair

4.3 Calibration – Support Equipment

General laboratory support equipment is calibrated / verified at least annually using NIST traceable references over the range of use. Specific calibration of support equipment is covered in SOP **QS08**.

Calibration – Major Equipment

SOP **QS08** provide general guidance on calibration for technical methodologies; however, method specific calibration information is listed within each method's SOP. The laboratory calibrates its analytical instrumentation at a frequency consistent with the methodologies referenced above. Calibration standards are obtained from several commercial suppliers. The formulations of calibration standards are documented in the LIMS including information for traceability such as the supplier, lot number, and expiration date where traceability to national standards of measurement is not applicable, the laboratory has evidence of correlation of data, for example the participation in inter-laboratory comparisons, proficiency testing or independent evaluations. The laboratory's current LIMS system allows for the complete entry and tracking of all calibration and quality control standards.

4.4 Equipment Maintenance

Where applicable, the laboratory performs daily, weekly, and / or monthly maintenance. Based upon the instrument type, maintenance may be needed less frequently. For all equipment, some of the recommended listed checks or changes are:

- Column
- Lines / Tubing
- Background / Baseline
- Injection Port (Septa, seal, etc.)
- Guard Column
- Pressure / Vacuum
- Flow
- Fittings
- Mobile Phase

Each technical SOP lists details specific to each instrument utilized to perform the method.

TABLE 4.2 – LABORATORY INSTRUMENTATION

TYPE OF EQUIPMENT	MANUFACTURER	MODEL #	SERIAL #	ANALYTICAL METHOD
Laboratory Information System	Promium	Element		NA
HP Unix Chemserver 4920	Hewlett Packard	Series 735	6503A00005	Organic backup
Gas Chromatograph (GC)	Hewlett Packard	5890 Series II GC	2643A11893	GC / MS VOA
Mass Spectrometer (MS)	Hewlett Packard	5971 MSD	3234A04299	GC / MS VOA
GC	Hewlett Packard	5890 Series II GC	3336A57710	GC / MS VOA
MS	Hewlett Packard	5972 MSD	3449A02107	GC / MS VOA
GC	Hewlett Packard	5890 GC / EPC	3203A40977	GC / MS VOA
MS	Hewlett Packard	5972 MSD	0402CCA0171	GC / MS VOA
GC	Agilent	6890 GC / EPC	US00036055	GC / MS VOA
MS	Hewlett Packard	5971 MSD	3118A02488	GC / MS BNA
GC	Agilent	6890 GC / EPC	US00009011	GC / MS BNA
MS	Agilent	5973 MSD	US94212162	GC / MS BNA
GC	Agilent	7890 GC	CN10821008	GC / MS BNA
MS	Agilent	5975 MSD	US80818976	GC / MS BNA
GC	Agilent	6890N GC	CN10305007	GC / MS BNA
MS	Agilent	5973N MSD	US30945049	GC / MS BNA
GC	Agilent	7890A GC	CN10381091	GC / MS BNA
MS	Agilent	5975C XL MSD	US80838702	GC / MS BNA
Purge and Trap	Tekmar	LSC 2000	91178007	GC / MS VOA
Auto Sampler	Varian	Archon 5100	13478	GC / MS VOA
Purge and Trap	Tekmar	LSC 3100	US01107022	GC / MS VOA
Auto Sampler	Varian	Archon 5100	12096	GC / MS VOA
Purge and Trap	Tekmar	LSC 3000	94067008	GC / MS VOA

TABLE 4.2 – LABORATORY INSTRUMENTATION				
TYPE OF EQUIPMENT	**MANUFACTURER**	**MODEL #**	**SERIAL #**	**ANALYTICAL METHOD**
Auto Sampler	Varian	Archon 5100	13509	GC / MS VOA
Purge and Trap	Tekmar	LSC 3100	00347006	GC / MS VOA
Auto Sampler	Varian	Archon 5100	14281	GC / MS VOA
Concentrator/Autosampler	ATOMX	ATOMX	US14066002	GC / MS VOA
Autosampler Tower	Hewlett Packard	7673A	3120A26207	GC / MS BNA
Autosampler Tower	Hewlett Packard	7673C	CN00001393	GC / MS BNA
Autosampler Tower	Agilent	7683	CN82249634	GC / MS BNA
Autosampler Tower	Agilent	7683	CN32631405	GC / MS BNA
Autosampler Tower	Agilent	7683	CN24428033	GC / MS BNA
Chemstation	Dell	GX150	GQZBH11	GC / MS VOA
Chemstation	Hewlett Packard	Kayak XA Series	US95169995	GC / MS VOA
Chemstation	Hewlett Packard	XA6 / 400 Series	US83851192	GC / MS VOA
Chemstation	Hewlett Packard	MSD Chemstation Software	G1701BA V# B.01.00	GC / MS BNA
Chemstation	Hewlett Packard		2UA81410WQ	GC / MS BNA
Chemstation	Hewlett Packard	MSD Chemstation Software	G1701DA D.03.00.611	GC / MS BNA
Chemstation	Agilent	MSD Chemstation Software	AZVX2-22242-6XW58-N82EA	GC / MS BNA
GC_FID1	Hewlett Packard	5890 FID / FID	3022A29035	GC DRO / EPH
GC_FID2	Hewlett Packard	5890 FID	3235A45128	GC DRO / EPH
GC_ECD2	Hewlett Packard	5890 ECD / ECD	2413A04722	GC Pest / PCB / Herb
GC_ECD3	Agilent	6890 ECD / ECD	US10225130	GC Pest / PCB / Herb
GC_ECD4	Agilent	6890 ECD / ECD	CN10525063	GC Pest / PCB / Herb
GC_VOA1	Hewlett Packard	5890 Series II PID / FID	3310A49411	GC GRO / VPH / MEE
GC_VOA2	Hewlett Packard	5890 Series II PID / FID	3310A48460	GC GRO / VPH
Purge and Trap	OI Analytical	4560	E330494	GC GRO / VPH
Purge and Trap	Tekmar	3000	94266004	GC GRO / VPH
Autosampler	Varian	Archon 5100	12096	GC GRO / VPH
Autosampler	Varian	Archon 5100	12072	GC GRO / VPH
Headspace Analyzer / Tray	Hewlett Packard	G1290-60583	3539I01567	GC MEE
Headspace Analyzer	Tekmar	7000	92112007	VOA Screening
50-Sample Carousel	Tekmar	7050	92104013	VOA Screening
Autosampler	Hewlett Packard	7673A	2847A12995	GC Pest / PCB / Herb
Autosampler	Hewlett Packard	7673A	2843A11536	GC Pest / PCB / Herb
Autosampler	Hewlett Packard	7673	3120A26200	GC Pest / PCB / Herb
Autosampler	Hewlett Packard	7673	3120A26207	GC Pest / PCB / Herb
Autosampler	Hewlett Packard	7673A	2704A06417	GC / DRO / EPH
Autosampler	Hewlett Packard	7673A	2936A14775	GC / DRO / EPH
Autosampler	Agilent	7683	CN24428105	GC Pest / PCB / Herb
Autosampler	Agilent	7683	CN24428103	GC Pest / PCB / Herb
Autosampler	Agilent	7683	CN52425706	GC Pest / PCB / Herb
Turbo-Vap	Zymark	Turbo-Vap II	TV9637R7036	Organic Extraction
Turbo-Vap	Zymark	Turbo-Vap II	TV0339N11934	Organic Extraction
Turbo-Vap	Zymark	Turbo-Vap II	TV9723R7528	Organic Extraction
Turbo-Vap	Zymark	Turbo-Vap II	TV0638N13277	Organic Extraction
Soxhlet / Soxtherm	OI-Analytical	SE-3A	4020298	Organic Extraction
Soxhlet / Soxtherm	OI-Analytical	SE-3A	492026	Organic Extraction
Soxhlet / Soxtherm	ABC	Extractor	451949	Organic Extraction
MARS	CEM	907501	MD1953	Organic Extraction
TOC Analyzer	Shimadzu	TOC-V WS	H51604200027	TOC
Auto Sampler	Shimadzu	ASI-V	H52104200823(SA)	TOC
TOC Analyzer	OI-Analytical	NDIR 1010	C325501193	TOC
Solids Module	OI-Analytical	S / N	C325501193	TOC
Integrator	OI-Analytical	WinTOC / REV. 3.0	322705270	TOC
CPU		Pentium II	CN284547	TOC
HPLC 1050	**Hewlett Packard**	**1050**		**HPLC**
Vacuum Degassing Module	Hewlett Packard	G1303A	3418J02815	HPLC
Autosampler And Upgrade	Hewlett Packard	79855A	3406A03098 3430a36031	HPLC
Solvent Module	Hewlett Packard	79856A	N-SN	HPLC
UV / VIS Detector	Hewlett Packard	79853A	N-SN	HPLC
Fluorescence Detector	Hewlett Packard	1046AX	3137G02110	HPLC
HPLC 1100	**Agilent**	**1100**		**HPLC**
Degasser	Agilent	G1379A	JP40723479	HPLC
Quarternary Pump	Agilent	G1311A	DE43632538	HPLC
Autosampler	Agilent	G1313A	DE33229698	HPLC
Column Compartment	Agilent	G1316A	DE43646892	HPLC
UV Detector	Agilent	G1314A	JP43827294	HPLC
HPLC 1260	**Agilent**	**1260**		**HPLC**
Quarternary Pump	Agilent	G1311B	DEAB704742	HPLC

TABLE 4.2 – LABORATORY INSTRUMENTATION				
TYPE OF EQUIPMENT	**MANUFACTURER**	**MODEL #**	**SERIAL #**	**ANALYTICAL METHOD**
Autosampler	Agilent	G1329B	DEAAC12640	HPLC
Column Compartment	Agilent	G1316A	DEACN13892	HPLC
Variable Wavelength Detector	Agilent	G1314F	DEABB02879	HPLC
HPLC 1200	**Agilent**	**1200**		**HPLC / MS**
Degasser	Agilent	G1322A	JP62357946	HPLC / MS
Quarternary Pump	Agilent	G1311A	DE62960168	HPLC / MS
Autosampler	Agilent	G1329A	DE67767160	HPLC / MS
Column Compartment	Agilent	G1316A	DE63066832	HPLC / MS
UV Detector	Agilent	G1314A	JP33324357	HPLC / MS
MSD	Agilent	6140	US73460217	HPLC / MS
Gel Permeation Chromatograph	J2 Scientific	AccuPrep MPS	05C-1154-4.1	Organics
Flow Injection Analyzer	LACHAT	2300-000	200-0524	General Chemistry
Dilstillation System	Environmental Express	SimpleDist SC154	8885CECW3811	General Chemistry
Ion Chromatograph	Thermo Scientific	Dionex ICS-2100	14036974	General Chemistry
Gradient Pump	Thermo Scientific	Dionex AS-DV	13030021	General Chemistry
Detector	Thermo Scientific	ICS Series VWD 1CH	11120038	General Chemistry
Chromatography Software	Thermo Scientific	Chromeleon 7	155862	General Chemistry
Ion Chromatograph	Dionex Corp.	DX500 / LC 20	99020581	General Chemistry
Gradient Pump	Dionex Corp.	GP50	98120789	General Chemistry
Detector	Dionex Corp.	CD20	99020199	General Chemistry
UV-VIS Spectrophotometer	Thermo Spectronic	Genesys 20	3SGF365005	General Chemistry
Turbidimeter	HACH	2100A	4449	General Chemistry
Mercury Analyzer	Perkin-Elmer	FIMS 100	1129	7470 / 7471
Autosampler	Perkin-Elmer	AS9C	2917	7470 / 7471
Conductivity Bridge	Orion	105A+	004029	General Chemistry
Dissolved Oxygen Meter	YSI	5000	04E8552	General Chemistry
Balance	Fisher	Accu-124	F124023010	Gravimetrics
Balance	Fisher	XT-3000	A1435	Gravimetrics
Balance	VMC	VB-302	VB30202110	Gravimetrics
Balance	Mettler Toledo	PL601S	6428300054	Gravimetrics
Balance	Sartorius	BL310	13003644	Gravimetrics
Balance	Ohaus	SP402	7132160685	Gravimetrics
Balance	Mettler Toledo	AE240	38695	Gravimetrics
Balance	Mettler Toledo	BASBAS#1	J41186	Gravimetrics
ICAP	Thermo Scientific	6500 Duo	20062310	6010
pH Meter	Orion Research	420A	3159	General Chemistry
pH Meter	Corning	240	2818	General Chemistry
LCMS-8060 Package	Shimadzu	220-91621-01	011105300116AE	LCMS-8060
Peak Nitrogen and Air Generator - Genius 1051 for LCMS-8050/8060	Shimadzu	220-91491-10	A16-08-329	LCMS-8060
UPS 5.2kVa, for LCMS-8030/8040/8050/8060 with N2 Generator, ABCDEF5200-22	Shimadzu	220-97823-12	2205295R-1610015	LCMS-8060
CBM-20A W/ Network Switch	Shimadzu	220-91398-20	L20235456594	LCMS-8060
DGU-20A5R, ROHS-compliant	Shimadzu	228-45019-58	L20705466729	LCMS-8060
Reservoir Tray, LC-20/30	Shimadzu	228-45041-91	L20305456402SL	LCMS-8060
Nexera LC-30AD HPLC Pump	Shimadzu	228-45162-42	A=L20555452464, B=L20555452403	LCMS-8060
Nexera SIL-30AC UHPLC Cooled Autosampler	Shimadzu	228-45157-42	L20565450710	LCMS-8060
CTO-20AC (column oven)	Shimadzu	228-45010-42	L20215452675	LCMS-8060
FCV-12AH 2-Pos 6-Port Flow Switching Valve	Shimadzu	228-45013-41	C20435450992	LCMS-8060

5.0 Data Analysis, Review, and Reporting

The laboratory has a three tier level of data analysis, review, and reporting which is detailed in SOP **QS03**.

6.0 Reporting Data to the External Client

The laboratory's LIMS allows for the reporting of sample results in various formats. SOP **QS03** describes data review and reporting into LIMS. Subsequently, based upon the Project or Client's needs, the laboratory routinely

reports results only through full data package report formats. The report format of the Final Report may change based upon the regulatory program within which the laboratory has to report the data, the data. Reporting requirements and processes are detailed in SOP **QS02**. Client notification of excursions is detailed in SOPs **QS05** and **QS15**.

The Final Report translates laboratory data into some key technical elements that encompass the scope of the laboratory's QA / QC processes. Laboratory SOP **QS08** provides information on each of these and the use of the listed technical terms is based upon the Project or Client's needs and the scope of the regulatory program.

7.0 Capabilities Summary/Certifications/Methods/Analytes

The laboratory is listing its capabilities within this section as a guidance table only. The laboratory carries an extensive list of certifications for many States and programs. The list provided below is updated on an on-going basis and additional certifications are applied for based upon the laboratories on-going project needs. Detailed scopes for FL NELAP (Table 7.1) and DOD ELAP (Table 7.2) follow this listing. Detailed scopes for other listed certifications are available upon request. The LIMS system maintains the most current information on the list of methods with the associated parameters and relevant statistical and technical data, such as the DL, LOD, LOQ, control limits for precision and accuracy, internal standard references, etc. For various regulatory programs, the terminologies for the listed information may vary. The laboratory's most common terminologies and their corresponding definitions, clarifications, and requirements are listed within Quality Systems **SOP QS08**.

DoD ELAP QSM5.0, Certificate Number L2226
- Aqueous
- Non-aqueous
- Expires: 11/30/2018

State of Florida, Department of Health – NELAP Primary, Lab ID: E87646
- Clean Water Act
- RCRA/CERCLA
- Expires: 06/30/2017

State of Georgia, Environmental Protection Agency – NELAP, Self Certification
- Expires: 06/30/2017

Commonwealth of Kentucky, Energy and Environment Cabinet – WWLCP, Laboratory Number: 98017
- Wastewater
- Expires: 12/31/2016

Commonwealth of Kentucky, Department of Environmental Protection – UST, Certificate Number: 77
- Aqueous
- Non-aqueous
- Expires: 06/30/2017

State of New Jersey, Department of Environmental Protection – NELAP, Lab ID: TN473
- Water Pollution
- Solid and Hazardous Waste
- Expires: 06/30/2017

State of North Carolina, Department of Environment and Natural Resources - Certificate Number: 643
- Aqueous
- Non-aqueous
- Expires: 12/31/2016

State of Texas, Commission on Environmental Quality – NELAP, Certificate Number: T104704307-16-12
- Aqueous
- Non-aqueous
- Expires: 12/31/2016

State of Utah, Department of Health – NELAP, Certificate Number: TN0042015-7

- Aqueous
- Non-aqueous
- Expires: 07/31/2016

Commonwealth of Virginia, Department of General Services – NELAP, Certificate Number: 8176, Lab ID: 460243

- Aqueous
- Non-aqueous
- Expires: 12/14/2016

State of Washington, Department of Ecology – NELAP, Lab ID: C934-16

- Groundwater
- Solid and Hazardous Waste
- Expires: 03/18/2017

Table 7.1 FL NELAP Parameter	Matrix Code	Approved Method
1,1,1,2-Tetrachloroethane	NPW	EPA 8260
1,1,1,2-Tetrachloroethane	SCM	EPA 8260
1,1,1-Trichloroethane	NPW	EPA 624
1,1,1-Trichloroethane	NPW	EPA 8260
1,1,1-Trichloroethane	SCM	EPA 8260
1,1,2,2-Tetrachloroethane	NPW	EPA 624
1,1,2,2-Tetrachloroethane	NPW	EPA 8260
1,1,2,2-Tetrachloroethane	SCM	EPA 8260
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	NPW	EPA 8260
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	SCM	EPA 8260
1,1,2-Trichloroethane	NPW	EPA 624
1,1,2-Trichloroethane	NPW	EPA 8260
1,1,2-Trichloroethane	SCM	EPA 8260
1,1-Dichloroethane	NPW	EPA 624
1,1-Dichloroethane	NPW	EPA 8260
1,1-Dichloroethane	SCM	EPA 8260
1,1-Dichloroethylene	NPW	EPA 624
1,1-Dichloroethylene	NPW	EPA 8260
1,1-Dichloroethylene	SCM	EPA 8260
1,1-Dichloropropene	NPW	EPA 8260
1,1-Dichloropropene	SCM	EPA 8260
1,2,3-Trichlorobenzene	NPW	EPA 8260
1,2,3-Trichlorobenzene	SCM	EPA 8260
1,2,3-Trichloropropane	NPW	EPA 8260
1,2,3-Trichloropropane	SCM	EPA 8260
1,2,4,5-Tetrachlorobenzene	NPW	EPA 8270
1,2,4,5-Tetrachlorobenzene	SCM	EPA 8270
1,2,4-Trichlorobenzene	NPW	EPA 625
1,2,4-Trichlorobenzene	NPW	EPA 8260
1,2,4-Trichlorobenzene	NPW	EPA 8270
1,2,4-Trichlorobenzene	SCM	EPA 8260
1,2,4-Trichlorobenzene	SCM	EPA 8270
1,2,4-Trimethylbenzene	NPW	EPA 8260
1,2,4-Trimethylbenzene	SCM	EPA 8260
1,2-Dibromo-3-chloropropane (DBCP)	NPW	EPA 8011
1,2-Dibromo-3-chloropropane (DBCP)	NPW	EPA 8260
1,2-Dibromo-3-chloropropane (DBCP)	SCM	EPA 8260
1,2-Dibromoethane (EDB, Ethylene dibromide)	NPW	EPA 8011
1,2-Dibromoethane (EDB, Ethylene dibromide)	NPW	EPA 8260
1,2-Dibromoethane (EDB, Ethylene dibromide)	SCM	EPA 8260
1,2-Dichlorobenzene	NPW	EPA 624
1,2-Dichlorobenzene	NPW	EPA 625
1,2-Dichlorobenzene	NPW	EPA 8260
1,2-Dichlorobenzene	NPW	EPA 8270
1,2-Dichlorobenzene	SCM	EPA 8260
1,2-Dichlorobenzene	SCM	EPA 8270
1,2-Dichloroethane	NPW	EPA 624
1,2-Dichloroethane	NPW	EPA 8260
1,2-Dichloroethane	SCM	EPA 8260
1,2-Dichloropropane	NPW	EPA 624
1,2-Dichloropropane	NPW	EPA 8260
1,2-Dichloropropane	SCM	EPA 8260
1,2-Diphenylhydrazine (as Azobenzene)	NPW	EPA 8270
1,2-Diphenylhydrazine (as Azobenzene)	SCM	EPA 8270
1,2-Diphenylhydrazine	SCM	EPA 8270
1,3,5-Trimethylbenzene	NPW	EPA 8260
1,3,5-Trimethylbenzene	SCM	EPA 8260
1,3,5-Trinitrobenzene (1,3,5-TNB)	NPW	EPA 8330
1,3,5-Trinitrobenzene (1,3,5-TNB)	SCM	EPA 8330
1,3-Dichlorobenzene	NPW	EPA 624
1,3-Dichlorobenzene	NPW	EPA 625
1,3-Dichlorobenzene	NPW	EPA 8260
1,3-Dichlorobenzene	NPW	EPA 8270
1,3-Dichlorobenzene	SCM	EPA 8260
1,3-Dichlorobenzene	SCM	EPA 8270
1,3-Dichloropropane	NPW	EPA 8260
1,3-Dichloropropane	SCM	EPA 8260
1,3-Dinitrobenzene (1,3-DNB)	NPW	EPA 8330
1,3-Dinitrobenzene (1,3-DNB)	SCM	EPA 8330
1,4-Dichlorobenzene	NPW	EPA 624
1,4-Dichlorobenzene	NPW	EPA 625
1,4-Dichlorobenzene	NPW	EPA 8260
1,4-Dichlorobenzene	NPW	EPA 8270
1,4-Dichlorobenzene	SCM	EPA 8260
1,4-Dichlorobenzene	SCM	EPA 8270
1,4-Dioxane (1,4-Diethyleneoxide)	NPW	EPA 8260
1,4-Dioxane (1,4-Diethyleneoxide)	NPW	EPA 8270
1,4-Dioxane (1,4-Diethyleneoxide)	SCM	EPA 8260
1,4-Dioxane (1,4-Diethyleneoxide)	SCM	EPA 8270
1-Chlorohexane	NPW	EPA 8260
1-Chlorohexane	SCM	EPA 8260
1-Chloronaphthalene	NPW	EPA 8270
1-Methylnaphthalene	NPW	EPA 8270

Table 7.1 FL NELAP Parameter	Matrix Code	Approved Method
1-Methylnaphthalene	SCM	EPA 8270
2,2 -Oxybis(1-chloropropane),bis(2-Chloro-1-methylethyl)ether (fka bis(2-Chloroisopropyl) ether	NPW	EPA 625
2,2 -Oxybis(1-chloropropane),bis(2-Chloro-1-methylethyl)ether (fka bis(2-Chloroisopropyl) ether	NPW	EPA 8270
2,2 -Oxybis(1-chloropropane),bis(2-Chloro-1-methylethyl)ether (fka bis(2-Chloroisopropyl) ether	SCM	EPA 8270
2,2-Dichloropropane	NPW	EPA 8260
2,2-Dichloropropane	SCM	EPA 8260
2,3,4,6-Tetrachlorophenol	NPW	EPA 8270
2,3,4,6-Tetrachlorophenol	SCM	EPA 8270
2,4,5-T	NPW	EPA 8151
2,4,5-Trichlorophenol	NPW	EPA 8270
2,4,5-Trichlorophenol	SCM	EPA 8270
2,4,6-Trichlorophenol	NPW	EPA 625
2,4,6-Trichlorophenol	NPW	EPA 8270
2,4,6-Trichlorophenol	SCM	EPA 8270
2,4,6-Trinitrotoluene (2,4,6-TNT)	NPW	EPA 8330
2,4,6-Trinitrotoluene (2,4,6-TNT)	SCM	EPA 8330
2,4-D	NPW	EPA 8151
2,4-DB	NPW	EPA 8151
2,4-Dichlorophenol	NPW	EPA 625
2,4-Dichlorophenol	NPW	EPA 8270
2,4-Dichlorophenol	SCM	EPA 8270
2,4-Dimethylphenol	NPW	EPA 625
2,4-Dimethylphenol	NPW	EPA 8270
2,4-Dimethylphenol	SCM	EPA 8270
2,4-Dinitrophenol	NPW	EPA 625
2,4-Dinitrophenol	NPW	EPA 8270
2,4-Dinitrophenol	SCM	EPA 8270
2,4-Dinitrotoluene (2,4-DNT)	NPW	EPA 625
2,4-Dinitrotoluene (2,4-DNT)	NPW	EPA 8270
2,4-Dinitrotoluene (2,4-DNT)	NPW	EPA 8330
2,4-Dinitrotoluene (2,4-DNT)	SCM	EPA 8270
2,4-Dinitrotoluene (2,4-DNT)	SCM	EPA 8330
2,6-Dichlorophenol	NPW	EPA 8270
2,6-Dichlorophenol	SCM	EPA 8270
2,6-Dinitrotoluene (2,6-DNT)	NPW	EPA 625
2,6-Dinitrotoluene (2,6-DNT)	NPW	EPA 8270
2,6-Dinitrotoluene (2,6-DNT)	NPW	EPA 8330
2,6-Dinitrotoluene (2,6-DNT)	SCM	EPA 8270
2,6-Dinitrotoluene (2,6-DNT)	SCM	EPA 8330
2-Amino-4,6-dinitrotoluene (2-am-dnt)	NPW	EPA 8330
2-Amino-4,6-dinitrotoluene (2-am-dnt)	SCM	EPA 8330
2-Butanone (Methyl ethyl ketone, MEK)	NPW	EPA 8260
2-Butanone (Methyl ethyl ketone, MEK)	SCM	EPA 8260
2-Chloroethyl vinyl ether	NPW	EPA 624
2-Chloroethyl vinyl ether	NPW	EPA 8260
2-Chloroethyl vinyl ether	SCM	EPA 8260
2-Chloronaphthalene	NPW	EPA 625
2-Chloronaphthalene	NPW	EPA 8270
2-Chloronaphthalene	SCM	EPA 8270
2-Chlorophenol	NPW	EPA 625
2-Chlorophenol	NPW	EPA 8270
2-Chlorophenol	SCM	EPA 8270
2-Chlorotoluene	NPW	EPA 8260
2-Chlorotoluene	SCM	EPA 8260
2-Hexanone	NPW	EPA 8260
2-Hexanone	SCM	EPA 8260
2-Methyl-4,6-dinitrophenol	NPW	EPA 625
2-Methyl-4,6-dinitrophenol	NPW	EPA 8270
2-Methyl-4,6-dinitrophenol	SCM	EPA 8270
2-Methylnaphthalene	NPW	EPA 8270
2-Methylnaphthalene	SCM	EPA 8270
2-Methylphenol (o-Cresol)	NPW	EPA 8270
2-Methylphenol (o-Cresol)	SCM	EPA 8270
2-Nitroaniline	NPW	EPA 8270
2-Nitroaniline	SCM	EPA 8270
2-Nitrophenol	NPW	EPA 625
2-Nitrophenol	NPW	EPA 8270
2-Nitrophenol	SCM	EPA 8270
2-Nitrotoluene	NPW	EPA 8330
2-Nitrotoluene	SCM	EPA 8330
3,3 -Dichlorobenzidine	NPW	EPA 625
3,3 -Dichlorobenzidine	NPW	EPA 8270
3,3 -Dichlorobenzidine	SCM	EPA 8270
3,5-Dinitroaniline	NPW	EPA 8330
3,5-Dinitroaniline	SCM	EPA 8330
3-Methylphenol (m-Cresol)	NPW	EPA 8270
3-Methylphenol (m-Cresol)	SCM	EPA 8270
3-Nitroaniline	NPW	EPA 8270
3-Nitroaniline	SCM	EPA 8270
3-Nitrotoluene	NPW	EPA 8330
3-Nitrotoluene	SCM	EPA 8330
4,4 -DDD	NPW	EPA 608
4,4 -DDD	NPW	EPA 8081

Table 7.1 FL NELAP Parameter	Matrix Code	Approved Method
4,4 -DDD	SCM	EPA 8081
4,4 -DDE	NPW	EPA 608
4,4 -DDE	NPW	EPA 8081
4,4 -DDE	SCM	EPA 8081
4,4 -DDT	NPW	EPA 608
4,4 -DDT	NPW	EPA 8081
4,4 -DDT	SCM	EPA 8081
4-Amino-2,6-dinitrotoluene (4-am-dnt)	NPW	EPA 8330
4-Amino-2,6-dinitrotoluene (4-am-dnt)	SCM	EPA 8330
4-Bromophenyl phenyl ether	NPW	EPA 625
4-Bromophenyl phenyl ether	NPW	EPA 8270
4-Bromophenyl phenyl ether	SCM	EPA 8270
4-Chloro-3-methylphenol	NPW	EPA 625
4-Chloro-3-methylphenol	NPW	EPA 8270
4-Chloro-3-methylphenol	SCM	EPA 8270
4-Chloroaniline	NPW	EPA 8270
4-Chloroaniline	SCM	EPA 8270
4-Chlorophenyl phenylether	NPW	EPA 625
4-Chlorophenyl phenylether	NPW	EPA 8270
4-Chlorophenyl phenylether	SCM	EPA 8270
4-Chlorotoluene	NPW	EPA 8260
4-Chlorotoluene	SCM	EPA 8260
4-Methyl-2-pentanone (MIBK)	NPW	EPA 8260
4-Methyl-2-pentanone (MIBK)	SCM	EPA 8260
4-Methylphenol (p-Cresol)	NPW	EPA 8270
4-Methylphenol (p-Cresol)	SCM	EPA 8270
4-Nitroaniline	NPW	EPA 8270
4-Nitroaniline	SCM	EPA 8270
4-Nitrophenol	NPW	EPA 625
4-Nitrophenol	NPW	EPA 8270
4-Nitrophenol	SCM	EPA 8270
4-Nitrotoluene	NPW	EPA 8330
4-Nitrotoluene	SCM	EPA 8330
Acenaphthene	NPW	EPA 625
Acenaphthene	NPW	EPA 8270
Acenaphthene	SCM	EPA 8270
Acenaphthylene	NPW	EPA 625
Acenaphthylene	NPW	EPA 8270
Acenaphthylene	SCM	EPA 8270
Acetone	NPW	EPA 8260
Acetone	SCM	EPA 8260
Acetonitrile	NPW	EPA 8260
Acetonitrile	SCM	EPA 8260
Acetophenone	NPW	EPA 8270
Acetophenone	SCM	EPA 8270
Acrolein (Propenal)	NPW	EPA 624
Acrolein (Propenal)	NPW	EPA 8260
Acrolein (Propenal)	SCM	EPA 8260
Acrylonitrile	NPW	EPA 624
Acrylonitrile	NPW	EPA 8260
Acrylonitrile	SCM	EPA 8260
Alachlor	SCM	EPA 8081
Aldrin	NPW	EPA 608
Aldrin	NPW	EPA 8081
Aldrin	SCM	EPA 8081
Alkalinity as CaCO3	NPW	SM 2320 B
Allyl chloride (3-Chloropropene)	NPW	EPA 8260
Allyl chloride (3-Chloropropene)	SCM	EPA 8260
alpha-BHC (alpha-Hexachlorocyclohexane)	NPW	EPA 608
alpha-BHC (alpha-Hexachlorocyclohexane)	NPW	EPA 8081
alpha-BHC (alpha-Hexachlorocyclohexane)	SCM	EPA 8081
alpha-Chlordane	NPW	EPA 8081
alpha-Chlordane	SCM	EPA 8081
Aluminum	NPW	EPA 200.7
Aluminum	NPW	EPA 6010
Aluminum	SCM	EPA 6010
Ammonia as N	NPW	SM 4500-NH3 G
Aniline	NPW	EPA 8270
Aniline	SCM	EPA 8270
Anthracene	NPW	EPA 625
Anthracene	NPW	EPA 8270
Anthracene	SCM	EPA 8270
Antimony	NPW	EPA 200.7
Antimony	NPW	EPA 6010
Antimony	SCM	EPA 6010
Aroclor-1016 (PCB-1016)	NPW	EPA 608
Aroclor-1016 (PCB-1016)	NPW	EPA 8082
Aroclor-1016 (PCB-1016)	SCM	EPA 8082
Aroclor-1221 (PCB-1221)	NPW	EPA 608
Aroclor-1221 (PCB-1221)	NPW	EPA 8082
Aroclor-1221 (PCB-1221)	SCM	EPA 8082
Aroclor-1232 (PCB-1232)	NPW	EPA 608
Aroclor-1232 (PCB-1232)	NPW	EPA 8082

Table 7.1 FL NELAP Parameter	Matrix Code	Approved Method
Aroclor-1232 (PCB-1232)	SCM	EPA 8082
Aroclor-1242 (PCB-1242)	NPW	EPA 608
Aroclor-1242 (PCB-1242)	NPW	EPA 8082
Aroclor-1242 (PCB-1242)	SCM	EPA 8082
Aroclor-1248 (PCB-1248)	NPW	EPA 608
Aroclor-1248 (PCB-1248)	NPW	EPA 8082
Aroclor-1248 (PCB-1248)	SCM	EPA 8082
Aroclor-1254 (PCB-1254)	NPW	EPA 608
Aroclor-1254 (PCB-1254)	NPW	EPA 8082
Aroclor-1254 (PCB-1254)	SCM	EPA 8082
Aroclor-1260 (PCB-1260)	NPW	EPA 608
Aroclor-1260 (PCB-1260)	NPW	EPA 8082
Aroclor-1260 (PCB-1260)	SCM	EPA 8082
Aroclor-1262 (PCB-1262)	NPW	EPA 8082
Aroclor-1262 (PCB-1262)	SCM	EPA 8082
Aroclor-1268 (PCB-1268)	NPW	EPA 8082
Aroclor-1268 (PCB-1268)	SCM	EPA 8082
Arsenic	NPW	EPA 200.7
Arsenic	NPW	EPA 6010
Arsenic	SCM	EPA 6010
Atrazine	NPW	EPA 8270
Atrazine	SCM	EPA 8270
Barium	NPW	EPA 200.7
Barium	NPW	EPA 6010
Barium	SCM	EPA 6010
Benzaldehyde	NPW	EPA 8270
Benzaldehyde	SCM	EPA 8270
Benzene	NPW	EPA 624
Benzene	NPW	EPA 8260
Benzene	SCM	EPA 8260
Benzidine	NPW	EPA 625
Benzidine	NPW	EPA 8270
Benzidine	SCM	EPA 8270
Benzo(a)anthracene	NPW	EPA 625
Benzo(a)anthracene	NPW	EPA 8270
Benzo(a)anthracene	SCM	EPA 8270
Benzo(a)pyrene	NPW	EPA 625
Benzo(a)pyrene	NPW	EPA 8270
Benzo(a)pyrene	SCM	EPA 8270
Benzo(b)fluoranthene	NPW	EPA 625
Benzo(b)fluoranthene	NPW	EPA 8270
Benzo(b)fluoranthene	SCM	EPA 8270
Benzo(g,h,i)perylene	NPW	EPA 625
Benzo(g,h,i)perylene	NPW	EPA 8270
Benzo(g,h,i)perylene	SCM	EPA 8270
Benzo(k)fluoranthene	NPW	EPA 625
Benzo(k)fluoranthene	NPW	EPA 8270
Benzo(k)fluoranthene	SCM	EPA 8270
Benzoic acid	NPW	EPA 8270
Benzoic acid	SCM	EPA 8270
Benzyl alcohol	NPW	EPA 8270
Benzyl alcohol	SCM	EPA 8270
Beryllium	NPW	EPA 200.7
Beryllium	NPW	EPA 6010
Beryllium	SCM	EPA 6010
beta-BHC (beta-Hexachlorocyclohexane)	NPW	EPA 608
beta-BHC (beta-Hexachlorocyclohexane)	NPW	EPA 8081
beta-BHC (beta-Hexachlorocyclohexane)	SCM	EPA 8081
Biochemical oxygen demand	NPW	SM 5210 B
Biphenyl	NPW	EPA 8270
Biphenyl	SCM	EPA 8270
bis(2-Chloroethoxy)methane	NPW	EPA 625
bis(2-Chloroethoxy)methane	NPW	EPA 8270
bis(2-Chloroethoxy)methane	SCM	EPA 8270
bis(2-Chloroethyl) ether	NPW	EPA 625
bis(2-Chloroethyl) ether	NPW	EPA 8270
bis(2-Chloroethyl) ether	SCM	EPA 8270
bis(2-Ethylhexyl) phthalate (DEHP)	NPW	EPA 625
bis(2-Ethylhexyl) phthalate (DEHP)	NPW	EPA 8270
bis(2-Ethylhexyl) phthalate (DEHP)	SCM	EPA 8270
Boron	NPW	EPA 200.7
Boron	NPW	EPA 6010
Boron	SCM	EPA 6010
Bromide	NPW	EPA 300.0
Bromide	NPW	EPA 9056
Bromide	SCM	EPA 9056
Bromobenzene	NPW	EPA 8260
Bromobenzene	SCM	EPA 8260
Bromochloromethane	NPW	EPA 8260
Bromochloromethane	SCM	EPA 8260
Bromodichloromethane	NPW	EPA 624
Bromodichloromethane	NPW	EPA 8260
Bromodichloromethane	SCM	EPA 8260

Table 7.1 FL NELAP Parameter	Matrix Code	Approved Method
Bromoform	NPW	EPA 624
Bromoform	NPW	EPA 8260
Bromoform	SCM	EPA 8260
Butyl benzyl phthalate	NPW	EPA 625
Butyl benzyl phthalate	NPW	EPA 8270
Butyl benzyl phthalate	SCM	EPA 8270
Cadmium	NPW	EPA 200.7
Cadmium	NPW	EPA 6010
Cadmium	SCM	EPA 6010
Calcium	NPW	EPA 200.7
Calcium	NPW	EPA 6010
Calcium	SCM	EPA 6010
Caprolactam	NPW	EPA 8270
Caprolactam	SCM	EPA 8270
Carbazole	NPW	EPA 8270
Carbazole	SCM	EPA 8270
Carbon disulfide	NPW	EPA 8260
Carbon disulfide	SCM	EPA 8260
Carbon tetrachloride	NPW	EPA 624
Carbon tetrachloride	NPW	EPA 8260
Carbon tetrachloride	SCM	EPA 8260
Carbonaceous BOD (CBOD)	NPW	SM 5210 B
Chemical oxygen demand	NPW	EPA 410.4
Chlordane (tech.)	NPW	EPA 608
Chlordane (tech.)	NPW	EPA 8081
Chlordane (tech.)	SCM	EPA 8081
Chloride	NPW	EPA 300.0
Chloride	NPW	EPA 9056
Chloride	SCM	EPA 9056
Chlorobenzene	NPW	EPA 624
Chlorobenzene	NPW	EPA 8260
Chlorobenzene	SCM	EPA 8260
Chloroethane	NPW	EPA 624
Chloroethane	NPW	EPA 8260
Chloroethane	SCM	EPA 8260
Chloroform	NPW	EPA 624
Chloroform	NPW	EPA 8260
Chloroform	SCM	EPA 8260
Chloroprene	NPW	EPA 8260
Chloroprene	SCM	EPA 8260
Chromium VI	NPW	EPA 7196
Chromium	NPW	EPA 200.7
Chromium	NPW	EPA 6010
Chromium	SCM	EPA 6010
Chrysene	NPW	EPA 625
Chrysene	NPW	EPA 8270
Chrysene	SCM	EPA 8270
cis-1,2-Dichloroethylene	NPW	EPA 8260
cis-1,2-Dichloroethylene	SCM	EPA 8260
cis-1,3-Dichloropropene	NPW	EPA 624
cis-1,3-Dichloropropene	NPW	EPA 8260
cis-1,3-Dichloropropene	SCM	EPA 8260
cis-1,4-Dichloro-2-butene	NPW	EPA 8260
cis-1,4-Dichloro-2-butene	SCM	EPA 8260
Cobalt	NPW	EPA 200.7
Cobalt	NPW	EPA 6010
Cobalt	SCM	EPA 6010
Conductivity	NPW	EPA 120.1
Copper	NPW	EPA 200.7
Copper	NPW	EPA 6010
Copper	SCM	EPA 6010
Corrosivity (pH)	SCM	EPA 9040
Cyclohexane	NPW	EPA 8260
Cyclohexane	SCM	EPA 8260
Dalapon	NPW	EPA 8151
delta-BHC	NPW	EPA 608
delta-BHC	NPW	EPA 8081
delta-BHC	SCM	EPA 8081
Dibenz(a,h)anthracene	NPW	EPA 625
Dibenz(a,h)anthracene	NPW	EPA 8270
Dibenz(a,h)anthracene	SCM	EPA 8270
Dibenzofuran	NPW	EPA 8270
Dibenzofuran	SCM	EPA 8270
Dibromochloromethane	NPW	EPA 624
Dibromochloromethane	NPW	EPA 8260
Dibromochloromethane	SCM	EPA 8260
Dibromomethane	NPW	EPA 8260
Dibromomethane	SCM	EPA 8260
Dicamba	NPW	EPA 8151
Dichlorodifluoromethane	NPW	EPA 624
Dichlorodifluoromethane	NPW	EPA 8260
Dichlorodifluoromethane	SCM	EPA 8260
Dichloroprop (Dichlorprop)	NPW	EPA 8151

Table 7.1 FL NELAP Parameter	Matrix Code	Approved Method
Dieldrin	NPW	EPA 608
Dieldrin	NPW	EPA 8081
Dieldrin	SCM	EPA 8081
Diesel range organics (DRO)	NPW	EPA 8015
Diesel range organics (DRO)	SCM	EPA 8015
Diethyl phthalate	NPW	EPA 625
Diethyl phthalate	NPW	EPA 8270
Diethyl phthalate	SCM	EPA 8270
Di-isopropylether (DIPE)	SCM	EPA 8260
Dimethyl phthalate	NPW	EPA 625
Dimethyl phthalate	NPW	EPA 8270
Dimethyl phthalate	SCM	EPA 8270
Di-n-butyl phthalate	NPW	EPA 625
Di-n-butyl phthalate	NPW	EPA 8270
Di-n-butyl phthalate	SCM	EPA 8270
Di-n-octyl phthalate	NPW	EPA 625
Di-n-octyl phthalate	NPW	EPA 8270
Di-n-octyl phthalate	SCM	EPA 8270
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	NPW	EPA 8151
Diphenylamine	NPW	EPA 8270
Diphenylamine	SCM	EPA 8270
Endosulfan I	NPW	EPA 608
Endosulfan I	NPW	EPA 8081
Endosulfan I	SCM	EPA 8081
Endosulfan II	NPW	EPA 608
Endosulfan II	NPW	EPA 8081
Endosulfan II	SCM	EPA 8081
Endosulfan sulfate	NPW	EPA 608
Endosulfan sulfate	NPW	EPA 8081
Endosulfan sulfate	SCM	EPA 8081
Endrin aldehyde	NPW	EPA 608
Endrin aldehyde	NPW	EPA 8081
Endrin aldehyde	SCM	EPA 8081
Endrin ketone	NPW	EPA 8081
Endrin ketone	SCM	EPA 8081
Endrin	NPW	EPA 608
Endrin	NPW	EPA 8081
Endrin	SCM	EPA 8081
Ethane	NPW	RSK-175
Ethyl methacrylate	NPW	EPA 8260
Ethyl methacrylate	SCM	EPA 8260
Ethylbenzene	NPW	EPA 624
Ethylbenzene	NPW	EPA 8260
Ethylbenzene	SCM	EPA 8260
Ethylene	NPW	RSK-175
Ethyl-t-butylether (ETBE)	NPW	EPA 8260
Ethyl-t-butylether (ETBE)	SCM	EPA 8260
Fluoranthene	NPW	EPA 625
Fluoranthene	NPW	EPA 8270
Fluoranthene	SCM	EPA 8270
Fluorene	NPW	EPA 625
Fluorene	NPW	EPA 8270
Fluorene	SCM	EPA 8270
Fluoride	NPW	EPA 300.0
Fluoride	NPW	EPA 9056
Fluoride	SCM	EPA 9056
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	NPW	EPA 608
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	NPW	EPA 8081
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	SCM	EPA 8081
gamma-Chlordane	NPW	EPA 8081
gamma-Chlordane	SCM	EPA 8081
Gasoline range organics (GRO)	NPW	EPA 8015
Gasoline range organics (GRO)	SCM	EPA 8015
Hardness (calc.)	NPW	EPA 200.7
Hardness	NPW	SM 2340 B
Heptachlor epoxide	NPW	EPA 608
Heptachlor epoxide	NPW	EPA 8081
Heptachlor epoxide	SCM	EPA 8081
Heptachlor	NPW	EPA 608
Heptachlor	NPW	EPA 8081
Heptachlor	SCM	EPA 8081
Hexachlorobenzene	NPW	EPA 625
Hexachlorobenzene	NPW	EPA 8270
Hexachlorobenzene	SCM	EPA 8270
Hexachlorobutadiene	NPW	EPA 625
Hexachlorobutadiene	NPW	EPA 8260
Hexachlorobutadiene	NPW	EPA 8270
Hexachlorobutadiene	SCM	EPA 8260
Hexachlorobutadiene	SCM	EPA 8270
Hexachlorocyclopentadiene	NPW	EPA 625
Hexachlorocyclopentadiene	NPW	EPA 8270
Hexachlorocyclopentadiene	SCM	EPA 8270
Hexachloroethane	NPW	EPA 625

Table 7.1 FL NELAP Parameter	Matrix Code	Approved Method
Hexachloroethane	NPW	EPA 8270
Hexachloroethane	SCM	EPA 8270
Ignitability	NPW	EPA 1010
Ignitability	SCM	EPA 1010
Indeno(1,2,3-cd)pyrene	NPW	EPA 625
Indeno(1,2,3-cd)pyrene	NPW	EPA 8270
Indeno(1,2,3-cd)pyrene	SCM	EPA 8270
Iodomethane (Methyl iodide)	NPW	EPA 8260
Iodomethane (Methyl iodide)	SCM	EPA 8260
Iron	NPW	EPA 200.7
Iron	NPW	EPA 6010
Iron	SCM	EPA 6010
Isobutyl alcohol (2-Methyl-1-propanol)	NPW	EPA 8260
Isobutyl alcohol (2-Methyl-1-propanol)	SCM	EPA 8260
Isophorone	NPW	EPA 625
Isophorone	NPW	EPA 8270
Isophorone	SCM	EPA 8270
Isopropyl ether	NPW	EPA 8260
Isopropylbenzene	NPW	EPA 8260
Isopropylbenzene	SCM	EPA 8260
Kjeldahl nitrogen - total	NPW	EPA 351.2
Lead	NPW	EPA 200.7
Lead	NPW	EPA 6010
Lead	SCM	EPA 6010
Magnesium	NPW	EPA 200.7
Magnesium	NPW	EPA 6010
Magnesium	SCM	EPA 6010
Manganese	NPW	EPA 200.7
Manganese	NPW	EPA 6010
Manganese	SCM	EPA 6010
MCPA	NPW	EPA 8151
MCPP	NPW	EPA 8151
Mercury	NPW	EPA 245.1
Mercury	NPW	EPA 7470
Mercury	SCM	EPA 7471
Methacrylonitrile	NPW	EPA 8260
Methacrylonitrile	SCM	EPA 8260
Methane	NPW	RSK-175
Methoxychlor	NPW	EPA 8081
Methoxychlor	SCM	EPA 8081
Methyl acetate	NPW	EPA 8260
Methyl acetate	SCM	EPA 8260
Methyl bromide (Bromomethane)	NPW	EPA 624
Methyl bromide (Bromomethane)	NPW	EPA 8260
Methyl bromide (Bromomethane)	SCM	EPA 8260
Methyl chloride (Chloromethane)	NPW	EPA 624
Methyl chloride (Chloromethane)	NPW	EPA 8260
Methyl chloride (Chloromethane)	SCM	EPA 8260
Methyl methacrylate	NPW	EPA 8260
Methyl methacrylate	SCM	EPA 8260
Methyl tert-butyl ether (MTBE)	NPW	EPA 8260
Methyl tert-butyl ether (MTBE)	SCM	EPA 8260
Methylcyclohexane	NPW	EPA 8260
Methylcyclohexane	SCM	EPA 8260
Methylene chloride	NPW	EPA 624
Methylene chloride	NPW	EPA 8260
Methylene chloride	SCM	EPA 8260
Molybdenum	NPW	EPA 200.7
Molybdenum	NPW	EPA 6010
Molybdenum	SCM	EPA 6010
m-Xylene	NPW	EPA 8260
m-Xylene	SCM	EPA 8260
Naphthalene	NPW	EPA 625
Naphthalene	NPW	EPA 8260
Naphthalene	NPW	EPA 8270
Naphthalene	SCM	EPA 8260
Naphthalene	SCM	EPA 8270
n-Butylbenzene	NPW	EPA 8260
n-Butylbenzene	SCM	EPA 8260
n-Hexane	NPW	EPA 8260
n-Hexane	SCM	EPA 8260
Nickel	NPW	EPA 200.7
Nickel	NPW	EPA 6010
Nickel	SCM	EPA 6010
Nitrate as N	NPW	EPA 300.0
Nitrate	NPW	EPA 9056
Nitrate	SCM	EPA 9056
Nitrite as N	NPW	SM 4500-NO2-B
Nitrite	NPW	EPA 300.0
Nitrite	NPW	EPA 9056
Nitrite	SCM	EPA 9056
Nitrobenzene	NPW	EPA 625
Nitrobenzene	NPW	EPA 8270

Table 7.1 FL NELAP Parameter	Matrix Code	Approved Method
Nitrobenzene	NPW	EPA 8330
Nitrobenzene	SCM	EPA 8270
Nitrobenzene	SCM	EPA 8330
Nitrocellulose	NPW	EL SOP 234/UV-VIS
Nitrocellulose	SCM	EL SOP 234/UV-VIS
Nitroglycerin	NPW	EPA 8330
Nitroglycerin	NPW	EPA 8332
Nitroglycerin	SCM	EPA 8330
Nitroglycerin	SCM	EPA 8332
Nitroguanidine	NPW	EL SOP 233/HPLC-UV
Nitroguanidine	SCM	EL SOP 233/HPLC-UV
Nitroguanidine	SCM	EPA 8330
n-Nitrosodimethylamine	NPW	EPA 625
n-Nitrosodimethylamine	NPW	EPA 8270
n-Nitrosodimethylamine	SCM	EPA 8270
n-Nitrosodi-n-propylamine	NPW	EPA 625
n-Nitrosodi-n-propylamine	NPW	EPA 8270
n-Nitrosodi-n-propylamine	SCM	EPA 8270
n-Nitrosodiphenylamine	NPW	EPA 625
n-Nitrosodiphenylamine	NPW	EPA 8270
n-Nitrosodiphenylamine	SCM	EPA 8270
n-Propylbenzene	NPW	EPA 8260
n-Propylbenzene	SCM	EPA 8260
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	NPW	EPA 8330
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	SCM	EPA 8330
Oil & Grease	NPW	EPA 1664A
Orthophosphate as P	NPW	SM 4500-P E
o-Xylene	NPW	EPA 8260
o-Xylene	SCM	EPA 8260
Paint Filter Liquids Test	SCM	EPA 9095
Pentachlorophenol	NPW	EPA 625
Pentachlorophenol	NPW	EPA 8270
Pentachlorophenol	SCM	EPA 8270
Pentaerythritoltetranitrate (PETN)	NPW	EPA 8330
Pentaerythritoltetranitrate (PETN)	SCM	EPA 8330
Perchlorate	NPW	EPA 6850
Perchlorate	SCM	EPA 6850
pH	NPW	EPA 9040
pH	NPW	SM 4500-H+-B
pH	SCM	EPA 9040
pH	SCM	EPA 9045
Phenanthrene	NPW	EPA 625
Phenanthrene	NPW	EPA 8270
Phenanthrene	SCM	EPA 8270
Phenol	NPW	EPA 625
Phenol	NPW	EPA 8270
Phenol	SCM	EPA 8270
Phosphorus, total	NPW	SM 4500-P E
p-Isopropyltoluene	NPW	EPA 8260
p-Isopropyltoluene	SCM	EPA 8260
Potassium	NPW	EPA 200.7
Potassium	NPW	EPA 6010
Potassium	SCM	EPA 6010
Propionitrile (Ethyl cyanide)	NPW	EPA 8260
Propionitrile (Ethyl cyanide)	SCM	EPA 8260
p-Xylene	NPW	EPA 8260
p-Xylene	SCM	EPA 8260
Pyrene	NPW	EPA 625
Pyrene	NPW	EPA 8270
Pyrene	SCM	EPA 8270
Pyridine	NPW	EPA 625
Pyridine	NPW	EPA 8270
Pyridine	SCM	EPA 8270
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	NPW	EPA 8330
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	SCM	EPA 8330
Reactive sulfide	SCM	Sec. 7.3 SW-846
Residue-filterable (TDS)	NPW	SM 2540 C
Residue-nonfilterable (TSS)	NPW	SM 2540 D
Residue-total	NPW	SM 2540 B
sec-Butylbenzene	NPW	EPA 8260
sec-Butylbenzene	SCM	EPA 8260
Selenium	NPW	EPA 200.7
Selenium	NPW	EPA 6010
Selenium	SCM	EPA 6010
Silver	NPW	EPA 200.7
Silver	NPW	EPA 6010
Silver	SCM	EPA 6010
Silvex (2,4,5-TP)	NPW	EPA 8151
Sodium	NPW	EPA 200.7
Sodium	NPW	EPA 6010
Sodium	SCM	EPA 6010
Strontium	NPW	EPA 200.7
Strontium	NPW	EPA 6010

Table 7.1 FL NELAP Parameter	Matrix Code	Approved Method
Strontium	SCM	EPA 6010
Styrene	NPW	EPA 8260
Styrene	SCM	EPA 8260
Sulfate	NPW	EPA 300.0
Sulfate	NPW	EPA 9056
Sulfate	SCM	EPA 9056
Sulfide	NPW	SM 4500-S F
Synthetic Precipitation Leaching Procedure	SCM	EPA 1312
T-amylethylether (TAEE)	NPW	EPA 8260
T-amylethylether (TAEE)	SCM	EPA 8260
T-amylmethylether (TAME)	NPW	EPA 8260
T-amylmethylether (TAME)	SCM	EPA 8260
tert-Amyl alcohol (2-methyl-2-butanol)	NPW	EPA 8260
tert-Amyl alcohol (2-methyl-2-butanol)	SCM	EPA 8260
tert-Butyl alcohol (2-Methyl-2-propanol)	NPW	EPA 8260
tert-Butyl alcohol (2-Methyl-2-propanol)	SCM	EPA 8260
tert-Butylbenzene	NPW	EPA 8260
tert-Butylbenzene	SCM	EPA 8260
Tetrachloroethylene (Perchloroethylene)	NPW	EPA 624
Tetrachloroethylene (Perchloroethylene)	NPW	EPA 8260
Tetrachloroethylene (Perchloroethylene)	SCM	EPA 8260
Tetrahydrofuran (THF)	NPW	EPA 8260
Tetrahydrofuran (THF)	SCM	EPA 8260
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	NPW	EPA 8330
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	SCM	EPA 8330
Thallium	NPW	EPA 200.7
Thallium	NPW	EPA 6010
Thallium	SCM	EPA 6010
Tin	NPW	EPA 200.7
Tin	NPW	EPA 6010
Tin	SCM	EPA 6010
Titanium	NPW	EPA 200.7
Titanium	NPW	EPA 6010
Titanium	SCM	EPA 6010
Toluene	NPW	EPA 624
Toluene	NPW	EPA 8260
Toluene	SCM	EPA 8260
Total cyanide	NPW	EPA 9012
Total cyanide	SCM	EPA 9012
Total nitrate-nitrite	NPW	EPA 353.2
Total organic carbon	NPW	EPA 9060
Total organic carbon	NPW	SM 5310 C
Total organic carbon	SCM	EPA 9060
Total organic carbon	SCM	Lloyd Khan
Total Petroleum Hydrocarbons (TPH)	NPW	FL-PRO
Total Petroleum Hydrocarbons (TPH)	SCM	FL-PRO
Total phenolics	NPW	EPA 420.4
Toxaphene (Chlorinated camphene)	NPW	EPA 608
Toxaphene (Chlorinated camphene)	NPW	EPA 8081
Toxaphene (Chlorinated camphene)	SCM	EPA 8081
Toxicity Characteristic Leaching Procedure	SCM	EPA 1311
trans-1,2-Dichloroethylene	NPW	EPA 624
trans-1,2-Dichloroethylene	NPW	EPA 8260
trans-1,2-Dichloroethylene	SCM	EPA 8260
trans-1,3-Dichloropropene	NPW	EPA 624
trans-1,3-Dichloropropene	NPW	EPA 8260
trans-1,3-Dichloropropene	SCM	EPA 8260
trans-1,4-Dichloro-2-butene	NPW	EPA 8260
trans-1,4-Dichloro-2-butene	SCM	EPA 8260
Trichloroethene (Trichloroethylene)	NPW	EPA 624
Trichloroethene (Trichloroethylene)	NPW	EPA 8260
Trichloroethene (Trichloroethylene)	SCM	EPA 8260
Trichlorofluoromethane	NPW	EPA 624
Trichlorofluoromethane	NPW	EPA 8260
Trichlorofluoromethane	SCM	EPA 8260
Vanadium	NPW	EPA 200.7
Vanadium	NPW	EPA 6010
Vanadium	SCM	EPA 6010
Vinyl acetate	NPW	EPA 8260
Vinyl acetate	SCM	EPA 8260
Vinyl chloride	NPW	EPA 624
Vinyl chloride	NPW	EPA 8260
Vinyl chloride	SCM	EPA 8260
Xylene (total)	NPW	EPA 624
Xylene (total)	NPW	EPA 8260
Xylene (total)	SCM	EPA 8260
Zinc	NPW	EPA 200.7
Zinc	NPW	EPA 6010
Zinc	SCM	EPA 6010

Table 7.2 DOD ELAP Parameter (QSM4.2 or QSM5.0)	Matrix Code	Approved Method
1,1,1,2-Tetrachloroethane	NPW	EPA 8260B; EPA 624

Table 7.2 DOD ELAP Parameter (QSM4.2 or QSM5.0)	Matrix Code	Approved Method
1,1,1,2-Tetrachloroethane	SCM	EPA 8260B
1,1,1-Trichloroethane (1,1,1-TCA)	NPW	EPA 8260B; EPA 624
1,1,1-Trichloroethane (1,1,1-TCA)	SCM	EPA 8260B
1,1,2,2-Tetrachloroethane	NPW	EPA 8260B; EPA 624
1,1,2,2-Tetrachloroethane	SCM	EPA 8260B
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	NPW	EPA 8260B; EPA 624
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	SCM	EPA 8260B
1,1,2-Trichloroethane	NPW	EPA 8260B; EPA 624
1,1,2-Trichloroethane	SCM	EPA 8260B
1,1'-Biphenyl	NPW	EPA 8270C/D; EPA 625
1,1'-Biphenyl	SCM	EPA 8270C/D
1,1-Dichloroethane (1,1-DCA)	NPW	EPA 8260B; EPA 624
1,1-Dichloroethane (1,1-DCA)	SCM	EPA 8260B
1,1-Dichloroethene (1,1-DCE)	NPW	EPA 8260B; EPA 624
1,1-Dichloroethene (1,1-DCE)	SCM	EPA 8260B
1,1-Dichloropropene	NPW	EPA 8260B; EPA 624
1,1-Dichloropropene	SCM	EPA 8260B
1,2,3-Trichlorobenzene	NPW	EPA 8260B; EPA 624
1,2,3-Trichlorobenzene	SCM	EPA 8260B
1,2,3-Trichloropropane	NPW	EPA 8260B; EPA 624
1,2,3-Trichloropropane	SCM	EPA 8260B
1,2,4,5-Tetrachlorobenzene	NPW	EPA 8270C/D; EPA 625
1,2,4,5-Tetrachlorobenzene	SCM	EPA 8270C/D
1,2,4-Trichlorobenzene	NPW	EPA 8260B; EPA 624
1,2,4-Trichlorobenzene	NPW	EPA 8270C/D; EPA 625
1,2,4-Trichlorobenzene	SCM	EPA 8260B
1,2,4-Trichlorobenzene	SCM	EPA 8270C/D
1,2,4-Trimethylbenzene	NPW	EPA 8260B; EPA 624
1,2,4-Trimethylbenzene	SCM	EPA 8260B
1,2-Dibromo-3-chloropropane (DBCP)	NPW	EPA 8011
1,2-Dibromo-3-chloropropane (DBCP)	NPW	EPA 8260B; EPA 624
1,2-Dibromo-3-chloropropane (DBCP)	SCM	EPA 8260B
1,2-Dibromoethane (EDB)	NPW	EPA 8011
1,2-Dibromoethane (EDB)	NPW	EPA 8260B; EPA 624
1,2-Dibromoethane (EDB)	SCM	EPA 8260B
1,2-Dichlorobenzene	NPW	EPA 8260B; EPA 624
1,2-Dichlorobenzene	NPW	EPA 8270C/D; EPA 625
1,2-Dichlorobenzene	SCM	EPA 8260B
1,2-Dichlorobenzene	SCM	EPA 8270C/D
1,2-Dichloroethane (EDC)	NPW	EPA 8260B; EPA 624
1,2-Dichloroethane (EDC)	SCM	EPA 8260B
1,2-Dichloropropane	NPW	EPA 8260B; EPA 624
1,2-Dichloropropane	SCM	EPA 8260B
1,2-Diphenylhydrazine	NPW	EPA 8270C/D; EPA 625
1,2-Diphenylhydrazine	SCM	EPA 8270C/D
1,3,5-Trimethylbenzene	NPW	EPA 8260B; EPA 624
1,3,5-Trimethylbenzene	SCM	EPA 8260B
1,3,5-Trinitrobenzene	NPW	EPA 8330A/B
1,3,5-Trinitrobenzene	SCM	EPA 8330A
1,3,5-Trinitrobenzene	SCM	EPA 8330B
1,3-Dichlorobenzene	NPW	EPA 8260B; EPA 624
1,3-Dichlorobenzene	NPW	EPA 8270C/D; EPA 625
1,3-Dichlorobenzene	SCM	EPA 8260B
1,3-Dichlorobenzene	SCM	EPA 8270C/D
1,3-Dichloropropane	NPW	EPA 8260B; EPA 624
1,3-Dichloropropane	SCM	EPA 8260B
1,3-Dinitrobenzene	NPW	EPA 8330A/B
1,3-Dinitrobenzene	SCM	EPA 8330A
1,3-Dinitrobenzene	SCM	EPA 8330B
1,4-Dichlorobenzene	NPW	EPA 8260B; EPA 624
1,4-Dichlorobenzene	NPW	EPA 8270C/D; EPA 625
1,4-Dichlorobenzene	SCM	EPA 8260B
1,4-Dichlorobenzene	SCM	EPA 8270C/D
1,4-Dioxane	NPW	EPA 8260B; EPA 624
1,4-Dioxane	NPW	EPA 8270C/D; EPA 625
1,4-Dioxane	SCM	EPA 8260B
1,4-Dioxane	SCM	EPA 8270C/D
1-Chlorohexane	NPW	EPA 8260B; EPA 624
1-Chlorohexane	SCM	EPA 8260B
1-Methylnaphthalene	NPW	EPA 8270C/D; EPA 625
1-Methylnaphthalene	SCM	EPA 8270C/D
2,2-Dichloropropane	NPW	EPA 8260B; EPA 624
2,2-Dichloropropane	SCM	EPA 8260B
2,3,4,6-Tetrachlorophenol	NPW	EPA 8270C/D; EPA 625
2,3,4,6-Tetrachlorophenol	SCM	EPA 8270C/D
2,4,5-T	NPW	EPA 8151A
2,4,5-TP (Silvex)	NPW	EPA 8151A
2,4,5-Trichlorophenol	NPW	EPA 8270C/D; EPA 625

Table 7.2 DOD ELAP Parameter (QSM4.2 or QSM5.0)	Matrix Code	Approved Method
2,4,5-Trichlorophenol	SCM	EPA 8270C/D
2,4,6-Trichlorophenol (TCP)	NPW	EPA 8270C/D; EPA 625
2,4,6-Trichlorophenol (TCP)	SCM	EPA 8270C/D
2,4,6-Trinitrophenylmethylnitramine (Tetryl)	NPW	EPA 8330A/B
2,4,6-Trinitrophenylmethylnitramine (Tetryl)	SCM	EPA 8330A
2,4,6-Trinitrophenylmethylnitramine (Tetryl)	SCM	EPA 8330B
2,4,6-Trinitrotoluene (TNT)	NPW	EPA 8330A/B
2,4,6-Trinitrotoluene (TNT)	SCM	EPA 8330A
2,4,6-Trinitrotoluene (TNT)	SCM	EPA 8330B
2,4-D	NPW	EPA 8151A
2,4-DB	NPW	EPA 8151A
2,4-Dichlorophenol (DCP)	NPW	EPA 8270C/D; EPA 625
2,4-Dichlorophenol (DCP)	SCM	EPA 8270C/D
2,4-Dimethylphenol	NPW	EPA 8270C/D; EPA 625
2,4-Dimethylphenol	SCM	EPA 8270C/D
2,4-Dinitrophenol	NPW	EPA 8270C/D; EPA 625
2,4-Dinitrophenol	SCM	EPA 8270C/D
2,4-Dinitrotoluene (DNT)	NPW	EPA 8270C/D; EPA 625
2,4-Dinitrotoluene (DNT)	NPW	EPA 8330A/B
2,4-Dinitrotoluene (DNT)	SCM	EPA 8270C/D
2,4-Dinitrotoluene (DNT)	SCM	EPA 8330A
2,4-Dinitrotoluene (DNT)	SCM	EPA 8330B
2,6-Dichlorophenol	NPW	EPA 8270C/D; EPA 625
2,6-Dichlorophenol	SCM	EPA 8270C/D
2,6-Dinitrotoluene	NPW	EPA 8270C/D; EPA 625
2,6-Dinitrotoluene	NPW	EPA 8330A/B
2,6-Dinitrotoluene	SCM	EPA 8270C/D
2,6-Dinitrotoluene	SCM	EPA 8330A
2,6-Dinitrotoluene	SCM	EPA 8330B
2-Amino-4,6-dinitrotoluene	NPW	EPA 8330A/B
2-Amino-4,6-dinitrotoluene	SCM	EPA 8330A
2-Amino-4,6-dinitrotoluene	SCM	EPA 8330B
2-Butanone (Methyl ethyl ketone; MEK)	NPW	EPA 8260B; EPA 624
2-Butanone (Methyl ethyl ketone; MEK)	SCM	EPA 8260B
2-Chloroethyl vinyl ether	NPW	EPA 8260B; EPA 624
2-Chloroethyl vinyl ether	SCM	EPA 8260B
2-Chloronaphthalene	NPW	EPA 8270C/D; EPA 625
2-Chloronaphthalene	SCM	EPA 8270C/D
2-Chlorophenol	NPW	EPA 8270C/D; EPA 625
2-Chlorophenol	SCM	EPA 8270C/D
2-Chlorotoluene	NPW	EPA 8260B; EPA 624
2-Chlorotoluene	SCM	EPA 8260B
2-Hexanone (Methyl butyl ketone; MBK)	NPW	EPA 8260B; EPA 624
2-Hexanone (Methyl butyl ketone; MBK)	SCM	EPA 8260B
2-Methylnaphthalene	NPW	EPA 8270C/D; EPA 625
2-Methylnaphthalene	SCM	EPA 8270C/D
2-Methylphenol (o-Cresol)	NPW	EPA 8270C/D; EPA 625
2-Methylphenol (o-Cresol)	SCM	EPA 8270C/D
2-Nitroaniline	NPW	EPA 8270C/D; EPA 625
2-Nitroaniline	SCM	EPA 8270C/D
2-Nitrophenol (ONP)	NPW	EPA 8270C/D; EPA 625
2-Nitrophenol (ONP)	SCM	EPA 8270C/D
2-Nitrotoluene (ONT)	NPW	EPA 8330A/B
2-Nitrotoluene (ONT)	SCM	EPA 8330A
2-Nitrotoluene (ONT)	SCM	EPA 8330B
3,3'-Dichlorobenzidine (DCB)	NPW	EPA 8270C/D; EPA 625
3,3'-Dichlorobenzidine (DCB)	SCM	EPA 8270C/D
3,5-Dinitroaniline	NPW	EPA 8330A/B
3,5-Dinitroaniline	NPW	EPA 8330A/B
3,5-Dinitroaniline	SCM	EPA 8330A
3,5-Dinitroaniline	SCM	EPA 8330B
3-Methylphenol/4-Methylphenol	NPW	EPA 8270C/D; EPA 625
3-Methylphenol/4-Methylphenol	SCM	EPA 8270C/D
3-Nitroaniline	NPW	EPA 8270C/D; EPA 625
3-Nitroaniline	SCM	EPA 8270C/D
3-Nitrotoluene	NPW	EPA 8330A/B
3-Nitrotoluene	SCM	EPA 8330A
3-Nitrotoluene	SCM	EPA 8330B
4,4'-DDD	NPW	EPA 8081A/B
4,4'-DDD	SCM	EPA 8081A/B
4,4'-DDE	NPW	EPA 8081A/B
4,4'-DDE	SCM	EPA 8081A/B
4,4'-DDT	NPW	EPA 8081A/B
4,4'-DDT	SCM	EPA 8081A/B
4,6-Dinitro-2-methylphenol (DNOC)	NPW	EPA 8270C/D; EPA 625
4,6-Dinitro-2-methylphenol (DNOC)	SCM	EPA 8270C/D
4-Amino-2,6-dinitrotoluene	NPW	EPA 8330A/B
4-Amino-2,6-dinitrotoluene	SCM	EPA 8330A

Table 7.2 DOD ELAP Parameter (QSM4.2 or QSM5.0)	Matrix Code	Approved Method
4-Amino-2,6-dinitrotoluene	SCM	EPA 8330B
4-Bromophenyl phenyl ether	NPW	EPA 8270C/D; EPA 625
4-Bromophenyl phenyl ether	SCM	EPA 8270C/D
4-Chloro-3-methylphenol	NPW	EPA 8270C/D; EPA 625
4-Chloro-3-methylphenol	SCM	EPA 8270C/D
4-Chloroaniline	NPW	EPA 8270C/D; EPA 625
4-Chloroaniline	SCM	EPA 8270C/D
4-Chlorophenyl phenyl ether	NPW	EPA 8270C/D; EPA 625
4-Chlorophenyl phenyl ether	SCM	EPA 8270C/D
4-Chlorotoluene	NPW	EPA 8260B; EPA 624
4-Chlorotoluene	SCM	EPA 8260B
4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	NPW	EPA 8260B; EPA 624
4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	SCM	EPA 8260B
4-Methylphenol (p-Cresol)	NPW	EPA 8270C/D; EPA 625
4-Methylphenol (p-Cresol)	SCM	EPA 8270C/D
4-Nitroaniline (PNA)	NPW	EPA 8270C/D; EPA 625
4-Nitroaniline (PNA)	SCM	EPA 8270C/D
4-Nitrophenol (PNP)	NPW	EPA 8270C/D; EPA 625
4-Nitrophenol (PNP)	SCM	EPA 8270C/D
4-Nitrotoluene (PNT)	NPW	EPA 8330A/B
4-Nitrotoluene (PNT)	SCM	EPA 8330A
4-Nitrotoluene (PNT)	SCM	EPA 8330B
Acenaphthene	NPW	EPA 8270C/D; EPA 625
Acenaphthene	SCM	EPA 8270C/D
Acenaphthylene	NPW	EPA 8270C/D; EPA 625
Acenaphthylene	SCM	EPA 8270C/D
Acetone	NPW	EPA 8260B; EPA 624
Acetone	SCM	EPA 8260B
Acetonitrile	NPW	EPA 8260B; EPA 624
Acetonitrile	SCM	EPA 8260B
Acetophenone	NPW	EPA 8270C/D; EPA 625
Acetophenone	SCM	EPA 8270C/D
Acrolein	NPW	EPA 8260B; EPA 624
Acrolein	SCM	EPA 8260B
Acrylonitrile	NPW	EPA 8260B; EPA 624
Acrylonitrile	SCM	EPA 8260B
Aldrin	NPW	EPA 8081A/B
Aldrin	SCM	EPA 8081A/B
Alkalinity	NPW	SM 2320 B-2011
Allyl chloride	NPW	EPA 8260B; EPA 624
Allyl chloride	SCM	EPA 8260B
alpha-BHC (alpha-HCH)	NPW	EPA 8081A/B
alpha-BHC (alpha-HCH)	SCM	EPA 8081A/B
alpha-Chlordane	NPW	EPA 8081A/B
alpha-Chlordane	SCM	EPA 8081A/B
Aluminum	NPW	EPA 6010B/C; EPA 200.7
Aluminum	SCM	EPA 6010B/C
Ammonia	NPW	SM 4500-NH3 G-2011
Aniline	NPW	EPA 8270C/D; EPA 625
Aniline	SCM	EPA 8270C/D
Anthracene	NPW	EPA 8270C/D; EPA 625
Anthracene	SCM	EPA 8270C/D
Antimony	NPW	EPA 6010B/C; EPA 200.7
Antimony	SCM	EPA 6010B/C
Aroclor-1016	NPW	EPA 8082A
Aroclor-1016	SCM	EPA 8082A
Aroclor-1221	NPW	EPA 8082A
Aroclor-1221	SCM	EPA 8082A
Aroclor-1232	NPW	EPA 8082A
Aroclor-1232	SCM	EPA 8082A
Aroclor-1242	NPW	EPA 8082A
Aroclor-1242	SCM	EPA 8082A
Aroclor-1248	NPW	EPA 8082A
Aroclor-1248	SCM	EPA 8082A
Aroclor-1254	NPW	EPA 8082A
Aroclor-1254	SCM	EPA 8082A
Aroclor-1260	NPW	EPA 8082A
Aroclor-1260	SCM	EPA 8082A
Aroclor-1262	NPW	EPA 8082A
Aroclor-1262	SCM	EPA 8082A
Aroclor-1268	NPW	EPA 8082A
Aroclor-1268	SCM	EPA 8082A
Arsenic	NPW	EPA 6010B/C; EPA 200.7
Arsenic	SCM	EPA 6010B/C
Atrazine	NPW	EPA 8270C/D; EPA 625
Atrazine	SCM	EPA 8270C/D
Barium	NPW	EPA 6010B/C; EPA 200.7
Barium	SCM	EPA 6010B/C

Table 7.2 DOD ELAP Parameter (QSM4.2 or QSM5.0)	Matrix Code	Approved Method
Benzaldehyde	NPW	EPA 8270C/D; EPA 625
Benzaldehyde	SCM	EPA 8270C/D
Benzene	NPW	EPA 8260B; EPA 624
Benzene	SCM	EPA 8260B
Benzidine	NPW	EPA 8270C/D; EPA 625
Benzidine	SCM	EPA 8270C/D
Benzo(a)anthracene	NPW	EPA 8270C/D; EPA 625
Benzo(a)anthracene	SCM	EPA 8270C/D
Benzo(a)pyrene	NPW	EPA 8270C/D; EPA 625
Benzo(a)pyrene	SCM	EPA 8270C/D
Benzo(b)fluoranthene	NPW	EPA 8270C/D; EPA 625
Benzo(b)fluoranthene	SCM	EPA 8270C/D
Benzo(g,h,i)perylene	NPW	EPA 8270C/D; EPA 625
Benzo(g,h,i)perylene	SCM	EPA 8270C/D
Benzo(k)fluoranthene	NPW	EPA 8270C/D; EPA 625
Benzo(k)fluoranthene	SCM	EPA 8270C/D
Benzoic Acid	NPW	EPA 8270C/D; EPA 625
Benzoic Acid	SCM	EPA 8270C/D
Benzyl alcohol	NPW	EPA 8270C/D; EPA 625
Benzyl alcohol	SCM	EPA 8270C/D
Beryllium	NPW	EPA 6010B/C; EPA 200.7
Beryllium	SCM	EPA 6010B/C
beta-BHC (beta-HCH)	NPW	EPA 8081A/B
beta-BHC (beta-HCH)	SCM	EPA 8081A/B
bis(2-Chloroethoxy)methane	NPW	EPA 8270C/D; EPA 625
bis(2-Chloroethoxy)methane	SCM	EPA 8270C/D
bis(2-Chloroethyl)ether (BCEE)	NPW	EPA 8270C/D; EPA 625
bis(2-Chloroethyl)ether (BCEE)	SCM	EPA 8270C/D
bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)	NPW	EPA 8270C/D; EPA 625
bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)	SCM	EPA 8270C/D
bis(2-Ethylhexyl)phthalate (BEHP)	NPW	EPA 8270C/D; EPA 625
bis(2-Ethylhexyl)phthalate (BEHP)	SCM	EPA 8270C/D
BOD	NPW	SM 5210 B-2011
Boron	NPW	EPA 6010B/C; EPA 200.7
Boron	SCM	EPA 6010B/C
Bromide	NPW	EPA 300.0
Bromide	NPW	EPA 9056A
Bromide	SCM	EPA 9056A
Bromobenzene	NPW	EPA 8260B; EPA 624
Bromobenzene	SCM	EPA 8260B
Bromochloromethane	NPW	EPA 8260B; EPA 624
Bromochloromethane	SCM	EPA 8260B
Bromodichloromethane	NPW	EPA 8260B; EPA 624
Bromodichloromethane	SCM	EPA 8260B
Bromoform	NPW	EPA 8260B; EPA 624
Bromoform	SCM	EPA 8260B
Bromomethane	NPW	EPA 8260B; EPA 624
Bromomethane	SCM	EPA 8260B
Butyl benzyl phthalate (BBP)	NPW	EPA 8270C/D; EPA 625
Butyl benzyl phthalate (BBP)	SCM	EPA 8270C/D
Cadmium	NPW	EPA 6010B/C; EPA 200.7
Cadmium	SCM	EPA 6010B/C
Calcium	NPW	EPA 6010B/C; EPA 200.7
Calcium	SCM	EPA 6010B/C
Caprolactam	NPW	EPA 8270C/D; EPA 625
Caprolactam	SCM	EPA 8270C/D
Carbazole	NPW	EPA 8270C/D; EPA 625
Carbazole	SCM	EPA 8270C/D
Carbon Disulfide	NPW	EPA 8260B; EPA 624
Carbon Disulfide	SCM	EPA 8260B
Carbon Tetrachloride	NPW	EPA 8260B; EPA 624
Carbon Tetrachloride	SCM	EPA 8260B
CBOD	NPW	SM 5210 B-2011
Chlordane (n.o.s.)	NPW	EPA 8081A/B
Chlordane (n.o.s.)	SCM	EPA 8081A/B
Chloride	NPW	EPA 300.0
Chloride	NPW	EPA 9056A
Chloride	SCM	EPA 9056A
Chlorobenzene	NPW	EPA 8260B; EPA 624
Chlorobenzene	SCM	EPA 8260B
Chloroethane	NPW	EPA 8260B; EPA 624
Chloroethane	SCM	EPA 8260B
Chloroform	NPW	EPA 8260B; EPA 624
Chloroform	SCM	EPA 8260B
Chloromethane	NPW	EPA 8260B; EPA 624
Chloromethane	SCM	EPA 8260B
Chloroprene	NPW	EPA 8260B; EPA 624
Chloroprene	SCM	EPA 8260B

Table 7.2 DOD ELAP Parameter (QSM4.2 or QSM5.0)	Matrix Code	Approved Method
Chromium, total	NPW	EPA 6010B/C; EPA 200.7
Chromium, total	SCM	EPA 6010B/C
Chrysene	NPW	EPA 8270C/D; EPA 625
Chrysene	SCM	EPA 8270C/D
cis-1,2-Dichloroethene (cis-1,2-DCE)	NPW	EPA 8260B; EPA 624
cis-1,2-Dichloroethene (cis-1,2-DCE)	SCM	EPA 8260B
cis-1,3-Dichloropropene	NPW	EPA 8260B; EPA 624
cis-1,3-Dichloropropene	SCM	EPA 8260B
cis-1,4-Dichloro-2-butene	NPW	EPA 8260B; EPA 624
cis-1,4-Dichloro-2-butene	SCM	EPA 8260B
Cobalt	NPW	EPA 6010B/C; EPA 200.7
Cobalt	SCM	EPA 6010B/C
COD	NPW	EPA 410.4
Copper	NPW	EPA 6010B/C; EPA 200.7
Copper	SCM	EPA 6010B/C
Cyanide	NPW	EPA 9012A/B,
Cyanide	SCM	EPA 9012A/B
Cyclohexane	NPW	EPA 8260B; EPA 624
Cyclohexane	SCM	EPA 8260B
Dalapon	NPW	EPA 8151A
delta-BHC (delta-HCH)	NPW	EPA 8081A/B
delta-BHC (delta-HCH)	SCM	EPA 8081A/B
Dibenz(a,h)anthracene	NPW	EPA 8270C/D; EPA 625
Dibenz(a,h)anthracene	SCM	EPA 8270C/D
Dibenzofuran (DBF)	NPW	EPA 8270C/D; EPA 625
Dibenzofuran (DBF)	SCM	EPA 8270C/D
Dibromochloromethane	NPW	EPA 8260B; EPA 624
Dibromochloromethane	SCM	EPA 8260B
Dibromomethane	NPW	EPA 8260B; EPA 624
Dibromomethane	SCM	EPA 8260B
Dicamba	NPW	EPA 8151A
Dichlorodifluoromethane (CFC-12)	NPW	EPA 8260B; EPA 624
Dichlorodifluoromethane (CFC-12)	SCM	EPA 8260B
Dichlorprop	NPW	EPA 8151A
Dieldrin	NPW	EPA 8081A/B
Dieldrin	SCM	EPA 8081A/B
Diethyl phthalate (DEP)	NPW	EPA 8270C/D; EPA 625
Diethyl phthalate (DEP)	SCM	EPA 8270C/D
Di-isopropyl ether	NPW	EPA 8260B; EPA 624
Di-isopropyl ether	SCM	EPA 8260B
Dimethyl phthalate (DMP)	NPW	EPA 8270C/D; EPA 625
Dimethyl phthalate (DMP)	SCM	EPA 8270C/D
Di-n-butyl phthalate (DBP)	NPW	EPA 8270C/D; EPA 625
Di-n-butyl phthalate (DBP)	SCM	EPA 8270C/D
Di-n-octyl phthalate (DNOP)	NPW	EPA 8270C/D; EPA 625
Di-n-octyl phthalate (DNOP)	SCM	EPA 8270C/D
Dinoseb	NPW	EPA 8151A
Endosulfan I	NPW	EPA 8081A/B
Endosulfan I	SCM	EPA 8081A/B
Endosulfan II	NPW	EPA 8081A/B
Endosulfan II	SCM	EPA 8081A/B
Endosulfan sulfate	NPW	EPA 8081A/B
Endosulfan sulfate	SCM	EPA 8081A/B
Endrin	NPW	EPA 8081A/B
Endrin	SCM	EPA 8081A/B
Endrin aldehyde	NPW	EPA 8081A/B
Endrin aldehyde	SCM	EPA 8081A/B
Endrin ketone	NPW	EPA 8081A/B
Endrin ketone	SCM	EPA 8081A/B
ETBE	NPW	EPA 8260B; EPA 624
ETBE	SCM	EPA 8260B
Ethane	NPW	RSK-175
Ethene	NPW	RSK-175
Ethyl methacrylate	NPW	EPA 8260B; EPA 624
Ethyl methacrylate	SCM	EPA 8260B
Ethylbenzene	NPW	EPA 8260B; EPA 624
Ethylbenzene	SCM	EPA 8260B
Fluoranthene	NPW	EPA 8270C/D; EPA 625
Fluoranthene	SCM	EPA 8270C/D
Fluorene	NPW	EPA 8270C/D; EPA 625
Fluorene	SCM	EPA 8270C/D
Fluoride	NPW	EPA 300.0
Fluoride	NPW	EPA 9056A
Fluoride	SCM	EPA 9056A
gamma-BHC (Lindane; gamma-HCH)	NPW	EPA 8081A/B
gamma-BHC (Lindane; gamma-HCH)	SCM	EPA 8081A/B
gamma-Chlordane	NPW	EPA 8081A/B
gamma-Chlordane	SCM	EPA 8081A/B

Table 7.2 DOD ELAP Parameter (QSM4.2 or QSM5.0)	Matrix Code	Approved Method
Heptachlor	NPW	EPA 8081A/B
Heptachlor	SCM	EPA 8081A/B
Heptachlor epoxide	NPW	EPA 8081A/B
Heptachlor epoxide	SCM	EPA 8081A/B
Hexachlorobenzene (HCB)	NPW	EPA 8270C/D; EPA 625
Hexachlorobenzene (HCB)	SCM	EPA 8270C/D
Hexachlorobutadiene	NPW	EPA 8260B; EPA 624
Hexachlorobutadiene	SCM	EPA 8260B
Hexachlorobutadiene (HCBD)	NPW	EPA 8270C/D; EPA 625
Hexachlorobutadiene (HCBD)	SCM	EPA 8270C/D
Hexachlorocyclopentadiene (HCCPD)	NPW	EPA 8270C/D; EPA 625
Hexachlorocyclopentadiene (HCCPD)	SCM	EPA 8270C/D
Hexachloroethane (HCE)	NPW	EPA 8270C/D; EPA 625
Hexachloroethane (HCE)	SCM	EPA 8270C/D
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	NPW	EPA 8330A/B
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	SCM	EPA 8330A
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	SCM	EPA 8330B
Hexane	NPW	EPA 8260B; EPA 624
Hexane	SCM	EPA 8260B
Hexavalent Chromium	NPW	EPA 7196A
Hexavalent Chromium	SCM	EPA 7196A
Hexavalent Chromium	SCM	NJ Modified 3060A
Ignitability / Flashpoint	NPW	EPA 1010A
Ignitability/Flashpoint	SCM	EPA 1010A
Indeno(1,2,3-cd)pyrene	NPW	EPA 8270C/D; EPA 625
Indeno(1,2,3-cd)pyrene	SCM	EPA 8270C/D
Iodomethane	NPW	EPA 8260B; EPA 624
Iodomethane	SCM	EPA 8260B
Iron	NPW	EPA 6010B/C; EPA 200.7
Iron	SCM	EPA 6010B/C
Isobutyl alcohol	NPW	EPA 8260B; EPA 624
Isobutyl alcohol	SCM	EPA 8260B
Isophorone	NPW	EPA 8270C/D; EPA 625
Isophorone	SCM	EPA 8270C/D
Isopropylbenzene (Cumene)	NPW	EPA 8260B; EPA 624
Isopropylbenzene (Cumene)	SCM	EPA 8260B
Lead	NPW	EPA 6010B/C; EPA 200.7
Lead	SCM	EPA 6010B/C
m,p-Xylenes	NPW	EPA 8260B; EPA 624
m,p-Xylenes	SCM	EPA 8260B
Magnesium	NPW	EPA 6010B/C; EPA 200.7
Magnesium	SCM	EPA 6010B/C
Manganese	NPW	EPA 6010B/C; EPA 200.7
Manganese	SCM	EPA 6010B/C
MCPA	NPW	EPA 8151A
MCPP (Mecoprop)	NPW	EPA 8151A
Mercury	NPW	EPA 7470A; EPA 245.1
Mercury	SCM	EPA 7471A/B
Metals digestion	NPW	EPA 3005A
Metals digestion	NPW	EPA 3010A
Metals Digestion	SCM	EPA 3050B
Methacrylonitrile	NPW	EPA 8260B; EPA 624
Methacrylonitrile	SCM	EPA 8260B
Methane	NPW	RSK-175
Methoxychlor	NPW	EPA 8081A/B
Methoxychlor	SCM	EPA 8081A/B
Methyl Acetate	NPW	EPA 8260B; EPA 624
Methyl Acetate	SCM	EPA 8260B
Methyl methacrylate	NPW	EPA 8260B; EPA 624
Methyl methacrylate	SCM	EPA 8260B
Methyl Tertiary Butyl Ether (MTBE)	NPW	EPA 8260B; EPA 624
Methyl Tertiary Butyl Ether (MTBE)	SCM	EPA 8260B
Methylcyclohexane	NPW	EPA 8260B; EPA 624
Methylcyclohexane	SCM	EPA 8260B
Methylene Chloride, or Dichloromethane	NPW	EPA 8260B; EPA 624
Methylene Chloride, or Dichloromethane	SCM	EPA 8260B
Molybdenum	NPW	EPA 6010B/C; EPA 200.7
Molybdenum	SCM	EPA 6010B/C
Naphthalene	NPW	EPA 8260B; EPA 624
Naphthalene	NPW	EPA 8270C/D; EPA 625
Naphthalene	SCM	EPA 8260B
Naphthalene	SCM	EPA 8270C/D
n-Butylbenzene	NPW	EPA 8260B; EPA 624
n-Butylbenzene	SCM	EPA 8260B
Nickel	NPW	EPA 6010B/C; EPA 200.7
Nickel	SCM	EPA 6010B/C
Nitrate	NPW	EPA 300.0
Nitrate	NPW	EPA 9056A

Table 7.2 DOD ELAP Parameter (QSM4.2 or QSM5.0)	Matrix Code	Approved Method
Nitrate	SCM	EPA 9056A
Nitrate/Nitrite	NPW	EPA 353.2
Nitrite	NPW	EPA 300.0
Nitrite	NPW	EPA 9056A
Nitrite	SCM	EPA 9056A
Nitrite as N	NPW	SM 4500-NO2 B-2011
Nitrobenzene	NPW	EPA 8270C/D; EPA 625
Nitrobenzene	NPW	EPA 8330A/B
Nitrobenzene	SCM	EPA 8270C/D
Nitrobenzene	SCM	EPA 8330A
Nitrobenzene	SCM	EPA 8330B
Nitrocellulose	NPW	EPA 353.2 MOD
Nitrocellulose	SCM	EPA 353.2 MOD
Nitroglycerin	NPW	EPA 8330A/B
Nitroglycerin	SCM	EPA 8330A
Nitroglycerin	SCM	EPA 8330B
Nitroguanidine	NPW	EPA 8330A/B
Nitroguanidine	SCM	EPA 8330A
Nitroguanidine	SCM	EPA 8330B
N-Nitrosodimethylamine	NPW	EPA 8270C/D; EPA 625
N-Nitrosodimethylamine	SCM	EPA 8270C/D
N-Nitroso-di-n-propylamine (NDPA)	NPW	EPA 8270C/D; EPA 625
N-Nitroso-di-n-propylamine (NDPA)	SCM	EPA 8270C/D
N-nitrosodiphenylamine (NDPHA)	NPW	EPA 8270C/D; EPA 625
N-nitrosodiphenylamine (NDPHA)	SCM	EPA 8270C/D
n-Propylbenzene	NPW	EPA 8260B; EPA 624
n-Propylbenzene	SCM	EPA 8260B
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	NPW	EPA 8330A/B
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	SCM	EPA 8330A
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	SCM	EPA 8330B
Oil and Grease	NPW	EPA 1664A
Organics Liquid Extraction	NPW	EPA 3510C
Organics Microwave Extraction	SCM	EPA 3546
Organics Sonication	SCM	EPA 3550B/C
Ortho-Phosphate (as P)	NPW	SM 4500-P E-2011
o-Xylene	NPW	EPA 8260B; EPA 624
o-Xylene	SCM	EPA 8260B
Paint Filter	NPW	EPA 9095B
Pentachlorophenol	NPW	EPA 8270C/D; EPA 625
Pentachlorophenol	SCM	EPA 8270C/D
Percent Solids (Percent Moisture)	SCM	SM 2540 B-1997
Perchlorate	NPW	EPA 6850
Perchlorate	SCM	EPA 6850
PETN	NPW	EPA 8330A/B
PETN	SCM	EPA 8330A
PETN	SCM	EPA 8330B
Petroleum Range Organics	NPW	FLPRO
Petroleum Range Organics	SCM	FLPRO
pH (Corrosivity)	SCM	EPA 9045C/D
pH(Corrosivity)	NPW	EPA 9040B/C, SM 4500-H+ B-2011
Phenanthrene	NPW	EPA 8270C/D; EPA 625
Phenanthrene	SCM	EPA 8270C/D
Phenol	NPW	EPA 8270C/D; EPA 625
Phenol	SCM	EPA 8270C/D
p-Isopropyltoluene	NPW	EPA 8260B; EPA 624
p-Isopropyltoluene	SCM	EPA 8260B
Potassium	NPW	EPA 6010B/C; EPA 200.7
Potassium	SCM	EPA 6010B/C
Propionitrile	NPW	EPA 8260B; EPA 624
Propionitrile	SCM	EPA 8260B
Purge and Trap Solid	SCM	EPA 5035 /A
Purge and Trap Water	NPW	EPA 5030A/B
Pyrene	NPW	EPA 8270C/D; EPA 625
Pyrene	SCM	EPA 8270C/D
Pyridine	NPW	EPA 8270C/D; EPA 625
Pyridine	SCM	EPA 8270C/D
Reactive Sulfide	NPW	Chap.7, Sect. 7.3.4 Mod.
Reactive Sulfide	SCM	Chap.7, Sect. 7.3.4 Mod.
sec-Butylbenzene	NPW	EPA 8260B; EPA 624
sec-Butylbenzene	SCM	EPA 8260B
Selenium	NPW	EPA 6010B/C; EPA 200.7
Selenium	SCM	EPA 6010B/C
Silver	NPW	EPA 6010B/C; EPA 200.7
Silver	SCM	EPA 6010B/C
Sodium	NPW	EPA 6010B/C; EPA 200.7
Sodium	SCM	EPA 6010B/C
SPLP	SCM	EPA 1312

Table 7.2 DOD ELAP Parameter (QSM4.2 or QSM5.0)	Matrix Code	Approved Method
Strontium	NPW	EPA 6010B/C; EPA 200.7
Strontium	SCM	EPA 6010B/C
Styrene	NPW	EPA 8260B; EPA 624
Styrene	SCM	EPA 8260B
Sulfate	NPW	EPA 300.0
Sulfate	NPW	EPA 9056A
Sulfate	SCM	EPA 9056A
Sulfide	NPW	SM 4500-S2 F-2011
t-Butyl alcohol	NPW	EPA 8260B; EPA 624
TCLP	NPW	EPA 1311
TCLP	SCM	EPA 1311
TDS	NPW	SM 2540 C-2011
tert-Amyl methyl ether	NPW	EPA 8260B; EPA 624
tert-Amyl methyl ether	SCM	EPA 8260B
tert-Butyl alcohol	SCM	EPA 8260B
tert-Butylbenzene	NPW	EPA 8260B; EPA 624
tert-Butylbenzene	SCM	EPA 8260B
Tetrachloroethene (PCE; PERC)	NPW	EPA 8260B; EPA 624
Tetrachloroethene (PCE; PERC)	SCM	EPA 8260B
Tetrahydrofuran	NPW	EPA 8260B; EPA 624
Tetrahydrofuran	SCM	EPA 8260B
Thallium	NPW	EPA 6010B/C; EPA 200.7
Thallium	SCM	EPA 6010B/C
Tin	NPW	EPA 6010B/C; EPA 200.7
Tin	SCM	EPA 6010B/C
Titanium	NPW	EPA 6010B/C; EPA 200.7
Titanium	SCM	EPA 6010B/C
Toluene	NPW	EPA 8260B; EPA 624
Toluene	SCM	EPA 8260B
Total Organic Carbon	NPW	EPA 9060A; SM 5310 C-2011
Total Organic Carbon	SCM	Lloyd Kahn
Total Phosphorus (as P)	NPW	SM 4500-P B5-2011
Toxaphene	NPW	EPA 8081A/B
Toxaphene	SCM	EPA 8081A/B
TPH DRO	NPW	EPA 8015B/C
TPH DRO	SCM	EPA 8015B/C
TPH GRO	NPW	EPA 8015B/C
TPH GRO	SCM	EPA 8015B/C
TPH ORO	NPW	EPA 8015B/C
TPH ORO	SCM	EPA 8015B/C
trans-1,2-Dichloroethene (trans-1,2-DCE)	NPW	EPA 8260B; EPA 624
trans-1,2-Dichloroethene (trans-1,2-DCE)	SCM	EPA 8260B
trans-1,3-Dichloropropene	NPW	EPA 8260B; EPA 624
trans-1,3-Dichloropropene	SCM	EPA 8260B
trans-1,4-Dichloro-2-butene	NPW	EPA 8260B; EPA 624
trans-1,4-Dichloro-2-butene	SCM	EPA 8260B
Trichloroethene (TCE)	NPW	EPA 8260B; EPA 624
Trichloroethene (TCE)	SCM	EPA 8260B
Trichlorofluoromethane (CFC-11)	NPW	EPA 8260B; EPA 624
Trichlorofluoromethane (CFC-11)	SCM	EPA 8260B
TSS	NPW	SM 2540 D-2011
Vanadium	NPW	EPA 6010B/C; EPA 200.7
Vanadium	SCM	EPA 6010B/C
Vinyl acetate	NPW	EPA 8260B; EPA 624
Vinyl acetate	SCM	EPA 8260B
Vinyl Chloride (VC)	NPW	EPA 8260B; EPA 624
Vinyl Chloride (VC)	SCM	EPA 8260B
Xylenes (Total)	NPW	EPA 8260B; EPA 624
Xylenes (Total)	SCM	EPA 8260B
Zinc	NPW	EPA 6010B/C; EPA 200.7
Zinc	SCM	EPA 6010B/C

Appendix C-2
TestAmerica Sacramento
Quality Assurance Manual

Quality Assurance Manual Cover Page

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Title Page:

Quality Assurance Manual

Approval Signatures

Laboratory Director – Crystal Pollock	5/19/2017 Date
Quality Manager – Lisa Stafford	May 18, 2017 Date
Technical Manager, Dioxins, LCMS, Inorganics Robert Hrabak	5/18/17 Date
Manager, Volatiles, Semivolatiles, Organic/Dioxin Prep Koroush Vaziri	5/18/17 Date

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CW-I-P-007	Computer Systems Password Policy
CA-L-P-002	Contract Compliance Policy
CA-Q-M-002	Corporate Quality Management Plan
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-006	Detection Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Corporate Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation s
CW-L-S-004	Subcontracting
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recalls
CA-C-S-001	Work Sharing Process

REFERENCED LABORATORY SOPs

SOP Reference	Title
WS-PQA-013	Procedures to Address Customer Complaints
WS-QA-0050	Management of Change
WS-QA-0009	Document Archiving
WS-QA-0022	Employee Orientation and Training
WS-QA-0021	Preparation and Management of Standard Operating Procedures
WS-QA-0006	Method Detection Limits (MDL) and Instrument Detection Limits (IDL)
WS-PQA-0011	Manual Integration Documentation Procedures
WS-PQA-003	Quality Control Program
WS-QA-0018	Subsampling and Compositing of Samples
WS-QA-0003	Sample Receipt and Procedures
WS-QA-0035	Statistical Process Control / Control Chart
WS-EHS-0001	Waste Disposal

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Sacramento's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*
- *U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.*
- *U.S. Department of Denfense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013.*
- *U.S. Department of Denfense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.1, 2017.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Statement of Work for Inorganics & Organics Analysis, SOM, ISM, DLF and CBC, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, and on-line Editions.
- *U.S. Department of Energy Order 414.1B, Quality Assurance, Approved April 29, 2004.*
- *U.S. Department of Energy Order 414.1C, Quality Assurance, June 17, 2005.*

- *U.S. Department of Energy Order 414.1D, Quality Assurance, April 25, 2011*
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge, tissue and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & Updating procedures (refer to SOP No. WS-QA-0021).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Sacramento is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., Chief Executive Officer, Executive VP Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Sacramento is presented in Figure 4-1.

4.2 Roles and Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Sacramento laboratory.

4.2.2 President and Chief Executive Officer (CEO)

The President and CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. The President and CEO establishes the overall quality standard and data integrity program for the Analytical Business, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.3 Senior Vice President of Operations (SVPO)

The COO reports directly to the President and CEO of TestAmerica. The SVPO~~VPO~~ oversees the operations of all TestAmerica. The VP's of Operations report directly to the SVPO.

4.2.4 Vice President of Operations

Each VP of Operations reports directly to the Senior VP of Operations and is a part of the Executive Committee. Each VP of Operations is responsible for the overall administrative and operational management of their respective laboratories. The VP's responsibilities include allocation of personnel and resources, long-term planning, goal setting, and achieving the financial, business, and quality objectives of TestAmerica. The VP's ensure timely compliance with Corporate Management directives, policies, and management systems reviews. The VP's are also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

4.2.5 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to the President and CEO. With the aid of the Executive Committee, Laboratory Directors, Quality Directors, Safety Manager, EH&S Coordinators and QA Managers, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and EH&S Programs within TestAmerica. Additional responsibilities include:

- Review of QA/QC and EHS aspects of Corporate SOPs & Policies, national projects and expansions or changes in services.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the analytical laboratories and a summary of any quality related initiatives and issues.
- Preparation of a monthly report that includes EH&S metrics across the analytical laboratories and a summary of any EH&S related initiatives and issues.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

4.2.6 Vice President of Client Service

The VP of Client Services leads the Client Service Organization (CSO) and is responsible for client satisfaction, driving operational excellence and improving client responsiveness. The VP provides direction to the Client Service Directors, Programs Managers and Project Managers.

4.2.7 Quality Assessment Director

The Quality Assessment Director reports to the VP-QA/EHS. The Quality Assessment Director has QA oversight of laboratories; responsible for the internal audit system, schedule and procedure; monitors laboratory internal audit findings; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Compliance Director, the Quality Systems Director, and the VP-QA/EHS, the Quality Assessment Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.8 Quality Compliance Director

The Quality Compliance Director reports to the VP-QA/EHS. The Quality Compliance Director has QA oversight of laboratories; monitors and communicates DoD / DoE requirements; develops corporate tools for ensuring and improving compliance; develops corporate assessment tools; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Systems Director and the VP-QA/EHS, the Quality Compliance Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.9 Quality Systems Director

The Quality Systems Director reports to the VP-QA/EHS. The Quality Systems Director has QA oversight of laboratories; develops quality policies, procedures and management tools; monitors and communicates regulatory and certification requirements; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Compliance Director and the VP-QA/EHS, the Quality Systems Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.10 Quality Information Manager

The Quality Information Manager is responsible for managing all company official documents (e.g., Policies, Procedures, Work Instructions), the company's accreditation database, intranet websites, external laboratory subcontracting, regulatory limits for clients on the company's TotalAccess website; internal and external client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions. The Quality Information Manager reports to the VP-QA/EHS; and works alongside the Quality Assessment, Quality Compliance and Quality System Directors and EHS Managers to support both the Analytical Quality Assurance and EHS Programs within TestAmerica.

4.2.11 Technical Services Director

The Technical Services Director is responsible for establishing, implementing and communicating TestAmerica's Analytical Business's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

4.2.12 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – Corporate Counsel & VP of Human Resources and the VP-QA/EHS. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President and CEO, VPOs, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.13 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

4.2.14 Environmental Health and Safety Managers (Corporate)

The EHS Managers report directly to the VP-QA/EHS. The EHS Managers are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.15 Laboratory Director

TestAmerica Sacramento's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the laboratory and reports to their respective VP of Operations. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Providing one or more technical managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence

exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.

- Ensuring that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensuring that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensuring TestAmerica's human resource policies are adhered to and maintained.
- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensuring that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits.
- Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviewing and approving all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursuing and maintaining appropriate laboratory certification and contract approvals.
- Supporting ISO 17025 requirements.
- Supporting DoD/DOE ELAP requirements.
- Supporting The NELAC Institute (TNI) Standard requirements
- Ensuring client specific reporting and quality control requirements are met.
- Directing the management team, consisting of the QA Manager, the Operations Manager, the EH&S Coordinator and the Office Manager as direct reports.

4.2.16 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system. The QA Manager reports directly to the Laboratory Director and their Corporate Quality Director. This person is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. This person has documented training and/or experience in QA/QC procedures and the laboratory's Quality System. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Serving as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.

- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintaining records of all ethics-related training, including the type and proof of attendance.
- Maintaining, improving, and evaluating the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Reviewing external audit reports and data validation requests.
- Following-up with audits to ensure client QAPP requirements are met.
- Establishing reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Developing suggestions and recommendations to improve quality systems.
- Researching current state and federal requirements and guidelines.
- Directing the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring communication with laboratory staff and monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluating of the thoroughness and effectiveness of training.
- Assuring compliance with ISO 17025.
- Assuring compliance with DoD/DOE ELAP.
- Assuring compliance with The NELAC Institute (TNI) Standard.

4.2.17 Technical Manager (Manager of Operations) or Designee

The Technical Manager(s) (noted as Manager of Operations on the organizational chart) report(s) directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i.e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design versus demonstrated versus first run yield) utilization.

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.

- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.

- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.

- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.

- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.

- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.

- Directing department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.

- Complying with ISO 17025, The NELAC Institute (TNI) Standard, DoD/DOE ELAP and the various QC programs implemented at the Sacramento laboratory.

4.2.18 Client Services Manager

The CSM reports directly to the Client Service Director (Western Region) and indirectly to the Laboratory Director. The CSM serves as the interface between the laboratory's Project Management team, technical departments, and clients. The CSM shall:

- Oversee training and growth of the Project Management team.
- Act as technical liaison for the Project Management team.
- Provide human resource management support to the Project Management team.
- Assist PMs with responses to client inquiries or with resolutions to problems or complaints.
- Ensure that client specifications, when known, are met by communicating project and QA requirements to the laboratory.
- Notify Department Managers or supervisors of incoming projects and sample delivery schedules.
- Discuss with client any project-related problems, resolve service issues, and coordinate technical details with the laboratory staff.
- Monitor the status of projects in-house to ensure timely and accurate delivery of reports.
- Prepare price quotes or project bids.

4.2.19 Manager of Project Managers

The Manager of Project Management reports to the Regional Client Services Director and serves as the interface between the laboratory's technical departments and the laboratory's clients. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the duties of this position are outlined below:

- Managing technical training and growth of the Project Management team
- Serving as technical liaison for the Project Management team
- Providing human resource management of the Project Management team
- Ensuring that clients receive the proper sampling supplies
- Overseeing response to client inquiries concerning sample status
- Assisting clients regarding the resolution of problems concerning COC
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory
- Notifying the supervisors of incoming projects and sample delivery schedules
- Being accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates

- Discussing with clients any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff
- Providing information to staff with respect to specific quotes, sample log-in review, and final report completeness
- Monitoring the status of all data package projects in-house to ensure timely and accurate delivery of reports
- Informing clients of data package-related problems and resolve service issues
- Coordinating requests for sample containers and other services (data packages)

4.2.20 Project Manager

Project Managers are a liaison between the laboratory's clients and the analytical staff. They report directly to the Manager of Program Management. The Project Managers have signature authority for final reports, and review project data packages for completeness and compliance with client needs and quality requirements.
The Project Manager's responsibilities include:

- Ensuring client specifications are met by communicating project and quality assurance requirements to the laboratory
- Notifying laboratory personnel of incoming projects and sample delivery schedules
- Monitoring the status of all projects in-house to ensure timely delivery of reports
- Informing clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff
- Coordinating client requests for sample containers and other services
- Scheduling sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples
- Coordinating subcontract work
- Assisting clients in procuring the proper sampling supplies
- Responding to client inquiries concerning sample status
- Assisting clients with resolution of problems concerning Chains-of-Custody
- Invoicing completed data packages
- Generating credit or debit invoices to ensure proper payment

4.2.21 Project Administrator

The Project Administrator reports to the Manager of Project Management and designated Project Manager. The Project Administrator assists the Project Manager in servicing the client's needs and communicating those needs to the laboratory. The Project Administrator's responsibilities include:

- Collating data reports, expanded deliverables, and electronic data deliverables (EDDs) for delivery to clients.
- Writing case narratives accompanying data packages to communicate anomalies to clients
- Coordinating client requests for sample containers and other services
- Assisting clients in procuring the proper sampling supplies
- Assisting Project Managers in changing compound lists, TAT, and other LIMS set up tasks.
- Monitoring report due dates for timely delivery
- Invoicing completed data packages
- Generating credit or debit invoices to ensure proper payment

4.2.22 Department Manager, Team Leader, or Supervisor

Department Managers report directly to the Operations Manager. They supervise the daily activities of analysis within a given laboratory area, and either oversee the review and approval, or perform the review and approval of all analytical data within that area.

Specific responsibilities include, but are not limited to:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results.
- Ensuring that analysts in their department adhere to applicable SOPs and the QA Manual.
- Coordinating the writing and reviewing of documentation for all test methods, i.e., SOPs, with regard to quality, integrity, regulatory requirements and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity includes insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances, improved LIMS utilization, capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Coordinating audit responses with the QA Manager.

- Complying with ISO 17025, The NELAC Institute (TNI) Standard, DoD/DOE QSM and the various QC programs implemented at the Sacramento laboratory.
- Participating in the selection, training (familiarization with SOP, QC, Safety and computer systems), developing performance objectives and standards of performance, appraising (measurement of objectives), scheduling, counseling, disciplining, and motivating analysts and documenting these activities in accordance with systems developed by the QA and Human Resources Departments.
- Evaluating staffing sufficiency and overtime needs.
- Encouraging the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Providing guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and corrective actions, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.
- Ensuring all logbooks are maintained, current, reviewed, and properly labeled or archived.
- Reporting all non-conformance conditions to the QA Manager, Operations Manager, and/or Laboratory Director.
- Ensuring that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/She has responsibility for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintaining adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieving optimum turnaround time on analyses and compliance with holding times.
- Conducting efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long term needs for budgetary planning.

4.2.23 Analyst

Analysts report to their respective Department Managers. They perform sample analyses and generate analytical data in accordance with documented procedures.

The responsibilities of the analysts are listed below:

- Collecting and preparing materials and supplies for the laboratory
- Retrieving samples from Sample Control for analysis

- Performing sample preparation by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Documenting standard and sample preparation, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Reporting all non-conformance situations, sample preparation problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Performing 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggesting method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Providing ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Working cohesively as a team member in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.24 Sample Custodian

The Sample Custodian ensures the implementation of proper sample receipt procedures, including maintaining chain-of-custody. The Sample Custodian logs samples into the LIMS and ensures that all samples are stored appropriately. Duties for the Sample Custodian include the following:

- Receiving and unloading samples or consignments in accordance with DOT regulations
- Verifying samples against the Chain of Custody (COC)
- Logging samples into the LIMS to assign a lot number for tracking purposes, and notifying Project Managers of any irregularities with the sample shipment.
- Labeling samples with lot number assigned and deliver the samples to the appropriate labs for analysis daily
- Monitoring freezer and cooler temperatures daily to confirm that the readings are within SOP guidelines
- Shipping all subcontracted samples to designated lab in accordance with DOT regulations as needed.

4.2.25 Quality Assurance Staff

The Quality Assurance staff members report to the QA manager. They have responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025, through involvement in the following activities:

- Assisting the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- Facilitating external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.
- Assisting the QA Manager in the preparation of new SOPs and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- Managing the performance testing (PT) studies, coordinating follow-up studies for failed analytes, and working with QA Manager and Laboratory Staff to complete needed corrective action reports.
- Serving as a project manager for proficiency testing samples and other QC samples.
- Reviewing and maintaining personnel training records.
- Assisting the QA Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- Managing certifications and accreditations.
- Monitoring for compliance with the following QA Metrics: Temperature Monitoring of refrigeration units; thermometer verifications and calibrations; balance verifications and callibrations; and Eppendorf/pipette calibrations.
- Periodically checking the proper use and review of logbooks.
- Assisting in the technical review of data packages which require QA review.
- Assisting the QA Manager in maintaining the laboratory's reference data to keep it current and accurate.
- Preparing certification applications for states as directed by QA Manager.
- Reviewing and maintaining personnel training records.
- Performing document control maintenance.
- Assisting departments in generating MDL spreadsheets and calculations, reviewing MDL studies submitted to QA.
- Assisting in control limit generation.
- Ensuring maintenance of records archives.

- Maintaining historical indices for all technical records including SOPs, QC records, laboratory data, etc.
- Assisting the QA Manager in meeting the responsibilities of the QA Department as described in laboratory policies and SOPs.

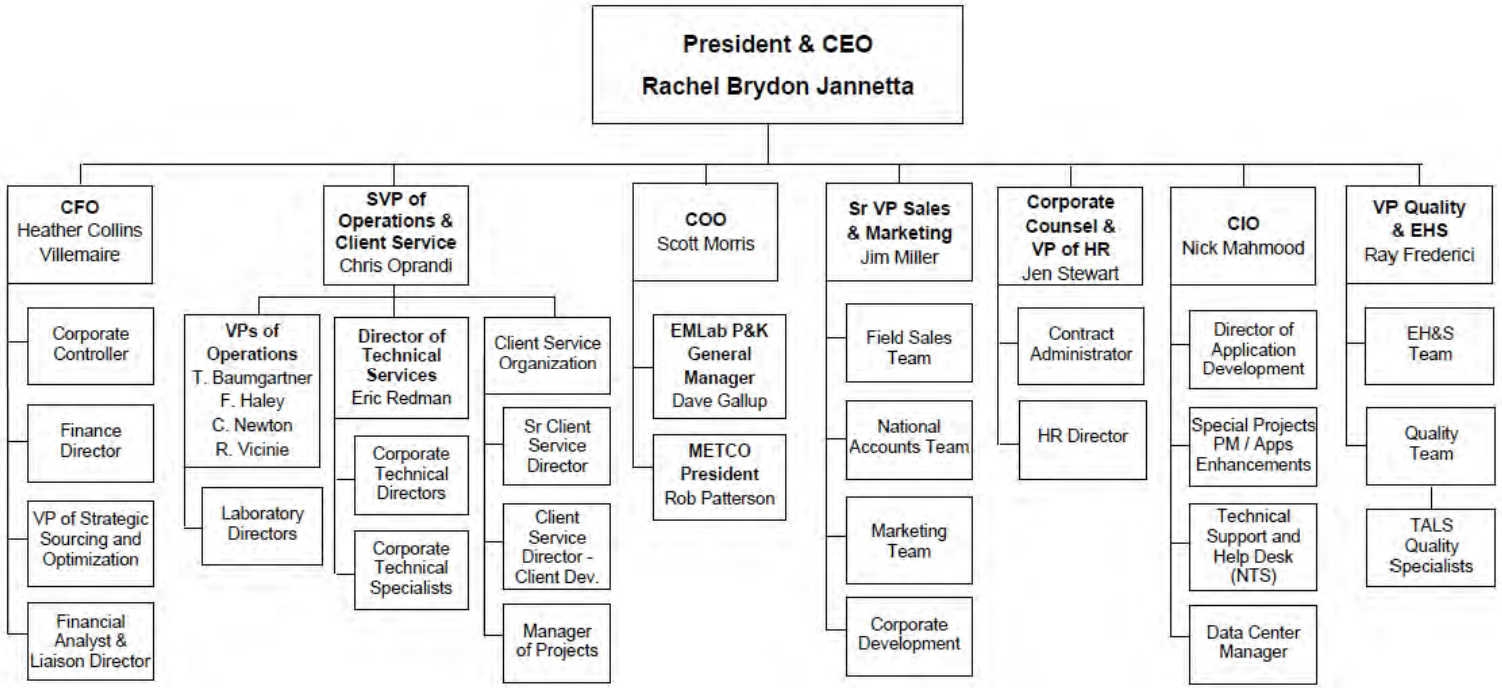
4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Crystal Pollock - Laboratory Director	Robert Hrabak - Technical Director, Manager of Dioxins, LCMS & Inorganics
Lisa Stafford - Quality Assurance Manager	Russell Evans - Quality Assurance Staff Crystal Pollock - Laboratory Director
Robert Hrabak - Technical Director, Manager of Dioxins, LCMS & Inorganics	Crystal Pollock - Laboratory Director
Koroush Vaziri – Manager of Volatiles, Semivolatiles, & Organic/Dioxin Prep	Robert Hrabak - Technical Director, Manager of Dioxins, LCMS & Inorganics Crystal Pollock - Laboratory Director
Jill Kellmann - Manager of Project Management	David Herbert - Client Relations Manager (Corporate)
Joe Schairer - EHS Coordinator	Richard Kester - Hazardous Materials Specialist

Figure 4-1. Corporate and Laboratory Organization Charts

All organizational charts are current as of the date noted. Contact the laboratory for the most recent organizational chart.



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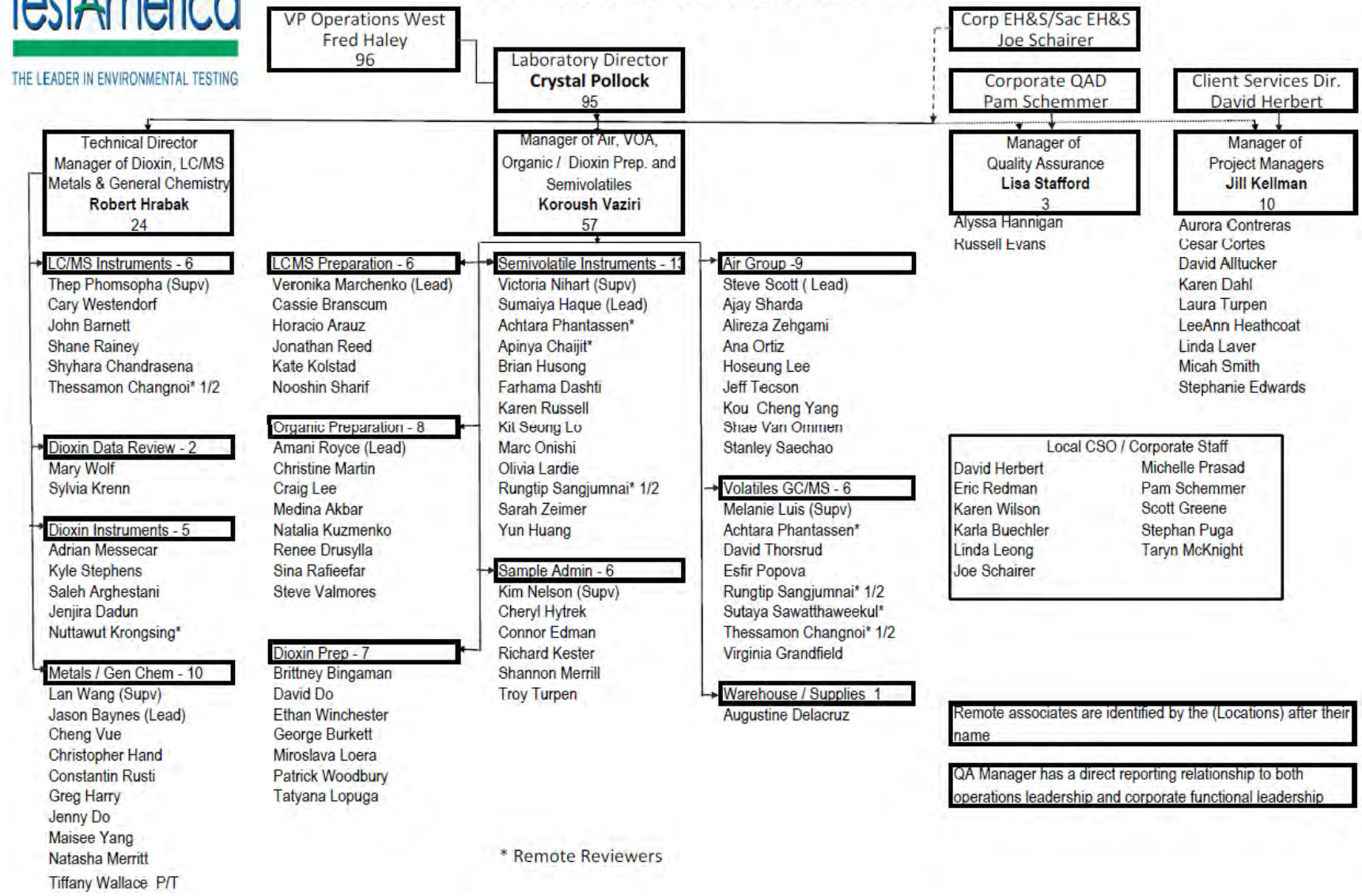
Sacramento Laboratory Organization



SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.
- Comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 Ethics and Data Integrity

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct (Corporate SOP No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-L-S-002).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.

- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterizes the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), inter-element corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 Criteria for Quality Indicators

The laboratory maintains Reference Data in the LIMS that summarizes the precision and accuracy acceptability limits for performed analyses. This data includes an effective date, is updated each time new limits are generated and is managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. The criteria for development of control limits is contained in SOP WS-QA-0035, "Statistical Process Control / Control Chart" and Section 24.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Manager and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in SOP WS-QA-0035, "Statistical Process Control / Control Chart" and Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. Control charts are generated according to laboratory SOP No. SOP WS-QA-0035, "Statistical Process Control / Control Chart"

5.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

6.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled

documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years (annually for documents applicable to drinking water and DoD/DOE programs), and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the QA share on the local server for the applicable revision, and are accessible using the laboratory's Intranet.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP and SOP No. WS-QA-0021, Preparation and Management of Standard Operating Procedures". The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by department in the QA office. There is a table of contents. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. WS-QA-0021, Preparation and Management of Standard Operating Procedures.

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. The PM will also get approval by the Laboratory Director to commit to delivery schedules that are shorter than the published standard turnaround times (TATs). The Laboratory Director updates these TATs on a routine basis, and it is the responsibility of CSMs and PMs to review them prior to making commitments for the laboratory.

It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Contract Administrator
- VP of Operations
- Client Relations Manager
- Laboratory Project Manager
- Laboratory and/or Corporate Technical Managers / Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Account Executives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contracts Administrator, Account Executive, or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Contracts Department maintains copies of all signed contracts. TestAmerica Sacramento's Customer Service Organization maintains copies of all signed contracts on the computer network for reference locally.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. These records are archived by client and project in a restricted network folder accessible to laboratory department managers, project managers, and senior managers.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. Each Laboratory Project Manager keeps a phone log of conversations with the client. In addition, all conversations involving notification of important information, or actions directed by the client are documented with a follow up e-mail and archived in the contracts folder or the SDG documentation and case narrative. Instances include change in scope, alterations to the requests listed on a chain of custody, directions to proceed in the event of a non-conformance, and any other conversation that changes the direction of a COC or contract.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, a PM is assigned to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements. Quality Assurance Project Plans, if submitted by the client, will be evaluated per policy WS-PQA-0018.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are updated to the Quality Assurance Summary (QAS) and introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO/IEC 17025 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Allow the client access to supplemental information that pertains to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 Client Communication

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Any member of the laboratory's senior staff or any of the laboratory's identified technical experts is available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOPs on Subcontracting Procedures (CW-L-S-004).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

Project Managers (PMs), Client Service Managers (CSM), or Account Executives (AE) for the Export Lab (TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder. Standard TestAmerica Terms and Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., DoD and DOE projects) may require notification prior to placing such work. Documentation of approval is stored electronically in the quote folder within SACSALES share on a local laboratory server.

8.2 Qualifying and Monitoring Subcontractors

Whenever a PM or Client Services Manager becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.

- Subcontractors reviewed by TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the TestAmerica intranet site.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

8.2.1 When the potential sub-contract laboratory has not been previously approved, PMs or CSMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM) or Laboratory Director. The CRM or Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CW-L-S-004, Subcontracting.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. After the Corporate QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified for JD Edwards.

The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractors on our approved list can only be recommended to the extent that we would use them.

8.3 Oversight and Reporting

8.3.1 The status and performance of qualified subcontractors will be monitored by the Corporate Quality department. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance, Legal and Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. CSO personnel will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any

laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all CSO Personnel, Laboratory Directors, QA Managers and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is stored electronically in the quote folder within the SACSALES share on a local laboratory server.

8.3.2 For continued use of a subcontractor, verification of certification is placed upon the subcontractor for the defined project. Samples are subcontracted under Chain of Custody with the program defined as 'Accreditation Required' and the following statement for verification upon sample receipt:

Note: Since laboratory accreditations are subject to change, TestAmerica Laboratories, Inc. places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under Chain of Custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analytes/tests/matrix being analyzed, the samples must be shipped back to the TestAmerica laboratory or other instructions will be provided. Any changes to accreditation status should be brought to TestAmerica Laboratories, Inc. attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to TestAmerica Laboratories, Inc.

For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

8.3.3 All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for all samples workshared within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time.

The use of any emergency subcontractor will require the PM to complete a JDE New Vendor Add Form in order to process payment to the vendor and add them to TALS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Controlled Purchase Requests and Fixed Asset Capitalization Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001. Approval information for the solvents and acids tested under SOP CA-Q-S-001 is stored on the TestAmerica Sharepoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. Many items used routinely are pre-qualified and placed into the on-site consignment system.

For items not available from the consignment system or items that are not used routinely, an order is placed in the JDE ordering system. Only personnel trained in the ordering program JDE may place orders using the program. All relevant information, including quantity, must be entered. Only approved vendors may be used. A vendor must be approved by corporate to be on the approved vendor list in JDE. The Laboratory Director or designee approves all orders placed in JDE.

9.3.2 Receiving

It is the responsibility of the purchasing manager to receive the shipment. For items received for the on-site consignment system, the purchasing manager verifies that the material received meets the quality level specified. This is documented by stamping the packing slip with "Received" and the date. For materials that are outside of the on-site consignment systems, it is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The purchasing manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared "public" folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of the grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals should not be used past the manufacturer's or SOP's expiration date unless 'verified' (refer to bullet 3 below). See laboratory SOP No. WS-QA-0017, "Standards and Reagent Preparation and Quality Control Check Procedures", for standard verification procedures.)

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained on the shared public folder on the computer network.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure would be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. For the automated “tank farm” in use through most of the laboratory, the minimum total pressure at which the system switches to the next bank of tanks is 250 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water’s specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified “clean” by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard. See laboratory SOP No. WS-QA-0017, “Standards and Reagent Preparation and Quality Control Check Procedures”, for standard QC procedures.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Each laboratory section maintains records of manufacturer’s certification and traceability statements on the network. These records include date of receipt, lot number (when

applicable), and expiration date (when applicable). Furthermore, certificates of analysis for standards are scanned and attached to the preparation record in the LIMS. Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager or QA Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench and inventoried in the master document list.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP WS-QA-0041, Calibration and Calibration Check of Balances. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed, and documentation of the review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc, are obtained from vendors with current and valid ISO 17025 accreditation for calibration of

the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory.

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following laboratory policy WS-PQA-013, Procedure to Address Customer Complaints.

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to laboratory policy WS-PQA-013, Procedure to Address Customer Complaints.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Manager and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

11.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the

Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Exec. Director of Quality & EHS and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, Executive VP of Operations, VP of Operations, and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.
When the laboratory discovers that erroneous or biased data may have been reported to clients or regulatory agencies, the procedures described in the corporate SOP CW-Q-S-005, Data Recalls, must be followed.
During investigation and correction of situations involving alleged incidents of misconduct or violation of the company's ethics policy, the procedures described in the corporate SOP CW-L-S-002, Internal Investigations, must be followed.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

11.4 Prevention of NonConforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically, on a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that

suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate VP of Operations and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc…). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Manager/Director, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memos (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints

- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports
- Health and Safety violations

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Laboratory SOP No. WS-QA-0023, Nonconformance and Corrective Action System, provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or CAR is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with the problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and CAR is entered into a database for tracking purposes and these are periodically reviewed to ensure that the corrective actions have taken effect.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. (Previously, a local database [name of local system here] served this purpose.) An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. iCAT allows associated document upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis. Refer to Figure 12-1.
- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.

- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The SOP also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 12-1.
Example - Corrective Action Report

TestAmerica
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Sacramento
Corrective Action Report

Title: <Enter Title Here>

Reference: <tracking information>

Initiated by: **Date:**

Responsible Party: **Date:** <date report submitted>

Description of Problem:
[enter text to briefly explain how the problem was discovered, who discovered it and when, and what work, if any, is affected]

Investigation Planned or Completed:
[enter text to briefly what was examined to determine the extent of the problem, when the investigation was conducted, what was the proximate cause(s), and what were the root causes. The key is to demonstrate that the investigation is comprehensive]

Root Cause Analysis
[True root cause analysis should involve multiple layers of questioning]
Examples:
- *Why did this problem occur?*
- *What weaknesses are indicated by this problem?*
- *What Quality Systems mechanisms are in place that should have prevented this problem from occurring?*
- *Is this issue acute or chronic?*
- *Are changes needed to existing SOPs to correct this problem and prevent its recurrence?*
- *Are other departments affected by this issue?*

Corrective Action Plan
[Based on the Root Cause Analysis outlined above, what action items need to be completed to correct this deficiency, and prevent its recurrence?]
Examples:
- *Identify impacted lots*
- *Revise results/reports*
- *Initiate formal Data Recall*
- *Revise SOP*
- *Re-train staff*

QA Monitoring of Corrective Action Status
[If an anomalous or isolated event, and no further action required, this section may be omitted. Otherwise, note the need for a routine follow-up assessment and the associated details (responsible party, due date, documentation necessary), or the need to add to the internal audit checklist for reassessment at a later date.

Closed by: _____ _____
<Name, title> Date

Table 12-1. Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	- Instrument response <MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards *(Analyst, Technical Manager(s))*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Technical Manager(s))*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in the LIMS or Project QAPP.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in the LIMS or Project QAPP.	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples *(QA Manager, Technical Manager(s))*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Technical Manager(s), Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002.
Client Complaints *(Project Managers, Lab Director/Manager, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director/Manager, *Technical Manager(s)*)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director/Manager, *Technical Manager(s)*)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:
1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants as defined in policy WS-PQA-003 **provided** they appear at similar levels in the reagent blank and samples. The ubiquitous contaminants include: methylene chloride, toluene, acetone, 2-butanone, phthalates and octachlorodibenzodioxin. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by the laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used ~~to~~ in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lesson Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.
The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/Process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any Preventive Actions/Process Improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in WS-QA-0050, Management of Change Procedures.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. More detailed information on retention of specific records is provided in CW-L-P-001, Records Retention Policy and CW-L-WI-001, TestAmerica Records Retention/Storage Schedule. Quality records are maintained by the QA department in a database or in specific folders on the local QA share on a corporate server, which is backed up as part of the regular backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by Department Managers.

Table 14-1. Record Index[1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)

	Record Types [1]:	Retention Time:
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Business Operations	Refer to CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	Disposal Records (Add Permits?)	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual. All HR documents have different retention times.
	Administrative Policies	Indefinitely
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to CW-L-WI-001
	IT Records	Refer to CW-L-WI-001
	Corporate Governance Records	Refer to CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Logs are maintained in each storage box to note removal and return of records. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 14-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (lab reports and raw data) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
Ohio VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement
OSHA	30 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 and WS-PQA-017 for more information.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by the LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set.) Refer to SOP WS-QA-0009, Document Archiving. Instrument data is stored by project, except for inorganics and calibration data. Inorganics and calibration data is stored sequentially by instrument as appropriate. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP WS-QA-0009.

- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

14.2 Technical and Analytical Records

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook, on a benchsheet or in the LIMS.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

14.2.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a documented supervisory or peer review on a monthly basis.

14.3 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);

- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and

are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in a logbook or in the LIMS. Records are considered archived when noted as such in the records management system (a.k.a., document control.)

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CW-Q-S-003)	Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint responsibility: a) QA Manager or designee c) Technical Manager or Designee (Refer to CW-Q-S-003)	SOP Compliance Review Frequency • Minimum of every two years. • Annually for all methods and administrative SOPs relating to DoD/DOE programs.
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The

audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., MintMiner and Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. (Annually for methods and administrative SOPs related to DoD/DOE programs.) It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Soil, Water Supply, Water Pollution, Air, and round-robin studies for sediments and biological materials. When available for parameters tested by the laboratory, the laboratory will also participate in the DOE administered MAPEP program.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found within the 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been

affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Managers, their Quality Director as well as the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, VP of Operations, or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technical Managers, and QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel can be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources.
    - Adequacy of policies and procedures.
    - Future plans for resources and testing capability and capacity.

- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations and Quality Directors receive a monthly report from the Exec Director of Quality & EHS summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific labs.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, Conductivity, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Managers – **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Managers – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP (WS-QA-0022, Employee Orientation and Training).

17.4 Data Integrity and Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 66,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. In the event of a power outage, the laboratory can be equipped with a back up power supply for sample storage, as detailed in SOP No. WS-QA-0005, Temperature Monitoring and Corrective Action for Refrigerators and Freezers.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys and alarm codes are distributed to employees as necessary.

Employees wear photographic identification name cards while on the premises.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP WS-QA-0021 (Preparation and Management of Standard Operating Procedures).
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD/DOE SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Analysis and Sampling Procedures; 40 CFR Part 136 as amended by Method Update Rule; May 18, 2012.*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics & Organics Analysis, SOM, DLM, CBC, and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*

- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015..*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *Underground Storage Tanks Procedures Manual, State of Alaska Department of Environmental Conservation, Division of Spill Prevention and Response Contaminated Sites Program, November 7, 2002*
- *Tri-Regional Board Staff Recommendations for Preliminary Investigation and Evaluation of Underground Tank Sites, North Coast Regional Water Quality Control Board, San Francisco Bay Regional Water Quality Control Board and Central Valley Regional Water Quality Control Board, August 10, 1990*
- *Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997*
- *Compendium of Methods for the Determination of Air Pollutants in Indoor Air, (EPA 600/4-90-10, April 1990)*
- *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, (EPA 625/R-96/010a, June 1999*
- *Methods for Determining Emissions of Toxic Air Contaminants from Stationary Sources, Stationary Source Test Methods, Volume 3, California Air Resources Board*
- *Leaking Underground Fuel Tank Guidance Manual, September 2012, California State Water Resources Control Board*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC, Lab SOP # WS-QA-0022) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst hasn't performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

19.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP. If the concentration is unspecified, the routine LCS spike level may be used.

19.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

In accordance with Arizona Administrative Code R9-14-616.5f, documentation of each analyst's performance of proficiency testing, as applicable, will be maintained in the training record.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the

analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP. An SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Refer to the Corporate SOP No. CW-Q-S-006 or the laboratory's SOP No. WS-QA-0006 for details on the laboratory's MDL process.

19.8 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

19.9 Verification of Detection and Reporting Limits

Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at no more than 3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and no more than 4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. The analytes must be qualitatively identified or see SOP No. WS-QA-0006 for other options. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

For DoD ELAP certified methods, and methods utilized in support of DOE programs: Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD/DOE QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD/DOE QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation or utilized in support of DOE programs. Refer to the laboratory SOP WS-QA-0006, Method Detection Limits (MDL) and Instrument Detection Limits (IDL) for further details.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit and annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

For DoD ELAP certified methods and methods utilized in support of DOE programs: The laboratory quantitation limit is equivalent to the DoD/DOE Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD/DOE QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with the three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD/DOE projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

19.11 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.12 Estimation of Uncertainty of Measurement

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly,

and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy. If a problem is uncovered then the re-analysis will be repeated correctly. If no problem is uncovered then the laboratory will consult with the client to decide on actions needed.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Manager or Laboratory Director if unsure.

19.14 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOP Nos. CW-I-P-006, "Computer Systems Account and Naming Policy", CW-I-P-007, "Computer Systems Password Policy and CA-I-S-006, "Software Testing, Validation and Verification." The laboratory is currently running the TestAmerica Laboratory Information Management System ("TALS") which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Sequel Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 **Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protections, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.14.1.2 **Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 **Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The

spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and WS-PQA-011, Manual Integration Documentation Procedures.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/L) or micrograms per liter (µg/L) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1**%.** Units are defined in each lab SOP.

19.14.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.14.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst may print a copy of what has been entered to check for errors. This printout and the instrument's data file of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained within Chrom or the LIMS, based on the type of data.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample

ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z'd" out, signed and dated.
- Worksheets are created with the approval of the Technical Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are outlined in several SOPs (WS-PQA-003, "Quality Control Program", WS-PQA-012, "Technical Data Review Requirements", WS-PM-0004, "Final Report Assembly and Third Level Data Review") to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (WS-PQA-0011, "Manual Integration Documentation and Practices"). The general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.14.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.14.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.14.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.14.4.5 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.14.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that the COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The project manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.14.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.4.8 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the

guideline for our internal SOP No. WS-PQA-0011, entitled “Manual Integration Documentation and Practices”.

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices. Instrument operators must assure that all manual integration documentation identifies the analyst, the date and the reason for the integration.

Figure 19-1. Example - Demonstration of Capability Documentation

Analyst Demonstration of Capability

TestAmerica Sacramento

Victoria Nihart

4/24/2017

Preparation Method(s):	3535
Analytical Method(s):	GCMSMS_NDMA
Matrix:	Water
Method Description:	Nitrosamines by Isotope Dilution and GC/CI/MS/MS
Preparation SOP No:	WS-IDP-0020 Rev. 3.3
Analytical SOP No:	WS-MS-0012 Rev. 2.1

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of test method(s) and laboratory SOPs are available for all personnel on-site.
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

Technical Director	**Signature**	**Date** 5/3/17
Quality Assurance Officer	**Signature**	**Date** 5/3/17

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Analyst Demonstration of Capability

ANALYST DEMONSTRATION OF CAPABILITY

Method GCMSMS_NDMA **Laboratory:** TestAmerica Sacramento
Method Desc: Nitrosamines by Isotope Dilution and GC/CI/MS/MS
Analyst: Victoria Nihart **Limit Group:** MSS - NDMA - Water - QC

Current Limits — Recovery / Precision

Demonstration of Capability — Recovery / Precision

N-Nitrosodimethylamine
All values within Control limits

Analysis Dates: 6/1/2016 to 6/3/2016

LCL	UCL	Std Dev	Units
		20	%

Mean	Std Dev	Units		Amount	Amount/RL
90.09	8.53983	%	Pass	2.0	1

Laboratory ID	Anal Date	Batch	Smp	Analyst	Prep Analyst	Result	Units	Spike Amount	RL	% Rec		In Rec Limits?
LLCS 320-110927/3-A	06/01/2016	112005	17	Nihart, Victoria M	Mantri, Anil	1.618	ng/L	2.0	2.0	81		Pass
LLCSD 320-110927/4-A	06/01/2016	112005	18	Nihart, Victoria M	Mantri, Anil	1.962	ng/L	2.0	2.0	98	19	Pass
LLCS 320-111960/3-A	06/03/2016	112484	4	Nihart, Victoria M	Kuzmenko, Natalia	1.931	ng/L	2.0	2.0	97		Pass
LLCSD 320-111960/4-A	06/03/2016	112484	5	Nihart, Victoria M	Kuzmenko, Natalia	1.695	ng/L	2.0	2.0	85	13	Pass

N-Nitrosodimethylamine-d6
All values within Control limits

Analysis Dates: 6/1/2016 to 6/3/2016

LCL	UCL	Std Dev	Units
25	150		%

Mean	Std Dev	Units		Amount
75.77	7.50265	%	Pass	100.0

Laboratory ID	Anal Date	Batch	Smp	Analyst	Prep Analyst	Result	Units	Spike Amount	% Rec		In Rec Limits?
LLCS 320-110927/3-A	06/01/2016	112005	17	Nihart, Victoria M	Mantri, Anil	80.88	ng/L	100.0	81		Pass
LLCSD 320-110927/4-A	06/01/2016	112005	18	Nihart, Victoria M	Mantri, Anil	81.20	ng/L	100.0	81	0.4	Pass
LLCS 320-111960/3-A	06/03/2016	112484	4	Nihart, Victoria M	Kuzmenko, Natalia	65.13	ng/L	100.0	65		Pass
LLCSD 320-111960/4-A	06/03/2016	112484	5	Nihart, Victoria M	Kuzmenko, Natalia	75.88	ng/L	100.0	76	15	Pass

Precision = standard deviation of percent recoveries of spiked control samples.

4/24/2017

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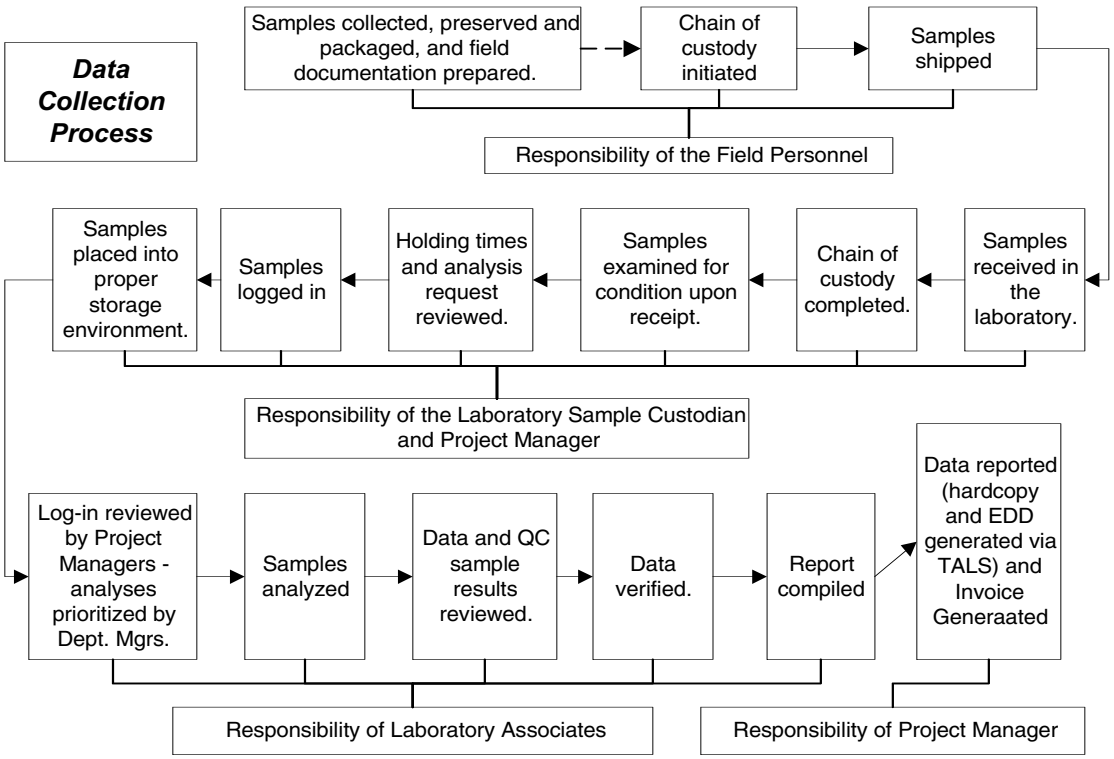
Figure 19-2. Example: Work Flow



SECTION 20. EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may be / are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or

instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLv) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by an ISO 17025 qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. See SOP No. WS-QA-0041, "Calibration and Calibration Check of Balances" for more details.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in their logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer at temperatures bracketing the range of use.

- If the temperature measuring device is used over a range of 10℃ or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10℃, then the verification must bracket the range of use.

IR thermometers, digital probes and thermocouples are calibrated quarterly. IR Thermometers should be calibrated over the full range of use, including ambient, iced (4 degrees) and frozen (0 to -5 degrees), per the Drinking Water Manual.

The digital NIST thermometer is recalibrated every five years by an approved outside service and the provided certificate of traceability is kept on file. Alternately a new NIST thermometer with certificate of traceability from the manufacturer may be purchased. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the SOP No. WS-QA-0016, "Thermometer Calibration."

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample storage are monitored 7 days a week; and each working day for units used for standard storage.

Ovens and water baths are monitored on days of use. Drying oven temperature must be recorded before and at the end of use. For example, an oven used for moisture determination must have its temperature recorded at the start and end of the drying process. Temperature must be ± 5% of set temperature for DoD/DOEwork.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept at > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, and water baths can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements. See SOP WS-QA-0004, “Maintenance and Calibration Check of Fixed and Adjustable Volume Autopipettors, Autodispensers and Volumetric Containers”.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy. The laboratory also assigns a unique ID# to each syringe. The delivery volume of each syringe is verified gravimetrically before initial use.

20.3.6 Autoclaves

Autoclaves used for sample digestion are capable of maintaining conditions of 15 psi at 120ºC for 15 minutes. The temperature of the autoclave is verified quarterly.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually however, the annual requirement does not apply to Isotope Dilution methods.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least each daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. (Exception: Some QC programs, such as the DoD/DOE QSM Version 5, require bracketing standards with internal standard calibration). The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements - see specific SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with unacceptable calibration verification may be fully useable under the following special conditions and reported based upon discussion and approval of the client:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision

level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Guidelines for evaluating and reporting TICs are in the specific laboratory SOPs.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS Tuning

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Example: Instrumentation List

Instrument Type	Number in Use
Autoanalyzer	1
Autotitrator	1
Cold-Vapor Analyzers	1
GC/HRMS	6
GC/MS - Semivolatiles	6
GC/MS - Volatiles	5
GC/MS – Volatile Air	5
GC/MS/MS	1
GC-ECD/ECD	7
GC-FID/FID	2
GC-FID	1
GC-FPD	1
GC-TCD/TCD	1
HPLC	5
HPLC/MS/MS	5
ICP	1
ICP/MS	1
Ion Chromatograph	3
Spectrometer	1

Table 20-2. Example: Schedule of Routine Maintenance

INSTRUMENT	MAINTENANCE	FREQUENCY
APCI/ESI LC/MS/MS	Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes.	As Needed
	Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that corona and multiplier are functioning.	Daily(2)
	Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable.	Semi-Annually
	Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.	Annually
HIGH PRESSURE LIQUID CHROMATOGRAPH(1)	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements. Rinse flow cell with 1N nitric acid if dirty flow cell. Change pump seals when flow becomes inconsistent. Backflush column if applicable. Change in-line filters for solvents.	As Needed
	Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse delivery lines to prevent contamination of the new solvent. Check gas supply if applicable. Flush with an appropriate solvent to remove all bubbles. Pre-filter all samples.	Daily(2)
	Change pump seals.	Every 6-9 Months

INSTRUMENT	MAINTENANCE	FREQUENCY
GAS CHROMATOGRAPH(1)	Replace septum. Clean injector port Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed. Replace or repair flow controller if constant gas flow cannot be maintained. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace igniter. ECD: follow manufacturers suggested maintenance schedule PID: Clean lamp window or replace. Replace seals. Replace fuse. Reactivate external carrier gas dryers. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents. Check inlets, septa.	As Needed
	Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Daily(2)
	Oxidation and Reduction Catalysts: Perform leak checks. Replace/condition when poor response is observed.	Quarterly
	ECD: perform wipe test.	Semi-Annually
PURGE AND TRAP SYSTEMS	Change trap. Check purge flow. Flush lines (after foaming sample). Periodic leak checks (when replace traps/spargers) Replace/condition traps and/or spargers (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), and clean or replace glassware/spargers. Bake trap as needed to correct for high background. Change trap whenever loss of sensitivity, or erratic response or failing resolution is observed. Purge & trap autosamplers: leak check system, clean sample lines, valves.	As Needed
	Bake out trap & analyze primers (as needed) prior to commencing analysis.	Daily(2)
GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROMETER(1)	Replace septum. Clean injector port. Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Replace injection port liner when front portion of capillary column is removed. Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed.	As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels. Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination. Replace filaments when both filaments burn out or performance indicates need for replacement. Check mass calibration (PFTBA or FC-43). Check ion source and analyzer (clean, replace parts as needed). Check vacuum, relays, gas pressures and flows. Change oil in the mechanical rough pump. Relubricate the turbomolecular pump-bearing wick. HP 7673 Autosampler: Replace syringe.	
	Check for sufficient gas supply. Check for correct column flow and/or inlet pressure. Check temperatures of injector, detector. Verify temperature programs. Check inlets, septa. Check baseline level. Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks. Autosampler: fill wash bottle, dispose of waste bottle contents. Air Autosampler: Check for proper operation. Leak check system.	Daily[2]
	Replace the exhaust filters on the mechanical rough pump every 1-2 years.	Annually
GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETER[1]	Full Bake-Out. Change oil in rotary pump. Change oil in diffusion pump. Replace o-rings. Solvent rinse the flight tube. Clean the first field free region. Check detector voltages. Clean and dust connectors, etc on the outside of the instrument. Check the vacuum: ~5 x. 10^{-7} MBAR on both analyzer ion gauges, and ~5 x 10^{-6} MBAR on the source, with no helium flowing. Check isolation valve for leaks, correct if needed. Check for thermal trip by taking the magnet to maximum current, and verify that the coolant flow is acceptable. Replace septum. Clean injector port. Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Replace injection port liner when front portion of capillary column is removed. Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms	As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination. Replace filaments when performance indicates need for replacement.	
	Check resolution sensitivity. Check stability. Check for sufficient gas supply. Check for correct column flow and/or inlet pressure. Check temperatures of injector, detector. Verify temperature programs. Check inlets, septa. Check baseline level. Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Daily(2)
COLD VAPOR ATOMIC ABSORPTION (LEEMAN PS 200) (1)	Change pump tubing. Check/change Hg lamp. Clean optical cell. Change drying tube. Grease pump.	As Needed
	Check sample tip for clogs. Check drying tube. Check pump tubing/drain tubing. Check gas pressure. Check liquid/gas separator. Check tubing.	Daily(2)
INDUCTIVELY COUPLED ARGON PLASMA/MASS SPECTROMETRY (ICAP/MS)(1)	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics. Measure quartz torch for proper alignment when removed and cleaned. Clean spray chamber and nebulizer. Clean all filters and fans. Check chiller coolant level. Check and drain oil mist eliminator on roughing pumps.	As Needed
	Check sample waste container level. Check quartz torch condition. Check RF coil. Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing. Check condition of sampler and skimmer cones. Check oil level of roughing pumps.	Daily(2)
	Replace oil in roughing pumps.	Every 2-3 Months
ICP(1)	Check that argon feed pressure is 80-120 psi. Check that chiller coolant pressure is 45-80 psig, no leaks. Check purge and shear gasses. Nitrogen purge gas pressure 40-120 psig, compressed air shear gas pressure 80-120 psig. Check radial purge and axial windows for deposits. Check that nebulizer is not clogged. Check that capillary tubing is clean and in good condition. Check that peristaltic pump windings are secure. Check that exhaust vent is operational Check that torch, glassware, aerosol injector tube are clean.	Daily(2)

INSTRUMENT	MAINTENANCE	FREQUENCY
	Clean plasma torch assembly to remove accumulated deposits. Check RF coil. Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance. Clean filters on back of power unit to remove dust. Replace when needed: peristaltic pump tubing. sample capillary tubing. autosampler sipper probe. Check performance with manganese. Check O-rings. Clean/lubricate pump rollers	Monthly or As Needed
	Check chiller coolant filter. (may require more or less frequently)	Semi-Annually
	Notify manufacturer service engineer for scheduled preventive maintenance service.	Annually
ION CHROMATOGRAPH[1]	Clean micromembrane suppressor when decreases in sensitivity are observed. Check fuses when power problems occur. Change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated. De-gas pump head when flow is erratic. Check all air and liquid lines for discoloration and crimping, if indicated. Check/change bed supports guard and analytical columns, if indicated.	As Needed
	Check plumbing/leaks. Check eluent level. Check gases. Check pump pressure. Check conductivity meter.	Daily[2]
	Check pump heads for leaks. Check filter (inlet).	Weekly
	Change pump seals. Change injection valve. Clean conductivity cell. Check conductivity cell for calibration.	Annually
ALPKEM COLORIMETRIC AUTO ANALYZER[1]	Prepare fresh reagents. Replace tubing. (About every 100 hours of use)	As Needed
	Check detector. Make sure there are no trapped bubbles in detector cell. Check Valves Check peristaltic tubing. Check sampler.	Daily[2]
	Clean pump, and XYZ Sampler.	Weekly
	Lubricate pump roller.	Monthly
	Clean pump rollers with steel wool and lubricate.	Semi-Annually
CHEMICAL OXYGEN DEMAND (COD) REACTOR[1]	Electronics serviced.	As Needed
	Check temperature with NIST reference thermometer.	Annually
AUTO TITRATOR[1]	Electronics serviced.	As Needed
	Calibrate with check standards.	Daily[2] (When Used)

INSTRUMENT	MAINTENANCE	FREQUENCY
	Inspect electrodes daily, clean as needed. Inspect electrode proper levels of filling solutions daily, fill as needed. Clean probe, each use. Prime buret Check rinse water reservoir.	
CONDUCTANCE METER(1)	Electronics serviced. Replace batteries	As Needed
SPECTROPHOTOMETER(1)	Replace lamp. Replace fuse.	As Needed
	Check instrument manual. Perform wavelength calibration. Replace lamp annually or when erratic response is observed.	Annually
PH METER(1)	Clean electrode. Refill reference electrode.	As Needed
	Inspect electrode. Verify electrodes are properly connected and filled. Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer.	Daily(2)
TURBIDIMETER(1)	Electronics serviced.	As Needed
	Clean instrument housing.	Monthly
DIGESTION BLOCK	Check temperature with NIST thermometer.	Annually
SONICATOR (1)	Replace probe tip. Disassemble and clean sonicator probe tips. Tune sonicator assembly (if recommended by manufacturer)	As Needed
	Inspect probe tips for inconsistencies (etching/pitting).	Daily(2) (When Used)
ANALYTICAL/TOP LOADING BALANCES(1)	Check using ASTM Class 3 weights once daily or before use. Clean pan and weighing compartment.	Daily(2)
REFRIGERATORS/WALK-IN COOLERS(1)	Manufacturer cleaning and calibration.	Annually
	Refrigerant system and electronics serviced.	As Needed
	Temperatures checked and logged.	Daily(2)
OVENS(1)	Electronics serviced.	As Needed
	Temperatures checked and logged.	Daily(2)
ZYMARK PE WORKSTATION	Change O-rings whenever there are visible leaks or poor sealing on the SPE columns. Sample lines are clean after samples have been extracted by SPE with a program “Clean Sample Lines” with methanol followed by water. Occasionally for a more rigorous cleaning, or after a highly contaminated sample, a mixture of methanol/DCM at 50:50 may be used in place of methanol, follow by methanol, then water (never use acetone). Syringe pump may be primed using a program “Prime Solvent Lines” whenever air bubbles are suspected in the lines from running out of solvents and whenever solvents are changed. Syringe pump in good condition – replace if showing signs of wear or suspected of poor performance. Sample pumps may be re-calibrated whenever major	As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	repairs are performed, or whenever the pumps are suspected to be out of calibration. Follow manufacturer's procedure for re-calibrating the sample pumps. For method 8330, the pump loads 1050 mL of sample on the SPE. It should used up the whole sample bottle (quart bottles and 1-L bottles).	
SONICATION WATER BATH(1)	If the water bath is dirty, empty and refill with tap water. A couple drops of anti-bacterial solution may be added to inhibit the growth of bacteria in the water. The water level in the sonication batch should be about 1.2 to 1 inch from the top while in operation. Do not allow sonication batch to operate with water bath at lower levels. If the level is low, add more water, if the levels is too high, remove water to the proper level.	As Needed

Footnotes to Preventive Maintenance Tables

(1) Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.

(2) Daily checks and verifications are performed prior to instrument startup and are not documented in maintenance logs unless problems are noted.

(3) Where there are differences between this table and the tables present in method SOPs, the table in the method SOP should be followed.

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program) or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International al Laboratory Accreditation Cooperation) or APLC (Asia-Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

The calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty.

The uncertainty calculations of the calibration laboratory are supported by uncertainty budgets and are represented by expanded uncertainties typically using a coverage factor of k=2 to approximate the 95% confidence level. This explanation accompanies the measurement result and the associated uncertainty.

The tolerance uncertainty ratio (TUR) is calculated using the expanded uncertainty of the measurement, not the collective uncertainty of the measurement standards. A statement to this effect accompanies the TUR along with the coverage factor and confidence level.

The calibration report or certificate submitted to TestAmerica Sacramento contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis

upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

The calibration laboratory supports in-house calibration systems: documented procedures for in-house calibrations, evidence by a report, certificate, or sticker, for an appropriate amount of time; training records of calibration personnel; certificates from accreditation services demonstrating traceability to national or international standards of measurement; procedures for evaluating measurement uncertainty; timely and documented recalibration of reference standards. When subcontracting to a calibration laboratory, TestAmerica Sacramento does not use a firm who subcontracts the work.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

.

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a

second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. [Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.]

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are scanned and retained on the local server. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and SOP No. WS-QA-0017, "Standards and Reagents and Quality Control Check Procedures".

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material (for 1613B dioxin/furan analyses the purity must be 98% or corrections must be made). Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database or standards logbook.

- Standard ID
- Description of Standard

- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically or in logbooks for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from the preparation logbook)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the SDS section of OASIS.

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions.
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificate may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However, there are some programs and regulators, which determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative.

As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located SOP Nos. WS-QA-0018, “Subsampling and Compositing of Samples (Method ASTM D 6323-98)” and WS-QA-0028, “Incremental Sampling Methodology of Soils and Sediments”.

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her

view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the COC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, legal COCs will be generated per the Manual for Certification of Laboratories Analyzing Drinking Water, Fifth Edition, January 2005, Appendix A, and SOP No. WS-QA-0003, "Sample Receipt and Procedures".

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections and in SOP No. WS-QA-0003, "Sample Receipt and Procedures".

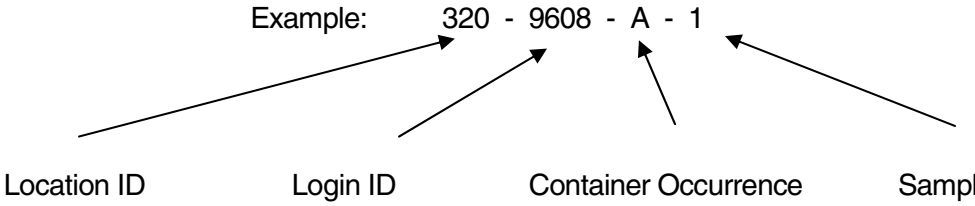
23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on the lot receipt checklist and within the non-conformance program and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record. Laboratory receipt procedures are described in more detail in SOP No. WS-QA-0003.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Sacramento Laboratory (Location 320) is the receiving laboratory. Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 320 - 9608 - A - 1 - **A** ←——**Secondary Container Occurrence**

Example: 320-9608-A-1-A would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. WS-QA-0003.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples are returned to the secure sample control area. Empty sample containers are marked as “DIT” (destroyed in testing) on the sample receiving check out form and are disposed by the analytical staff. All samples are kept in the refrigerators for 30 days past invoicing, unless other arrangements have been made with the client.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 Hazardous Samples and Foreign Soils

Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

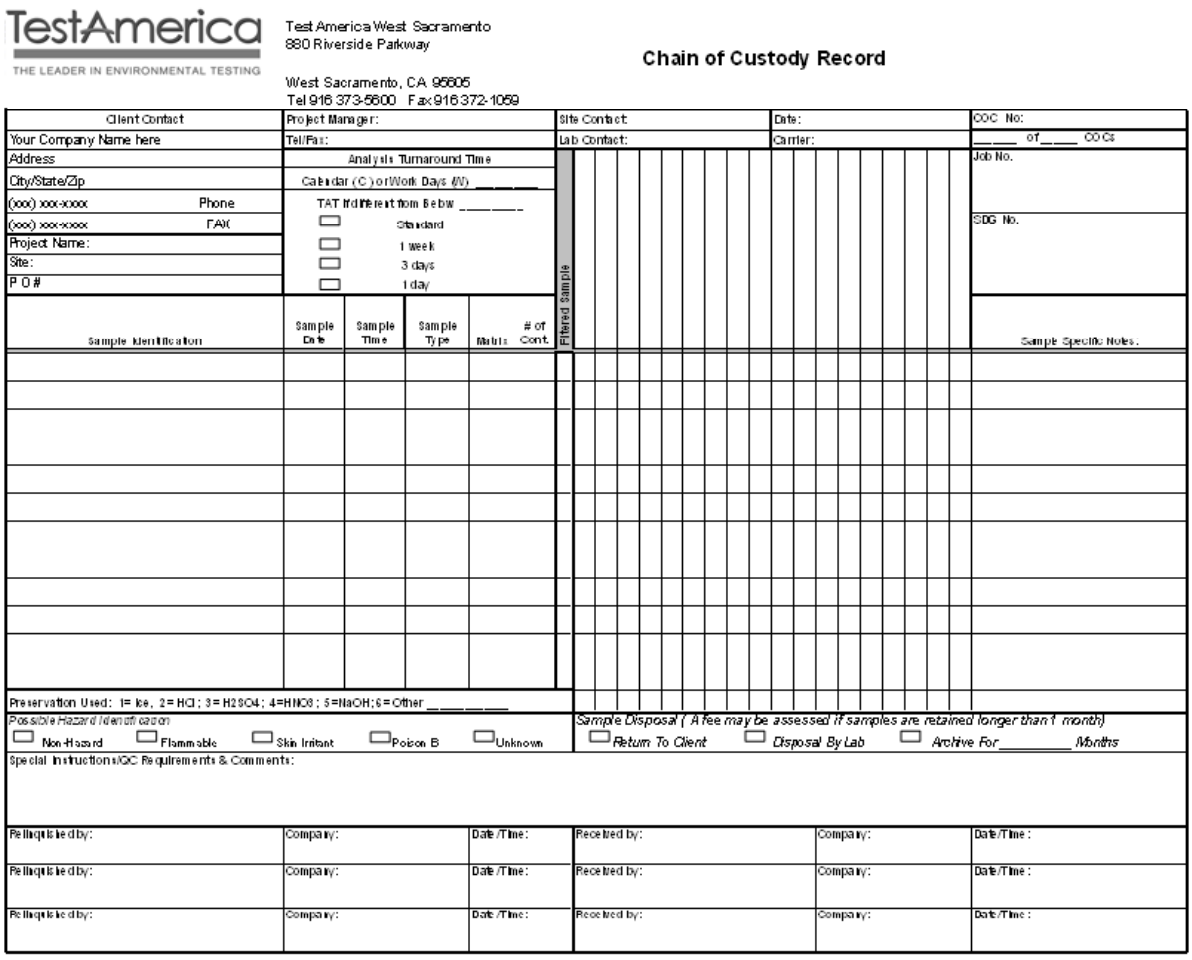
Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. An exception is samples contained in laboratory-owned air sample canisters. These are held for a minimum of 24 hours after the project report is sent, prior to evacuating the canister and returning it to the equipment pool. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: WS-EHS-001, "Waste Disposal"). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Example: Chain of Custody (COC)



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West Sacramento, CA 95605
Tel 916 373-5600 Fax 916 372-1059

Chain of Custody Record

Client Contact	Project Manager:	Site Contact:	Date:	COC No:
Your Company Name here	Tel/Fax:	Lab Contact:	Carrier:	____ of ____ COCs
Address	Analysis Turnaround Time			Job No.
City/State/Zip	Calendar (C) or Work Days (W) _____			
(xxx) xxx-xxxx Phone	TAT if different from Below _____			SDG No.
(xxx) xxx-xxxx FAX	☐ Standard			
Project Name:	☐ 1 week			
Site:	☐ 3 days			
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type	# of Cont. Matrix	Filtered Sample	Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For _____ Months*

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:

Figure 23-2. Example: Sample Acceptance Policy

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SACRAMENTO LABORATORY
SAMPLE ACCEPTANCE POLICY

(Effective 02/02/2015)

Page 1 of 2

The TNI Standard and TestAmerica Sacramento have specific requirements under which all samples will be received by the laboratory for analysis. TestAmerica Sacramento will review your sample shipment against those requirements as listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

VOA vials should be stored in controlled conditions. Exposure of trip blanks to temperature fluctuations is likely to cause development of bubbles in the trip blanks.

When completing the chain of custody form, please note that you must sign your name in the "relinquished by" box.

Requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples, shall be provided.
- Samples must be accompanied by written disclosure of the known or suspected presence of any hazardous substances, as defined by applicable federal or state law.
- Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).
- Each sample shall be collected in the appropriate sample container and labeled with unique, durable and indelible identification.
- Drinking water samples for Method 1613B that may have residual chlorine must be checked and treated in the field, or collected in sodium thiosulfate preserved containers.
- Containers of water meant for perchlorate analysis should have adequate headspace to prevent anaerobic microbial degradation. A void approximately 1/3 of the container volume is sufficient.
- The samples shall arrive at the laboratory with adequate remaining holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.
- Received samples must not exhibit obvious signs of damage, contamination or inadequate preservation.
- Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C and above freezing (0°C). For methods with other temperature criteria (e.g. some bacteriological methods require ≤ 10 °C), the samples must arrive within ± 2° C of the required temperature or within the method specified range.

    1. Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements above. In these cases, the samples shall be considered acceptable if the samples were received on ice.
    2. If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.
    3. Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

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SACRAMENTO LABORATORY SAMPLE ACCEPTANCE POLICY

(Effective 02/02/2015)

- For samples undergoing chemical warfare degradate analysis, the sample must be screened for agent prior to shipment in accordance with appendix 10 of our Sample Receipt Procedure (WS-QA-0003).
- Samples containing mammalian tissue will not be accepted without prior coordination with a project manager. Additional conditions for receipt and handling of tissue are outlined in appendix 11 of our Sample Receipt Procedure (WS-QA-0003).
- Air canisters (SUMMA® and other brands) have additional requirements:
    - Never write or affix a label directly on a canister. A special tag is attached to each canister for this purpose.
    - Complete the Canister Field Data Record with the initial and final vacuum/pressure reading for each canister during sampling.
    - Close all valves completely prior to shipping or transporting.
    - Return canisters, filters, flow controllers, vacuum flow regulators, and any other supplied equipment must be returned even if they were not used. Pack equipment carefully to minimize in-transit damage Sampling equipment that is damaged, lost or not returned will be invoiced to the client at the replacement cost. Delayed return of equipment to the laboratory may result in additional rental charges.
    - Do not attempt to adjust or alter any equipment, as it may result in loss of sample integrity as well as equipment damage that may be invoiced to the client.

The laboratory will notify the client/Project Manager upon sample receipt if the samples fail to meet any of the above requirements.

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Figure 23-3. Example: Cooler Receipt Form

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Sacramento
Sample Receiving Notes

Job:_____

Tracking #_____

Use this form to record Sample Custody Seal, Cooler Custody Seal, Temperature & corrected Temperature & other observations. File in the job folder with the COC.

Notes:_____

Therm. ID: **AK-1** / **AK-2** / **HACCP** /Other_____

Ice_____ Wet_____ Dry_____ Other_____

Cooler Custody Seal: _____

Sample Custody Seal: _____

Cooler ID:_____

Temp: Observed_____

Corrected: _____

From: Temp Blank ☐ Sample ☐

NCM Filed: Yes ☐ No ☐

	Yes	No	NA
Perchlorate has headspace?	☐	☐	☐
CoC is complete w/o discrepancies?	☐	☐	☐
Samples received within holding time?	☐	☐	☐
Sample preservatives verified?	☐	☐	☐
Cooler compromised/tampered with?	☐	☐	☐
Samples compromised/tampered with?	☐	☐	☐
COC and Samples w/o discrepancies?	☐	☐	☐
Sample containers have legible labels?	☐	☐	☐
Containers are not broken or leaking?	☐	☐	☐
Sample date/times are provided.	☐	☐	☐
Appropriate containers are used?	☐	☐	☐
Sample bottles are completely filled?	☐	☐	☐
Zero headspace?*	☐	☐	☐
Multiphasic samples are not present?	☐	☐	☐

Initials:_____ Date:_____

*Containers requiring zero headspace have no headspace, or bubble < 6 mm (1/4")

Q:\DOCUMENT-MANAGEMENT\FORMS\QA-812 REV. 1.3 SAMPLE RECEIVING NOTES 2017-02-27.DOC QA-812 RKE 02/27/2017

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Bottle Lot Inventory

Lot ID:_____

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
VOA*																				
VOAh*																				
AGB																				
AGBs																				
250AGB																				
250AGBs																				
250AGBn																				
500AGB																				
____AGJ																				
500AGJ																				
250AGJ																				
125AGJ																				
____CGJ																				
500CGJ																				
250CGJ																				
125CGJ																				
PJ																				
PJn																				
500PJ																				
500PJn																				
500PJna																				
500PJzn/na																				
250PJ																				
250PJn																				
250PJna																				
250PJzn/na																				
Acetate Tube																				
_____"CT																				
Encore																				
Folder/filter																				
PUF																				
Petri/Filter																				
XAD Trap																				
Ziploc																				
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20

h = hydrochloric acid s = sulfuric acid na = sodium hydroxide n = nitric acid zn = zinc acetate

Number of VOAs with air bubbles present / total number of VOA's

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SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, reflux, evaporation, and drying. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

Table 24-1. Example – Negative Controls

Control Type	Details
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous

volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 30 data points (more points are preferred, however, fewer (minimum of 20) may be used to establish tentative acceptance limits in select circumstances).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

- The maximum acceptable recovery limit will be 150%. Some specific methods or SOPs may allow for higher recoveries.

- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

- If either the high or low end of the control limit changes by ≤ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. See SOP WS-QA-0035 for further details.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.

- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Or, for TNI and DoD/DOE work, there are an allowable number of random Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed or prepared electronically on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report for Samples) with a "sample results" column header.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. work order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##, where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.
- In most cases, the applicable COC is an integral part of the report.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., Sampling information).

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.22 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.23 When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

25.2.24 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.25 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary report). A complete report must be sent once all of the work has been completed.

25.2.26 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.27 A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory's accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

25.2.28 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level II is a report with the features described in Section 25.2 above, plus summary information, including results for the method blank reported to the laboratory MDL if required, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. No raw data is provided unless it is necessary to provide the relevant calibration information.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in electronic deliverable form via e-mail, posting to an FTP site, or CD ROM. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services in addition to the test report as described in section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. TestAmerica Sacramento offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD, and a copy filed on the QA share of the local server.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CW-L-S-004).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationery and the report includes any accompanying documentation.

25.6 Client Confidentiality

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are to meet all requirements of this document and to include a cover letter.

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R". Every page will have the report generation date present, to prevent confusion between report versions.

When the report is re-issued, a notation of "Revision "with the revision number is placed on the cover/signature page of the report. The case narrative is updated *with* a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example*: *Report was revised on 11/3/11 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/11.*

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

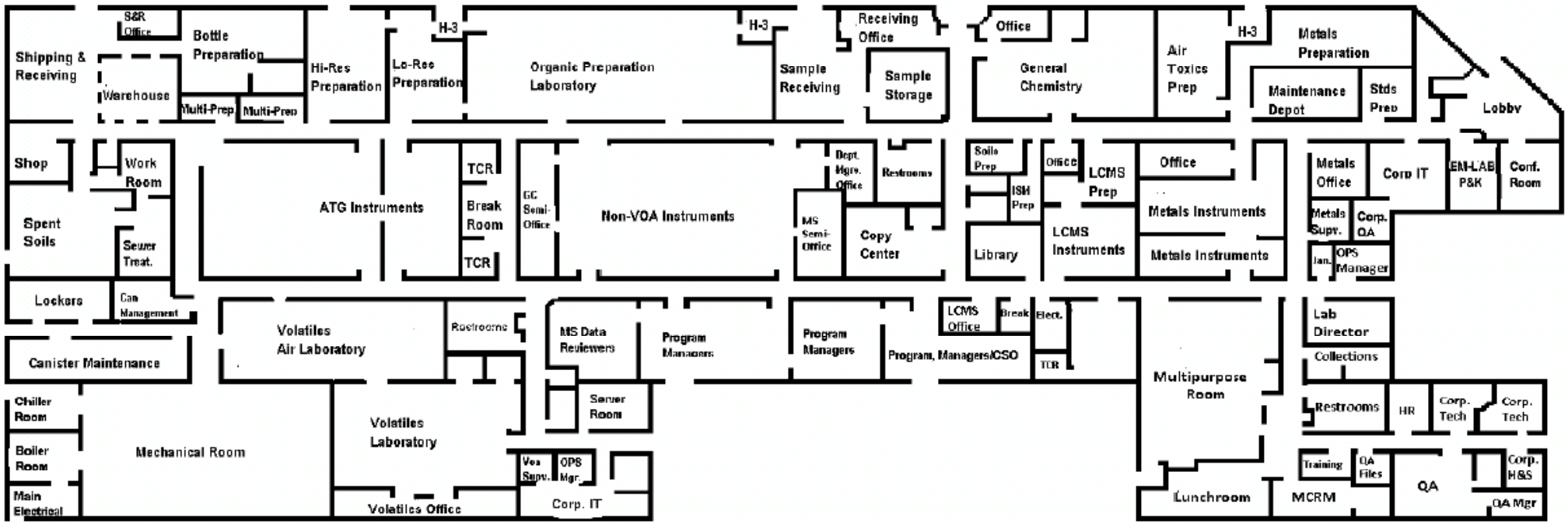
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. Laboratory Floor Plan



Facility Size	Square Feet
Total Area	66.000
Lab Area	44,725
Storage Area	5200
	Linear Feet
Bench Top	3036
Hoods	464

Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Air Sample Bag: A sampling container for air samples, commonly referred to as Flex-Film or Tedlar bag, in 1.0-L or 3.0-L volumes, that is constructed of proprietary material (E.G., SKC or ESS).

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Passivated Canister: A sampling container for air samples; commonly referred to as a SUMMA canister, SilcoCan or T.O.-Can in 1.0, 1.8 6, or 15 L volumes.

1) SUMMA canister: A spherical stainless steel canister, of which the interior has been specially treated by a process (SUMMA passivation) that renders all surfaces inert to VOCs.

2) SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most VOCs.

3) T.O.-Can: A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek using a proprietary electropolishing process and is extensively cleaned using an ultrasonic method that ensures a high-quality passivated surface that maintains the stability of VOCs during storage.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Quality System Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

A2LA – American Association for Laboratory Accreditation
ANSI – American National Standards Institute
ASQ – American Society for Quality
CAR – Corrective Action Report
CCB – Continuing Calibration Blank
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICB – Initial Calibration Blank
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SDS - Safety Data Sheet
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
TALS – TestAmerica LIMS system
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Sacramento maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

The certificates and accredited parameter lists are available, for each State/Program organization at www.testamericainc.com under Analytical Services Search – Certifications.

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TestAmerica Certifications

Laboratory	Program	Authority	Identification	Expiration Date
TestAmerica Sacramento	DoD ELAP	L-A-B	L2468	01/20/2018
TestAmerica Sacramento	Federal	US Fish & Wildlife	LE148388-0	10/31/2017
TestAmerica Sacramento	Federal	USDA	P030-11-00436	12/30/2017
TestAmerica Sacramento	Federal	USEPA UCMR	CA00044	11/06/2018
TestAmerica Sacramento	NELAP	Florida	E87570	06/30/2017
TestAmerica Sacramento	NELAP	Illinois	200060	03/17/2018
TestAmerica Sacramento	NELAP	Kansas	E-10375	10/31/2017
TestAmerica Sacramento	NELAP	Louisiana	30612	06/30/2017
TestAmerica Sacramento	NELAP	New Hampshire	2997	04/18/2018
TestAmerica Sacramento	NELAP	New Jersey	CA005	06/30/2017
TestAmerica Sacramento	NELAP	New York	11666	04/01/2018
TestAmerica Sacramento	NELAP	Oregon	4040	01/28/2018
TestAmerica Sacramento	NELAP	Pennsylvania	68-01272	03/31/2018
TestAmerica Sacramento	NELAP	Texas	T104704399	07/31/2017
TestAmerica Sacramento	NELAP	Utah	CA00044	02/28/2018
TestAmerica Sacramento	NELAP	Virginia	460278	03/14/2018
TestAmerica Sacramento	State Program	Alaska (UST)	UST-055	12/18/2017
TestAmerica Sacramento	State Program	Arizona	AZ0708	08/11/2017
TestAmerica Sacramento	State Program	Arkansas DEQ	88-0691	06/17/2018
TestAmerica Sacramento	State Program	California	2897	01/31/2018
TestAmerica Sacramento	State Program	Colorado	CA00044	08/31/2017
TestAmerica Sacramento	State Program	Connecticut	PH-0691	06/30/2017
TestAmerica Sacramento	State Program	Hawaii	N/A	01/29/2018
TestAmerica Sacramento	State Program	Maine	CA0004	04/18/2018
TestAmerica Sacramento	State Program	Michigan	9947	01/31/2018
TestAmerica Sacramento	State Program	Nevada	CA00044	07/31/2017
TestAmerica Sacramento	State Program	Washington	C581	05/05/2018
TestAmerica Sacramento	State Program	West Virginia (DW)	9930C	12/31/2017
TestAmerica Sacramento	State Program	Wyoming	8TMS-L	01/29/2017 *

Appendix 4: Listing of Methods Performed

Preparation Only Methods

Method	Aqueous	Solid	Waste	Biological	Air
Organics					
Calif. CAM-WET	X	X	X		
EPA 1311	X	X	X		
EPA 3510C	X				
EPA 3535	X				
EPA 3540B		X			
EPA 3542					X
EPA 3546		X			
EPA 3550B		X		X	
EPA 3580A			X		
EPA 3600C	X	X	X		
EPA 3620B	X	X	X		
EPA 3630C	X	X	X		
EPA 3640A	X	X		X	
EPA 5030B	X	X	X		
EPA 5035	X	X	X		
Inorganics					
Calif. CAM WET	X	X	X		
EPA 1311	X	X	X		
EPA 1312 (E/W)	X	X	X		
EPA 3005A	X				
EPA 3010A	X				
EPA 3050B		X	X	X	

Organics Methods Performed

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
Volatile Organics	SW846 8260B	X	X	X		
	SW846 8260C	X	X	X		
	EPA 624	X				
	TO-14A					X
	TO-15					X
Sulfur Containing Compounds	EPA 15/16					X
Fixed Gases	ASTM D1946					X
	EPA 3C					X
Base Neutrals and Acids (BNAs)	SW846 8270C	X	X	X	X	
	SW846 8270D	X	X	X	X	
	EPA 625	X				
	TO-13A					X
	IP-7					X
	EPA 23					X
Organochlorine Pesticides	SW846 8081A	X	X	X	X	
	SW846 8081B	X	X	X	X	
	EPA 608	X				
	TO-4A					X
	TO-10A					X
PCBs (Aroclors)	EPA 8082	X	X	X	X	
	EPA 8082A	X	X	X	X	
	EPA 608	X				
	TO-4A					X
	TO-10A					X
PCB Congeners	EPA 1668A	X	X	X	X	X
	EPA 1668C	X	X	X	X	X
Petroleum Hydrocarbons	EPA 8015B	X	X	X		
	EPA 8015D	X	X	X		
	CA LUFT	X	X	X		
	AK101	X	X	X		
	Ak102	X	X	X		
	AK103	X	X	X		
	GRO/DRO	X	X	X		
Nitroaromatics and Nitroamines	EPA 8330A	X	X	X		
	EPA 8330B	X	X	X		
	WS-LC-0010	X	X	X		
Nitrosamines	WS-MS-0012	X	X			

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
PAHs	EPA 8270C (SIM Isotope dilution)	X	X	X	X	X
	EPA 8270C (SIM)	X	X	X		
	CARB 429	X	X	X	X	X
	TO-13A					X
1,4-Dioxane	WS-MS-0010	X				
Alkyl Phenols	WS-MS-0013	X	X		X	
Perfluorinated Compounds	WS-LC-0025	X	X	X	X	
	ISO 25101	X				
(including PFOA/PFOS)	EPA 537	X				
Dioxins & Furans	EPA 1613B	X	X			
	EPA 8290	X	X	X	X	
	EPA 8290A	X	X	X	X	
	EPA 8280A	X	X	X	X	
	EPA 8280B	X	X	X	X	
	EPA 0023A					X
	EPA 23					X
	TO-9					X

Metals Methods Performed

Parameter	Methods	Aqueous	Solid	Waste	Biological	Air
Trace Metals	EPA 6010B	X	X	X	X	X
	EPA 6020	X	X	X	X	X
	EPA 0060					X
	EPA 200.7	X				
	EPA 200.8	X				
	EPA 12					X
	CARB 12					X
	EPA 29					X
	CARB 436					X
Hardness	SM 2340B	X				
Mercury	EPA 7470A	X				
	EPA 245.1	X				
	EPA 7471A		X	X	X	X
	EPA 101A					X
	ASTM D6784-02					X
	EPA 0060					X
	EPA 29					X
	CARB 436					X

Inorganics Methods Performed

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
Alkalinity (Carbonate, Bicarbonate, Total)	SM 2320B	X				
Bromide, Chloride, and Fluoride	EPA 300.0	X				
	EPA 9056	X	X			
	EPA 9057					X
	EPA 26A					X
	CARB 421					X
Chromium, Hexavalent	EPA 7196A	X				
	EPA 0061					X
	EPA 306					X
	CARB 426					X
Conductivity	EPA 9050A	X				
	SM 2510 B	X				
Demand, Chemical Oxygen	EPA 410.4	X				
Moisture	ASTM 2216		X			
Nitrate	EPA 353.2	X				
	EPA 300.0	X				
	EPA 9056	X	X			
	CARB 421					X
Nitrate-Nitrite	EPA 353.2	X				
Nitrite	EPA 353.2	X				
	EPA 300.0	X				
	EPA 9056	X	X			
	CARB 421					X
Nitrocellulose	EPA 353.2	X	X			
	WS-WC-0050	X	X			
Orthophosphate	EPA 300.0	X				
	EPA 9056	X	X			
Particulates in Air	EPA 5					X
	40 CFR Part 50					X
Perchlorate	EPA 314.0	X				
	EPA 331.0	X				
	EPA 6850	X	X			
pH	SM 4500 H+ B	X				
	EPA 150.2	X				
	EPA 9040A	X				
	EPA 9041A	X				
	EPA 9045C		X	X		
Solids, Total	SM 2540 B	X				
Solids, Total Dissolved	SM 2540 C	X				

Solids, Total Suspended	SM 2540 D	X				
Sulfate	EPA 300.0	X				
	EPA 9056	X	X			

Appendix 5. Data Qualifiers

Qualifier Organic	Qualifier Inorganic	Footnote
U	U	Analyte analyzed for but was not detected.
J	B	Estimated result. Result is less than RL.
E	I	Estimated result. Result concentration exceeds the calibration range.
B	J	Method blank contamination. The associated method blank contains the target analyte at a reportable level.
P	*	Relative percent difference (RPD) is outside stated control limits.
a	N	Spiked analyte recovery is outside stated control limits.
*		Surrogate recovery is outside stated control limits.
PG		The percent difference between the original and confirmation analyses is greater than 40%.

Appendix C-3
TestAmerica Pittsburgh
Quality Assurance Manual

Quality Assurance Manual

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Title Page:

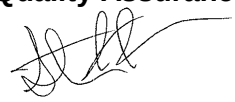
Quality Assurance Manual
Approval Signatures

6/16/2015

Laboratory Director - Deborah L. Lowe — Date

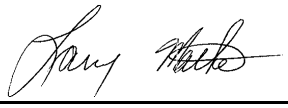
6/5/2015

Quality Assurance Manager – Virginia Zusman — Date



6/10/2015

Health and Safety Coordinator - Steve Jackson — Date



6/16/2015

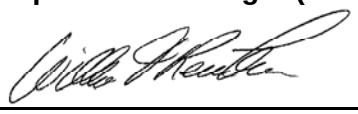
Technical Director - Larry Matko — Date

6/6/2015

Department Manager (Organics) - Sharon Bacha — Date

6/12/2015

Department Manager (Inorganics) - Roseann Ruyechan — Date



6/6/2015

Technical Supervisor (Metals) - Bill Reinheimer — Date

6/10/2015

Department Manager (Sample Management) - Chris Kovitch — Date

SECTION 2

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REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-Q-S-001	Acid & Solvent Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-006	Detection Limits
CA-Q-S-009	Root Cause Analysis
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process

SOP / Policy Reference	Title
CA-T-P-001	Qualified Products List
CW-F-S-007	Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual
CA-I-P-002	Electronic Reporting and Signature Policy

REFERENCED LABORATORY DOCUMENTS

SOP Reference	Title
PT-QA-001	Employee Orientation and Training (DOCs)
PT-QA-002	Internal Auditing
PT-QA-005	Measurement Uncertainty
PT-QA-006	Procurement of Standards and Materials; Labeling and Traceability
PT-QA-007	Detection Limits
PT-QA-008	Thermometer and Barometer Verification and Temperature Monitoring
PT-QA-010	Document and Spreadsheet Development & Control
PT-QA-012	Selection and Calibration of Balances and Weights
PT-QA-013	Independent QA Data Review
PT-QA-016	Nonconformance and Corrective Action System
PT-QA-017	Aqueous Pipette / Dispenser Calibration – Gravimetric Method
PT-QA-018	Technical Data Review Requirements
PT-QA-019	Records Management, Retention and Archive
PT-QA-020	Report Production
PT-QA-021	Laboratory Quality Control Program
PT-QA-022	Equipment Maintenance
PT-QA-024	Subsampling
PT-QA-WI-002	SOP List

SECTION 3
INTRODUCTION, SCOPE AND APPLICABILITY

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Pittsburgh's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E) (TNI). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP – CA-Q-M-002) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*
- *U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.*
- *Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, 22nd and on-line Editions.
- *U.S. Department of Energy* Order 414.1C, *Quality Assurance*, June 17, 2005.
- *U.S. Department of Energy*, *Quality Systems for Analytical Services*, Revision 3.6, November 2010.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP),* Version 4.0.02, May 2006.
- Nuclear Regulatory Commission (NRC) quality assurance requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management

policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among effluent water, groundwater, hazardous waste, sludge, soils and tissue. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in the Statement of Qualifications (SOQ) The current list of accredited methods is maintained in Total Access.The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.3.1 Specialty Analyses

3.3.1.1 Dredged Material Evaluations

TestAmerica Pittsburgh offers trace level testing of waters (site-waters and elutriates), sediments, and tissues in support of Dredged Material Evaluations for in-water (ocean and inland waters) and upland (Confined Disposal Facilities (CDFs), beneficial use, etc.) disposal options. In-house capabilities for commonly requested sediment program parameters include:

- Organochlorine Pesticides
- Organophosphorus Pesticides
- PCBs (as Aroclors and Congeners)
- Volatile Organics
- Semivolatile Organics
- Metals
- Cyanide
- Total Sulfides
- Acid Volatile Sulfide (AVS) and Simultaneously Extracted Metals (SEM)

- Nitrogen, Ammonia
- Nitrogen, Nitrate + Nitrite
- Biochemical Oxygen Demand (BOD)
- Chemical Oxygen Demand (COD)
- Total Organic Carbon (combustion procedure for sediments)
- Total Solids/Moisture Content
- Total Volatile Solids
- Lipids
    - With teaming arrangements with other TestAmerica facilities, additional sediment program capabilities include:
- Polychlorinated Dibenzo-Dioxins and Furans (PCDDs/PCDFs)
- Butyl Tins (mono – tetra)
- Total Kjeldahl Nitrogen
- Total Phosphorus
- Grain Size
- Specific Gravity
- Atterberg Limits

TestAmerica Pittsburgh also generates elutriate samples following appropriate U.S. Army Corps of Engineers procedures. These include:

- Standard Elutriate Test (SET) for in-water disposal evaluations, and
- Modified Elutriate Test (MET) or Effluent Elutriate Test (EET) for CDF disposal evaluations.
- Illinois Resuspension Tests (Supernatant and Elutriate Tests).
- Dredge Elutriate Test (DRET)

TestAmerica Pittsburgh currently supports dredge material evaluation projects following several state specific programs, as well as, under the following guidance documents:

- Ocean Testing Manual or OTM (USACE, 1991).
- New Jersey's Tidal Waters Technical Manual (NJDEP, 1997).
- Inland Testing Manual or ITM (USACE, 1998).
- Upland Testing Manual or UTM (USACE, 2003).

3.3.1.2 Tissue Analyses

TestAmerica Pittsburgh has extensive experience in supporting projects requiring tissue analyses. These include analyses of laboratory cultured reference species from bioaccumulation tests associated with dredged material evaluations to a variety of field collected species (aquatic and terrestrial). TestAmerica Pittsburgh has developed modifications to the standard solid methodologies (where possible) to allow for the use of smaller sample weights and achieve lower quantitation limits. In-house capabilities for commonly requested tissue parameters include:

- Organochlorine Pesticides
- PCBs (as Aroclors and Congeners)
- Semivolatile Organics
- Metals
- Lipids
- Moisture Content

With teaming arrangements with other TestAmerica facilities, additional tissue capabilities include:

- Polychlorinated Dibenzo-Dioxins and Furans (PCDDs/PCDFs)
- Butyl Tins (mono – tetra)

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed biannually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & Updating procedures (refer to SOP No. PT-QA-010, Preparation and Management of Standard Operating Procedures (SOPs) and Other Controlled Documents).

SECTION 4

MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Pittsburgh is a local operating unit of TestAmerica Laboratories, Inc.. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Executive Officer, Chief Financial Officer, Corporate Quality etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Pittsburgh is presented in Figure 4-1.

4.2 Roles And Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Pittsburgh laboratory.

4.2.2 Vice President of Operations (VPO)

Each VP of operations reports directly to the Executive VP of Operations and is part of the Executive Committee. Each VP of operations is responsible for the overall administrative and operational management of their respective laboratories. The VP's responsibilities include allocation of personnel and resources, long-term planning, goal setting, and achieving the financial, business, and quality objectives of TestAmerica. The VP's ensure timely compliance with Corporate Management directives, policies, and management systems reviews. The VP's are also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

4.2.3 Laboratory Director

Pittsburgh's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program. The Laboratory Director can also serve as the Technical Manager.

Specific responsibilities include, but are not limited to:

- Providing one or more technical managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensuring that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensuring that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensuring TestAmerica's human resource policies are adhered to and maintained.
- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensuring that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviewing and approving all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursuing and maintaining appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensuring client specific reporting and quality control requirements are met.
- Captaining the management team, consisting of the QA Manager, the Technical Managers and the Department Managers.
- Monitoring the validity of the analyses performed and data generated in the laboratory.
- Providing training and development programs to applicable laboratory staff as new hires and, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- The Technical Manager meets the requirements specified in the Section 5.2.6.1 of the TNI standards.

4.2.4 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system. The QA Manager reports directly to the Laboratory Director and their Corporate Quality Director. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA Specialists to accomplish specific responsibilities, which include, but are not limited to:

- Serving as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.

- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintaining records of all ethics-related training, including the type and proof of attendance.
- Maintaining improving, and evaluating the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Having the responsibility and final authority to accept or reject data and to stop work in progress in the event that procedures and practices compromise the validity and integrity of analytical data.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information. Controlling distribution of controlled documents.
- Reviewing a percentage of all final data reports for internal consistency, including Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Reviewing of external audit reports and data validation requests.
- Following-up with audits to ensure client QAPP requirements are met.
- Establishing reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Approving quality control reference data in TALS.
- Developing suggestions and recommendations to improve quality systems.
- Researching current state and federal requirements and guidelines.
- Captaining the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluating the thoroughness and effectiveness of training.
- Ensuring Compliance with ISO 17025.

4.2.5 Technical Director

The Technical Director serves as a technical resource for TestAmerica's personnel and clients in their field of expertise. The Technical Director reports directly to the Laboratory Director. Specific responsibilities include, but are not limited to:

- Managing technical projects and evaluating technologies, reviewing technical data
- Solving technical problems in the laboratory including troubleshooting instruments and developing or modifying methods as needed to meet customer requirements.
- Maintaining and repairing analytical instruments to reduce downtime.
- Consulting with clients, regulators, and others regarding technical aspects of analyses.
- Suggesting and implementing process improvements to maximize productivity, save costs, and decrease turn-around time.
- Participating in TestAmerica's best practice process to spread best technical practices and developing TestAmerica Standard Operating Procedures (SOPs). Leads the implementation and follow-up of the best practices and SOPs in the laboratory.
- Evaluating and adapting new technologies and methodologies. Performs non-routine analysis as required to meet the needs of current long-term clients or as a means to capture new clients in support of business development efforts.
- Training analysts and technicians in area of expertise.
- Assisting with the development of health and safety protocols.
- Consulting with Project Managers and sales staff regarding analytical techniques and capabilities.
- Investigating issues raised by clients, QA, sales, and other departments to find root cause and implement corrective action and proper response.
- Contributing technical information and evaluation for deciding major new equipment purchases and capital expenditures.
- Ensuring compliance with ISO 17025.

4.2.6 Quality Assurance Specialist

The QA Specialist is responsible for QA documentation and involvement in the following activities:

- Assisting the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- Facilitating external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.
- Assisting the QA Manager in the preparation of new SOPs and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- Managing the performance testing (PT) studies, coordinates follow up studies for failed analytes and works with QA Manager and Laboratory Staff to complete needed corrective action reports.
- Assisting with review and maintenance of training records.
- Assisting the Quality Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- Assisting with management of and applications for certifications and accreditations.

- Monitoring for compliance the following QA Metrics: temperature monitoring of refrigeration units and incubators; thermometer calibrations; balance calibrations; eppendorf/pipette calibrations; and proper standard/reagent storage.
- Performing Technical Data Audits and the Mint-miner data file review process for organic instrumentation. Maintain tracking of reviews.
- Assisting with technical review of data packages which require QA review.

4.2.7 Technical Manager or Designee

The Technical Manager(s) report(s) directly to the Laboratory Director. The scope of responsibility ranges from the new-hire training and existing technology through the ongoing training and development programs for existing analysts and new instrumentation and for compliance with the ISO 17025 Standard. Specific responsibilities include, but are not limited to:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc..

- Captaining department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinating audit responses with the QA Manager.
- Ensuring compliance with ISO 17025.

4.2.8 Client Service Manager (CSM)

The Client Services Manager reports directly to the Client Services Director with dotted line reporting to the Laboratory Director. He/She has signature authority for contracts for laboratory services, as detailed in TestAmerica policy, and for laboratory reports. The responsibilities of the CSM include, but are not limited to:

- Defining customer requirements through project definition.
- Assessing and assuring customer satisfaction.
- Providing feedback to management on changing customer needs.
- Bringing together resources necessary to ensure customer satisfaction.

4.2.9 Manger of Project Management

The Manager of Project Management reports directly to the Client Services Director with dotted line reporting to the Laboratory Director. There is an entire staff of Project Managers that makes up the Project Management team. With the overall goal of total client satisfaction. In addition to the responsibilities of the Project Manager, listed in section 4.2.10, the MPM's responsibilities include, but are not limited to:

- Training project managers in technical procedures and promoting the growth of the Project Management Team.
- Acting as liaison between laboratory management and the Project Management Team.
- Managing human resources for the Project Management Team.

4.2.10 Project Manager

The PM reports to the Manager of Project Management and serves as the interface between the laboratory's technical departments and the laboratory's clients. The responsibilities of this position include, but are not limited to:

- Ensuring that clients receive the proper sampling supplies.
- Responding to client inquiries concerning sample status.
- Assisting clients with the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Maintaining communication with clients on sample progress from daily status meeting with agreed-upon due dates.
- Discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.

- Familiarizing laboratory staff with specific quotes, sample log-in review, and final report completeness.
- Informing QA Manager of special client requests that are outside of standard operating procedure.
- Monitoring the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Informing clients of data package-related problems and resolve service issues.
- Coordinating requests for sample containers and other services.

4.2.11 Project Manager Assistant (PMA)

The PMA reports to the Manager of Project Management and serves as the interface between the laboratory's technical departments and the laboratory's clients. The responsibilities of this position include, but are not limited to:

- Collating data reports, expanded deliverables and CLP data packages for delivery to clients and reviews for accuracy.
- Assisting the CSMs and PMs in the reporting process.
- Printing reports as needed for Project Managers.
- Monitoring report due dates for timely delivery.
- Providing clerical support to the CSMs, PMs and other laboratory staff as needed
- Generating credit or debit invoices to ensure proper payment in compliance with client requirements as established and communicated.
- Sending final data to clients via email or courier.

4.2.12 Department Manager

The Organics Manager oversees the GC and GCMS and Organic Preparation groups. The Inorganics Manager oversees the Metals and Wet Chemistry groups. Department Managers report directly to the Laboratory Director or designee. The responsibilities of a Department Manager include, but are not limited to:

- Ensuring that analysts in their department adhere to applicable SOPs and the QA Manual.
- Performing frequent SOP review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents, and approving revised SOPs.
- Participating in the interview and selection of, and overseeing, training, development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documenting these activities in accordance with systems developed by the QA and Personnel Departments. Evaluating staffing sufficiency and overtime needs.
- Ensuring sufficient training of analysts to meet the requirements of the SOP and the QA system.
- Encouraging the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Providing guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Managers and/or QA Manager. Each is

responsible ensuring 100% implementation of the data review and documentation, non-conformance and corrective action issues, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.

- Ensuring all logbooks are maintained, current, and properly labeled or archived.
- Reporting all non-conformance conditions to the QA Manager, Technical Managers and/or Laboratory Director.
- Ensuring that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/she is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintaining adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Determining ways to achieve optimum turnaround time on analyses and compliance with holding times.
- Conducting efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Assisting QA department with root cause investigations and corrective action proposals for responses to external and internal audit issues, system failures and client complaints.

4.2.13 Team Leader/Supervisor

The Team Leader/Supervisor reports directly to the Organics or Inorganics Manager and/or Laboratory Director or designee. The responsibilities of this position include, but are not limited to:

- Ensuring that analysts in their department adhere to applicable SOPs and the QA Manual. He/she performs frequent SOP review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- Overseeing training, development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments.
- Providing guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager(s) and/or QA Manager. Each is responsible ensuring 100% implementation of the data review and documentation, non-conformance and corrective action issues, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.
- Ensuring that all logbooks are maintained, current, and properly labeled or archived.
- Ensuring that all data is properly entered into the LIMS system and is reviewed and approved as required by laboratory documentation policy.
- Reporting all non-conformance conditions to the QA Manager and Department Manager.
- Ensuring that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/she is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintaining adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.

- Achieving optimum turnaround time on analyses and compliance with holding times.
- Assisting QA department with root cause investigations and corrective action proposals for responses to external and internal audit issues, system failures and client complaints.

4.2.14 Laboratory Analyst

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the team leader or supervisor. The responsibilities of the analysts include, but are not limited to:

- Performing analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Ensuring sample and data integrity by adhering to internal chain-of-custody procedures.
- Documenting standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on bench sheets, lab notebooks, run logs, and/or the Non-Conformance Database.
- Reporting all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, Department Manager, and/or the QA Manager or member of QA staff.
- Performing 100% review of the data generated prior to entering and submitting for secondary level review. Performs data processing using available tools/software.
- Suggesting method improvements to their supervisor, the Technical Manager (s), and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Working cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.
    - A “work cell” is considered to be all those individuals who see a sample through the complete process of preparation, extraction, and analysis. To ensure that the entire preparation, extraction, and analysis process is completed by a group of capable individuals, the laboratory shall ensure that each member of the work cell (including a new member entering an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. Even though the work cell operates as a “team,” the demonstration of capability at each individual step in the sequence, as performed by each individual analyst/team member, remains of utmost importance. A work cell may NOT be defined as a group of analysts who perform the same step in the same process (for example, extractions for Method 8270), represented by one analyst who has demonstrated capability for that step.

4.2.15 Sample Management/Log-in Manager

The Sample Receiving/Login Manager reports to the Laboratory Director and Client Services Manager. The responsibilities of this position include, but are not limited to:

- Ensuring implementation of proper sample receipt procedures, including maintenance of chain-of-custody.
- Reporting nonconformances associated with condition-upon-receipt of samples.
- Ensuring accurate login of samples into the LIMS.

- Ensuring that all samples are stored in the proper environment.
- Assisting Environmental Health and Safety staff with sample disposal.

4.2.16 Field Service Technician

The Field Service Technicians report to the Sample Management Department Manager. The responsibilities of the Field Service Technician include, but are not limited to:

- Performing sample collection and sample pick-up
- Ensuring sample containers are prepared for sampling
- Performing field tests and measurements and operating and maintaining equipment used for those purposes.

4.2.17 Environmental Health and Safety Coordinator

The Health and Safety Coordinator reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. The EH&S Coordinator :

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica’s medical consultants.

4.2.18 Hazardous Waste Coordinator

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties include, but are not limited to:

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

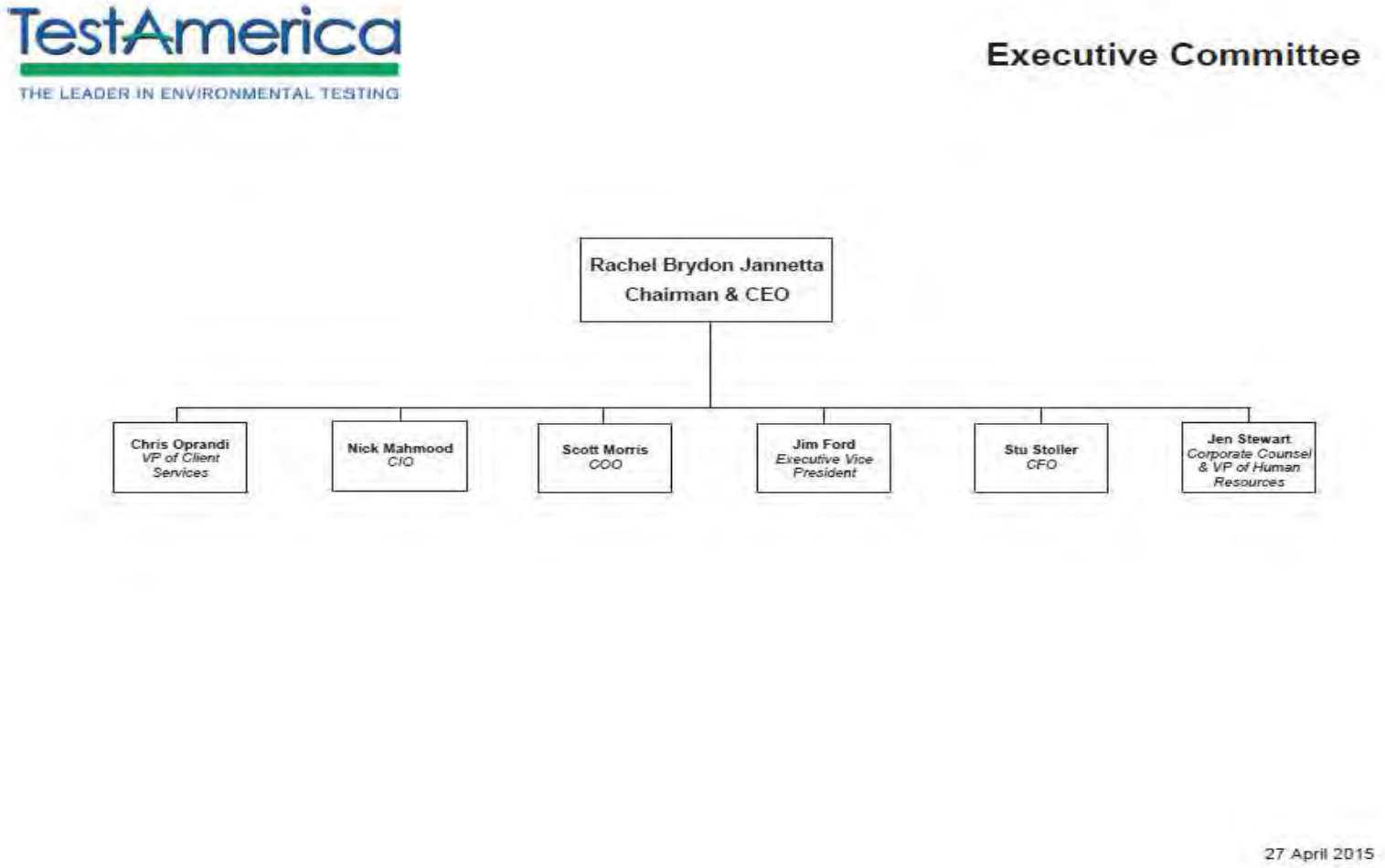
4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy	Comment
Laboratory Director: Deborah Lowe	Project Technical Manager – Dave Dunlap	NELAP Technical Manager (entire laboratory)
Quality Assurance Manager: Virginia Zusman	Quality Assurance Specialist: Pam Dudeck	
Technical Manager: Larry Matko	Laboratory Director: Deborah Lowe	NELAP Technical Manager (Lipids & 8141)
Organics Department Manager: Sharon Bacha	Designated Senior GC and GCMS Analyst	NELAP Technical Manager (Organics)
Inorganics Department Manager: Roseann Ruyechan	Designated Metals and Wet Chemistry Supervisors	
Metals Supervisor: Bill Reinheimer	Designated Senior Metals Analyst	NELAP Technical Manager (Inorganics)
Wet Chemistry Supervisor: Mike Wesoloski	Designated Senior Wet Chemistry Analyst	NELAP Technical Manager (Inorganics, Non-metals)
Organic Prep Team Leader: Sharon Bacha/Larry Matko	Designated Senior Organic Prep Analyst	
Sample Receiving Department Manager: Christina Kovitch	Lab Director or Designated person in the group	

If the NELAP Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.

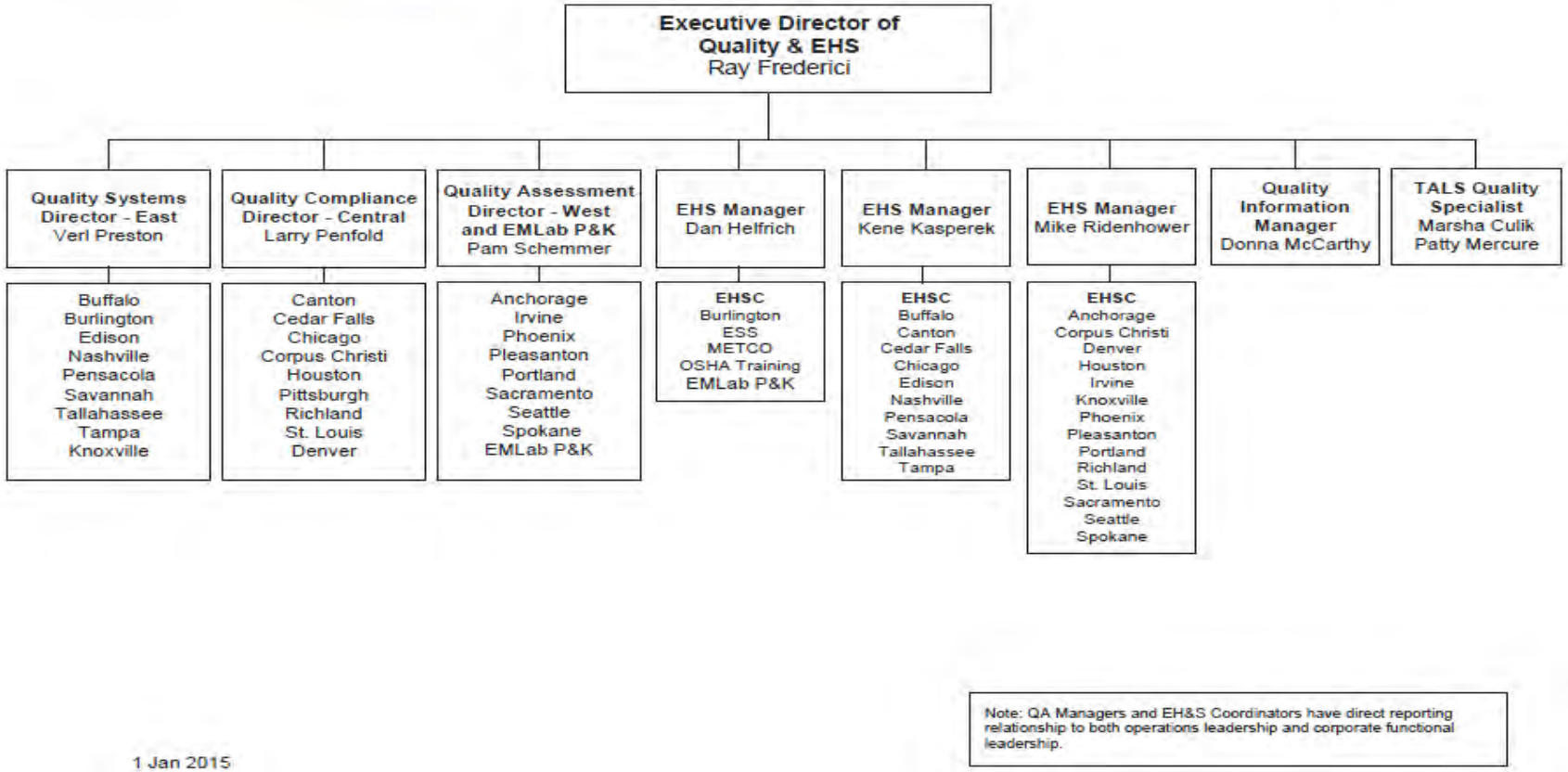
Figure 4-1. Corporate and Laboratory Organization Charts



Note: Organization Charts are subject to change, contact the laboratory for the most recent version

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

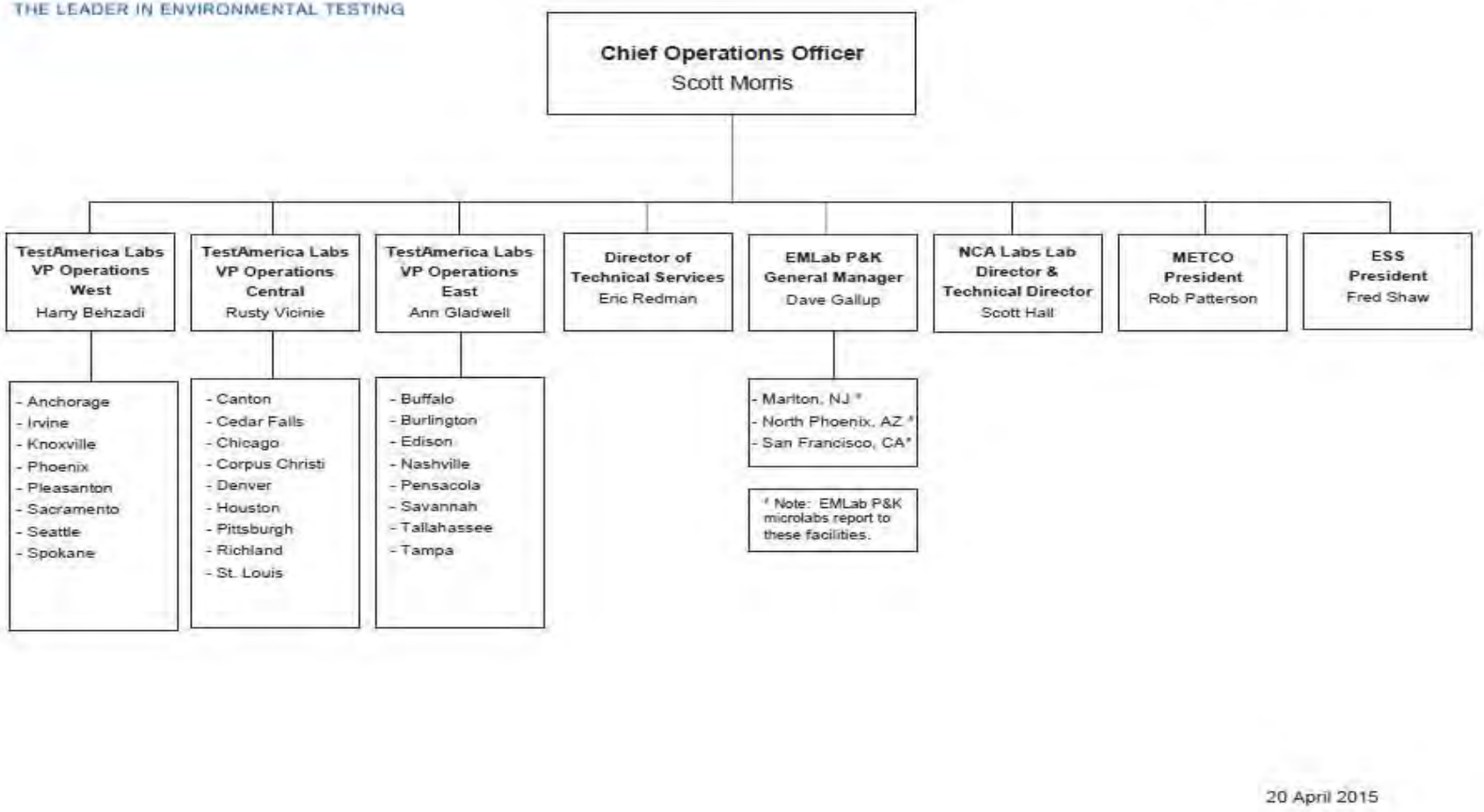
Quality & EHS



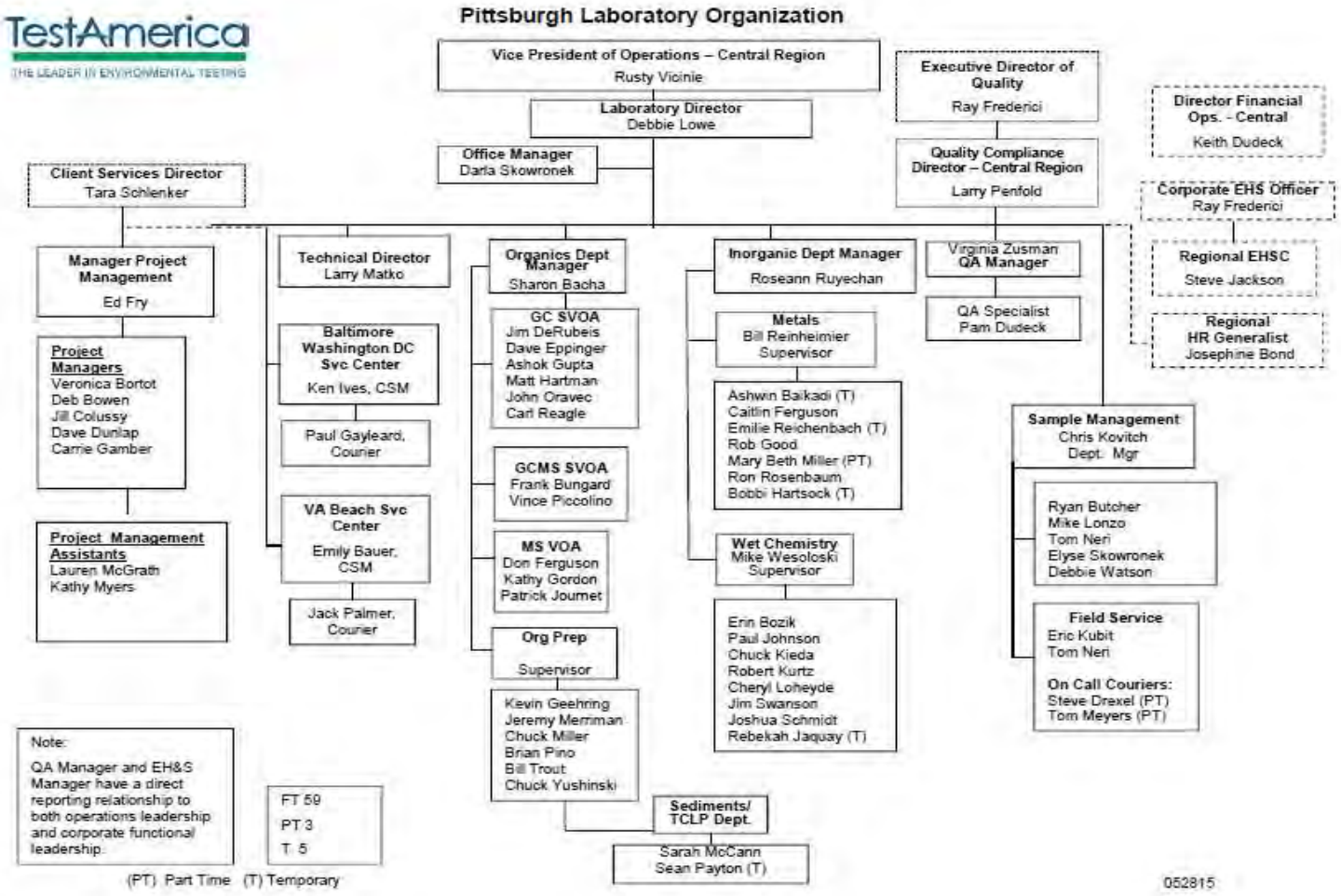
Note: Organization Charts are subject to change, contact the laboratory for the most recent version

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Operations



Note: Organization Charts are subject to change, contact the laboratory for the most recent version



Note: Organization Charts are subject to change, contact the laboratory for the most recent version

SECTION 5

QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.
- To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 Ethics And Data Integrity

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements
- Ethics and Compliance Officers (ECOs)
- A Training Program
- Self-governance through disciplinary action for violations
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-L-S-002)
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15)
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs)
- Present services in a confidential, honest and forthright manner
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public

- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same
- Educate clients as to the extent and kinds of services available
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them

5.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC Objectives For The Measurement Of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 Criteria For Quality Indicators

The laboratory maintains a Quality Control Limit Summary (from LIMS) that contains tables that summarizes the precision and accuracy acceptability limits for analyses performed at TestAmerica Pittsburgh. This summary includes an activation date, is updated each time new limits are generated and is located in the LIMS. Current limits are controlled through the LIMS. The limits in effect for a given date are archived in the LIMS with the associated sample data. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in Section 24.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the area Technical Manager/supervisor and QA Manager) as entered into the Laboratory Information Management

System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. These limits are maintained in the LIMS as part of the analytical historical record. If a method defines the QC limits, the method limits are used. For further details refer to SOP No. PT-QA-021.

If a method defines the QC limits, the method limits are used, unless laboratory developed limits are tighter.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

The generation and use of QC Charts (Control Charts) are described in the laboratory SOP PT-QA-021, Laboratory Quality Control Program.

5.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

5.8 Laboratory Certification/Accreditation

The Laboratory Quality System is designed to meet the requirements of all governing bodies through which it holds certification / accreditation.

- A list of certifications and accredited scopes is maintained by the QA Department, and current certificates are posted in the laboratory lobby. Expired certificates are maintained in the QA archive.
- Certification renewal is completed on an annual basis for most state agencies.
- The laboratory indicates clearly in its reports which certifications it holds. This list can include which test methods and analytes are not covered under the applicable certification.
- If certification is lost or suspended, the laboratory shall update the reporting information to reflect this immediately.

SECTION 6

DOCUMENT CONTROL

6.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Logbooks and Calculation Spreadsheets
- Corporate Policies and Procedures distributed outside the intranet
- External documents that are used as part of the laboratory's Quality System

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. PT-QA-010.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports and Nonconformance Memos (NCMs). Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 Document Approval And Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or and 'end of document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a technical manager/supervisor submits an electronic or paper draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain that document as the official document on file. That document is then provided to all applicable operational units (may

include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures For Document Control Policy

For changes to the QA Manual, refer to SOP No. PT-QA-010. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the laboratory SharePoint website by lab area as pdf. Editable copies are stored on a restricted access drive. Uncontrolled, editable copies are issued as drafts for review and revision, and are then stored on the restricted access drive.

For changes to SOPs and QA manual, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP and laboratory SOP PT-QA-010. The SOP identified above also defines the process of revising SOPs.

Controlled documents are marked as such, and posted to the intranet (OASIS SharePoint site) by the QA department. Controlled distribution is achieved electronically. Details of the numbering system, required format, and controlled distribution of documents are described in SOP No. PT-QA-010, “Preparation and Management of Standard Operating Procedures (SOPs).

Forms, worksheets, work instructions and information are organized by department by the QA office. Electronic versions are kept on a hard drive in the QA department; hard copies can be printed out as needed. All forms used in the laboratory are tracked in the controlled documents database which can be accessed by the QA department and the IT group. The procedure for the care of these documents is in SOP No. PT-QA-010, “Document and Spreadsheet Development and Control”.

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. PT-QA-019.

SECTION 7

SERVICE TO THE CLIENT

7.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.
All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence And Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director if applicable
- Customer Service Manager
- The Laboratory Project Management
- The Laboratory Director Technical Manager
- Laboratory Quality Assurance Manager
- PM or CSM reviews the formal laboratory quote. The Laboratory Director makes final acceptance for their facility.

The Sales Director, Legal Contracts Director, Account Executive or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts**.** In Pittsburgh laboratory copies of contracts are maintained in the laboratory network public drive (L:\Weekly\Quotes_Scanned) by the sales/marketing personnel.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Contracts review documentation is forwarded to the Human Resources Coordinator and is maintained in the network public drive.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM and the Lab Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log or electronic mail of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during operations meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 25).

Note: ISO/IEC 17025 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 Client Communication

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical or Department Managers, or their designees, are available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality, client service and testing activities. Both complaints and compliments are tracked in the monthly quality status report.

TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8

SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOPs on Subcontracting Procedures (CA-L-S-002).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client’s Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client’s analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), Account Executives (AE) or designee for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

Note: In addition to the client, some regulating agencies, (e.g, USDA) or contracts (e.g, certain USACE projects) may require notification prior to placing such work**.**

For DOD projects the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories are evaluated following the procedures outlined below and as seen in Figure 8-1. The subcontractor laboratory must receive project-specific approval from the DoD client before any samples are analyzed.

The QSM has 5 specific requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.
2. Subcontractor laboratories must be approved by the specific DoD Component laboratory approval process.
3. Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of PT samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.

4. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
5. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives

8.2 Qualifying And Monitoring Subcontractors

Whenever a PM, Account Executive (AE) or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable, (e.g., on the subcontractors TNI, A2LA accreditation or State Certification).
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- TNI or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

When the potential sub-contract laboratory has not been previously approved, Account Executives, CSMs or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site and notify the finance group for JD Edwards.

8.2.2 The client will assume responsibility for the quality of the data generated from the

use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Laboratory Directors/Managers, QA Managers and Sales Personnel.

8.3 Oversight And Reporting

The PM or CSM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or AE or CSM) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on the project folder or scanned into LIMS. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples workshared within TestAmerica. Client CoCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1

Example - Subcontracted Sample Form

Date/Time: _____

Subcontracted Laboratory Information:

- Subcontractor's Name: _____
- Subcontractor Point of Contact: _____
- Subcontractor's Address: _____
- Subcontractor's Phone: _____
- Analyte/Method: _____
- Certified for State of Origin: _____
- TNI Certified: Yes_____No_____
- **USDA Permit (__Domestic __ Foreign)** **Yes**_____**No**_____
- A2LA (or ISO 17025) Certified: Yes_____No_____
- CLP-like Required: Yes_____No_____
  (Full doc required)
- Requested Sample Due Date: _____
  (Must be put on COC)
- **Client POC Approval on-file to Subcontract Samples to Sub Laboratory:** **Yes**_____**No**_____

Project Manager: _____

Laboratory Sample # Range: _____
(Only of Subcontracted Samples)

Laboratory Project Number (Billing Control #): _____

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

PM Signature_____**Date**_____

SECTION 9

PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001 and TestAmerica Pittsburgh SOP No. PT-QA-006, Procurement of Standards and Materials.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst completes the Purchase Requisition Form (Figure 9-1) when requesting reagents, standards, or supplies***:*** The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use. If an item is not in the consignment system, the analyst must obtain approval from the area team leader/supervisor and Laboratory Director prior to placing the order. All the

orders are submitted to the Department Managers by completing the Purchase Requisition Form (Figure 9-1). The Department Manager will enter the orders into the JD Edwards system (JDE). The Department Manager or designated personnel also places the orders for rush items and obtains purchase orders for instrument/equipment repairs and maintenance. The laboratory Director will approve or deny the order in the JDE. Every order is given a purchase order number in the JDE. The actual order to the vendor is placed through the purchasing department in the TestAmerica North Canton Laboratory.

9.3.2 Receiving

It is the responsibility of the Sample Receiving department to receive the shipment. It is the responsibility of the analyst or laboratory area designee who ordered the materials to document the date the materials were received. Once the ordered reagents or materials are received, the analyst or designee compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. The analyst or designee dates and initials the packing slip and returns it to Sample Receiving for filing. Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOPs expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date cannot be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.

- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained with each lab department and copy forwarded to QA office.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. To prevent a tank from going to dryness, close observation of the tank gauge must take place as pressure decreases towards 500psig, or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- µmho /cm (or specific resistivity of greater than 1.0 megohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers/Supervisors must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

NOTE: Each bottleware type must be documented as clean down to the laboratory MDL for all target analytes for use with samples from Wisconsin.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager (s) or QA Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 Purchase Of Equipment/Instruments/Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Department Manager, Technical Manager (s) and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products

List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained in the laboratory in a designated area or near the instrument.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Laboratory Technical Manager / Director.

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form available on OASIS.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

Figure 9-1

Example - Purchase Requisition Form

Date:	**For Purchasing Use Only**
Vendor Name:	**Order Date:**
Exact Date Needed:	**Account Number:**
Requested By:	**Order Number:**
Department Name/Number:	**P.O. Number:**

Item	Quantity	Unit of Measure	Catalog No.	Description	Unit Cost	Total Cost
1.						
2.						
3.						
4.						
5.						
6.						
7.						
8.						

Authorized Signature Date

SECTION 10

COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following the Non Conformance and Corrective Action System, SOP No. PT-QA-016. It is the laboratory's goal to provide a satisfactory resolution to complaints in a timely and professional manner.

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint in the database, according to (SOP No. PT-QA-016).

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the laboratory and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

SECTION 11

CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for advice. The supervisor may elect to discuss it with the Laboratory Director or QA Manager or have a PM contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Laboratory Director and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

11.2 Responsibilities And Authorities

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CW-L-S-002), outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc.. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information is documented on a Nonconformance Memo (NCM) and may also be documented in logbooks

and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Executive Director of Quality & EHS and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, the COO, General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation Of Significance And Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CW-L-S-002) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

11.4 Prevention Of Nonconforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, or on a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process must be followed.

11.5 Method Suspension/Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate VP of Operations and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

SECTION 12

CORRECTIVE ACTION

12.1 Overview

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memos (NCM) in LIMS (Figure 12-1) or the Corrective Action Reports (CAR) using the corrective action database (Figures 12-2 and 12-3).

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc..

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client Complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips
- Anomalies that occur during sample receipt, preparation or analysis

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs
- Internal and external audit findings
- Unacceptable PT results
- Corrective actions that cross multiple departments in the laboratory
- Systematic reporting / calculation errors.
- Client complaints
- Data recall investigations

- Identified poor process or method performance trends
- Excessive revised reports

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM, CAR or the documentation in the complaint database must be initiated. Someone is assigned to investigate the issue and the event is investigated for root cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the root cause is not readily obvious, the Supervisor, Laboratory Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or CAR is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed;

and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and CAR is entered into a database for tracking purposes and a monthly summary of all NCMs is reviewed to aid in ensuring that the appropriate corrective actions have taken effect. CARs are also compiled and reviewed monthly. Corrective actions or complaints that result in corrective action are also reviewed monthly.
- The QA Manager reviews NCMs and CARs monthly for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 12-1

Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Technical Manager(s))*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Technical Manager(s))*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in LIMS.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in LIMS,	- Batch must be re-prepared and re-analyzed. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), generally with low bias samples are reprepared and reanalzyed.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1] For common lab contaminants, no analytes detected at greater than and equal to RL.	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit and is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples *(QA Manager, Technical Manager(s))*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Internal / External Audits *(QA Manager, Technical Manager(s), Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, *Technical Manager(s)*, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002.
Client Complaints *(Project Managers, Lab Director, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director, *Technical Manager(s)*)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director, *Technical Manager(s)*)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:

1. Except as noted below for certain compounds, the method blank should be below the reporting limit unless there is a client specific requirement. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

SECTION 13

PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process if improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management system reviews, review of the monthly QA Metrics Report, evaluation of internal or external audits, results and evaluation of proficiency testing (PT) performance, review of control charts and QC results, data analysis & review processing operations, client complaints, staff observation, etc..

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lesson Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and nonconformances provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.

- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/ Process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any Preventive Actions/ Process Improvements undertaken or attempted shall be taken into account during the Annual Management Systems Review (Section 16). A highly detailed report is not required; however a summary of success and failure within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management Of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of indicators monitored under this collective system include:

Change Type	Examples
Facility Changes	-movement of prep or instrument groups to a new location in the laboratory -introduction of significant changes in air handling or gas and solvent delivery systems -significant room additions or renovations -significant electrical or network upgrades or changes
Accreditation Changes	-voluntary surrender of accreditations no longer deemed necessary to the laboratory -loss of accreditation -addition of new accreditation programs
Reagents and Waste Streams	- new chemicals/reagents not previously used in the laboratory -deletion of chemicals/reagents that will mean they are no longer used at all in the laboratory -major changes to the volume of chemicals/reagents being used - a new waste stream must be developed
Addition or Deletion of Laboratory Capabilities	-implementation of new regulated methods -“retiring” of active methods -method development for “in-house” methods Note: New regulatory methods and method development require specific processes and documentation before the process can begin or the method can enter production. See QA and EHS for requirements.
Key Personnel Changes	-key personnel promotions and their effect on that individuals group (experience, productivity, leadership, manpower) -key personnel losses -impact of new personnel that may add new experience or capabilities to the laboratory
New Types of Instrumentation	-addition of a new instrument class/technology -significant instrument upgrades that impact sensitivity, productivity or capability Note: New instrumentation requires collection and submission of instrument IDOC information before entering production. See QA for requirements.
Changes in Quality Systems and Policies	-implementation of a new Corrective Action System -changes to the Internal Audit program -implementation of uploads for Proficiency Testing samples

SECTION 14

CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the Quality Assurance (QA) department electronically in laboratory's designated network drive which is backed up as part of the regular network backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by report production group, HR and the QA department and as outlined in SOP No. PT-QA-019.

Table 14-1 Record Index[1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals	5 Years from document retirement date*
QA Records	- Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP -SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*

	Record Types [1]:	Retention Time:
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits	7 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies Technical Training Records	7 years

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss at the laboratory or the Business Records Management Facility. Depending on the type of report requested, the onsite retention of laboratory data records varies. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether off-site storage is used, logs are maintained to note removal and return of records. All data records are uploaded into LIMS and maintained in LIMS. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 14-2. Special Record Retention Requirements

Program	[1]Retention Requirement
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to SOP No. PT-QA-019, Records Information Management and SOP No. PT-QA-020, Report Production.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (Records stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored with the invoice in LIMS. Details of this procedure is described in SOP No. PT-QA-019. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with the chain of custody, they are scanned into LIMS.
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set are described in SOP No. PT-QA-019. Instrument data is stored sequentially by instrument. Run logs are maintained for

each instrument; a copy of each day's run long or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data or the data is entered in LIMS. Standard and reagent information is recorded in electronic standard log in LIMS.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP No. PT-QA-019.
- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'**.**

14.2 Technical And Analytical Records

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code**;**
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook, on a benchsheet or in LIMS.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**

- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents**;**
- test results**;**
- standard and reagent origin, receipt, preparation, and use**;**
- calibration criteria, frequency and acceptance criteria**;**
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions**;**
- quality control protocols and assessment**;**
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats**.**

14.3 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)**;**
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value**;**
- copies of final reports**;**
- archived SOPs**;**
- correspondence relating to laboratory activities for a specific project**;**
- all corrective action reports, audits and audit responses**;**
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage And Disposal

14.5.1 All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

14.5.2 All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

14.5.3 Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

14.5.4 The laboratory has a record management system (a.k.a., document control) for control of laboratory instrument/run logbooks, standard logbooks, balance logs, maintenance logs, bench sheets where applicable and records for data reduction, validation and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All sample data are recorded in LIMS. Bench sheets are filed sequentially. Standards are maintained in the electronic standards in LIMS**.** Records are considered archived when noted as such in the records management system (a.k.a., document control).

14.5.5 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.6 Records Disposal

14.5.6.1 Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2 and SOP No. PT-QA-019).

14.5.6.2 Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

14.5.6.3 If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15

AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CW-Q-S-003. More detail on the specific elements for internal audits and data audit is described in Pittsburgh Laboratory's SOP No. PT-QA-002, and SOP No. PT-QA-013. Technical data review requirement are described in Section 19.14.4 and SOP No. PT-QA-018. The types and frequency of routine internal audits are shown in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1 Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CW-Q-S-003)	50% of methods annually
SOP Method Compliance	Joint responsibility: c) QA Manager or designee d) Technical Manager or Designee (Refer to CW-Q-S-003)	Every 2 years
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client, state

requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

Effectiveness of training will be determined during our annual QA systems evaluation. Evidence of successful training includes:

- 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 and continually improve the quality system:
- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge of QA Manual and SOPs. Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., MintMiner and Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Water Pollution Program, Water Supply Program, Hazardous Waste Program, client supplied PTs and Lab internal PTs.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process database or spreadsheet. The laboratory's corrective action responses for both types of audits may include action plans

that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.
Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within one business day of discovery of the problem and the client will be notified as soon as the impact is known but no later than two weeks after identification of the problem.

SECTION 16

MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Manager(s), their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, QA Manager, Technical Director, and Department Managers) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals & objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel is to be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management system review (Corporate SOP No. CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources.
    - Adequacy of policies and procedures.
    - Future plans for resources and testing capability and capacity.

- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.
- Review of Corrective and Preventative Actions, assessments by external bodies and recommendations for improvement.

A report is generated by the QA Manager and management. The report is distributed to the appropriate VP of Operation and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Quality Directors receive a monthly report from the Executive Director of Quality & EHS summarizing any current data integrity or data recall investigations. The VP's of Operation are also made aware of progress on these issues for their specific labs.

SECTION 17

PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education And Experience Requirements For Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, pipette or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Manager (s) – **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Manager (s) – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience
Technical Managers - **Microbiology**	Bachelor's degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.

- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP No. PT-QA-001.

17.4 Data Integrity And Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 33,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. Systems are controlled and monitored to assure constant and consistent test conditions.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical analysis areas, including sample preparation.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys and alarm codes are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19

TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory. A SOP list is included in Appendix 4. The most current list of SOPs is maintained in the QA SOP directory in PT-QA-W-002.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or Pittsburgh SOP No. PT-QA-010, Document and Spreadsheet Development and Control.
- SOPs are reviewed at a minimum of every 2 years, and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection Of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40CFR Part 136 as amended by Method Update Rule; May 18, 2012*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Statement of Work for Organics Analysis, OLM04.2, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th /on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*

- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

19.4.2.1 A demonstration of capability (DOC, Lab SOP # PT-QA-001) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel (e.g., analyst hasn't performed the test within the last 12 months). The IDOC must meet the control limits specified in the reference method, if any, or meet method LCS criteria if no IDOC specific controls are provided.

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

19.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Technical Director or Lab Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

19.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Initial Demonstration and Capability (IDOC) procedure is described in Pittsburgh SOP No. PT-QA-001.

A certification statement (Figure 19-1) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 Laboratory Developed Methods And Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation Of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Validation of a method is a planned activity. A coordinator is designated for the process, who's responsibility it is to communicate the process and progress to all involved personnel.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL)/ Limits Of Detection (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. All instruments used for analysis of client samples are included in the MDL study and verification. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. [To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used]
Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. PT-QA-007 for details on the laboratory's MDL process.

19.8 Instrument Detection Limits (IDL)

19.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation. If IDL is > than the MDL, it may be used as the reported MDL.

19.9 Verification Of Detection And Reporting Limits

19.9.1 Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Refer to the laboratory SOP PT-QA-007 Detection Limits (MDLs/DLs) for further details.

19.9.2 When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit and annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

19.9.3 The laboratory quantitation limit is equivalent to the Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

19.11 Evaluation Of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.12 Estimation Of Uncertainty Of Measurement

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of

an “expanded uncertainty”: the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k =3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L. Uncertainty determination is further described in SOP No. PT-QA-005.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as ‘reanalysis’) may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client’s request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supercede the following items.**

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client’s request, both results may be reported.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Technical Manager/Supervisor or Laboratory Director if unsure.

19.14 Control Of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the TALs LIMS which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Microsoft SQL Server, which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 Maintain the Database Integrity: Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.14.1.2 Ensure Information Availability: Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls such as password protection or website access approval, when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets,

or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s). The applicable data/spreadsheet is scanned in LIMS with the batch.

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, Acceptable Manual Integration Practices.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data must be retained, including computer file (if appropriate), and/or run log. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1**%.** Units are defined in each lab SOP.

19.14.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.14.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored on the server and every night backed up to a tape file.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the section Supervisor/ or Department Manager and QA Manager at the facility.
  - Any cells that perform calculations must have the calculation verified and the cell locked so that the formula cannot be changed.
  - The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Technical data review procedures are out lined in Pittsburgh SOP No. PT-QA-018 to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory uses the Corporate SOP No. CA-Q-S-002, Acceptable Manual Integration Practices, discussing Manual Integrations to ensure the authenticity of the data. The general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.14.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, documentation of standards and reagents, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.14.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial

calibration/calibration verification data in the day's analytical run, evaluation of QC data, documentation of standards and reagents, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.14.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager, Technical Director, Department Manager or section Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.14.4.5 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.14.4.6 As a final review prior to the release of the report, the Project Manager (or designee) reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.14.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager (or designee) then signs the final report. They also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.4.8 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of

data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline.

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1
Example - Demonstration of Capability Documentation

DEMONSTRATION OF CAPABILITIY (DOC)

Laboratory Name:_____
Laboratory Address:_____
Method:_____ Matrix:_____
Date:_____ Analyst(s):_____
Source of Analyte(s):_____

Analytical Results

Analyst	Conc. (Units)	Rep 1	Rep 2	Rep 3	Rep 4	Avg. % Recovery	% RSD
_____	_____	_____	_____	_____	_____	_____	_____

% RSD = Percent relative standard deviation = standard deviation divided by average % Recovery

Raw data reference: _____

Certification Statement:

We, the undersigned, certify that:
1. The cited test method has met Demonstration of Capability requirements.
2. The test method was performed by the analyst(s) identified on this certification.
3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on site.
4. The data associated with the method demonstration of capability are true, accurate, complete, and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility, and the associated information is well organized and available for review.

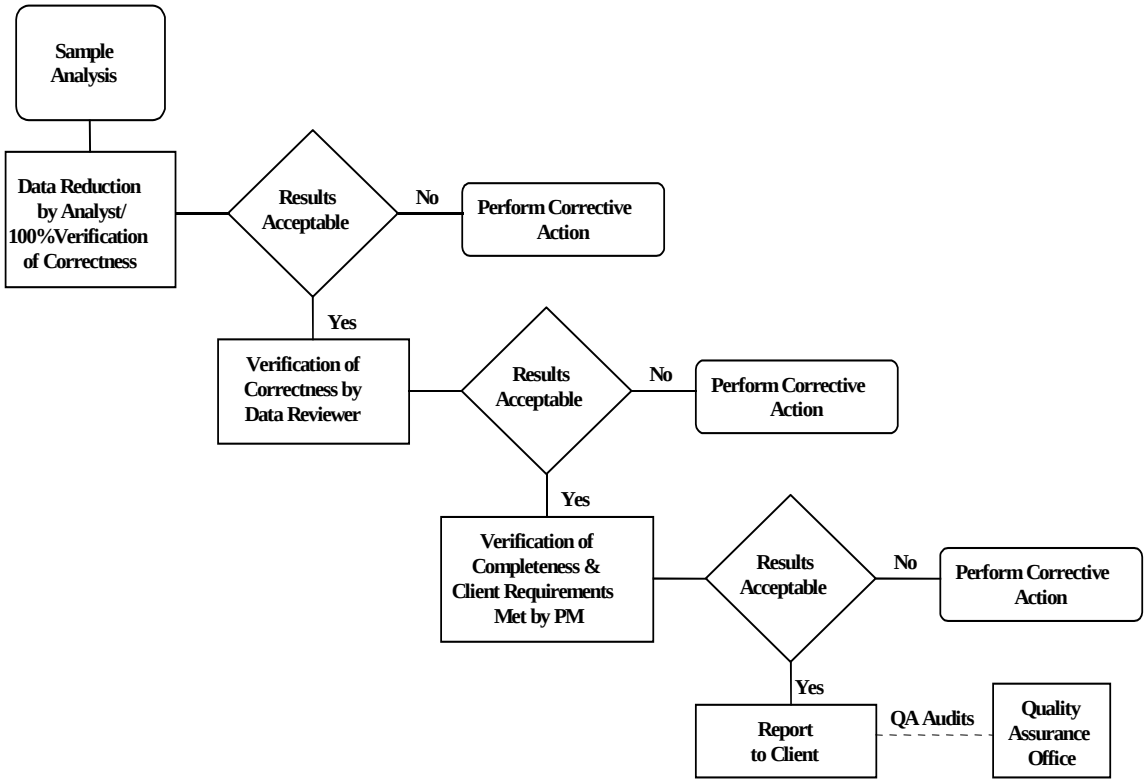
_____ _____
Analyst Signature Date

_____ _____
Technical Manager Signature Date

_____ _____
Quality Assurance Coordinator Signature Date

Figure 19-2

Work Flow



SECTION 20

EQUIPMENT AND CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

All instrumentation and support equipment is designated by a unique laboratory ID.

20.2 Preventive Maintenance

20.2.1 The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

20.2.2 Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

20.2.3 Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager and Supervisor to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may be / are also outlined in analytical SOPs or instrument manuals. Further detail for equipment maintenance is included in SOP No. PT-QA-022 and individual analytical SOPs. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

20.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

20.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement

of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

20.2.4.2 Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

20.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

20.2.5 If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

20.2.6 In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

20.2.7 If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

Support equipment that provides quantitative results are calibrated or calibration verified to a recognized national metrology standard, such as NIST, where available, over the expected range of use. The acceptability for use shall be according to the needs of the analysis or application for which the equipment is being used.

Calibration and calibration verification scheduling and documentation for support equipment is maintained by the QA department. All equipment is labeled with the most recent calibration information and the next verification due date.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains “calibration only” ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Refer to SOP No. PT-QA-012 for balance and weight calibration.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to $\pm$ 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly. IR thermometers are checked daily for calibration accuracy against an NIST thermometer daily before use.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method or device-specific logbooks. More information on this subject can be found in the thermometer calibration SOP No. PT-QA-008.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day. Sample storage temperatures are monitored continuously (24/7).

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and $\leq$ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks or electronically. Refer to SOP No. PT-QA-008 for temperature monitoring.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices (except Class A Glassware) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis. Glass micro-syringes are considered the same as Class A glassware.

For those dispensers that are not used for analytical measurements, a label is / can be applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements. Pipette calibration is described in Pittsburgh SOP No. PT-QA-017.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

20.3.6 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated semiannually by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service

until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP/ICP-MS methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i. e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after every 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (GCMS methods, for example) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective

action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with unacceptable calibration verification may be fully useable and may be reported, **based upon discussion and approval of the client**, under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. These calculations are available in the laboratory method SOPs. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on %Drift or %Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

Note: Some state programs require additional verification steps for linear and quadratic calibration – i.e. reading the low level standard against the curve, or verification at a low and a high concentration. See analytical SOPs and project notes for details.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1
TestAmerica Pittsburgh Equipment List
(see PT-QA-WI-045 for the most current listing)

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
GC w/ Dual ECD	Hewlett-Packard Lab ID: GC1	Chem Station Rev. A 09.03 [1417]	6890	US00024872	--	
GC w/ Dual ECD with EPC	Hewlett-Packard Lab ID: GC2	Real-Time Plot Version 4.1 ZF12	5890A	3235A48356	1991	New
GC w/ Dual FID	Hewlett-Packard Lab ID: GC3	Real-Time Plot Version 4.1 ZF12	5890 Series II	2921A23920		
GC w/ Dual NPD	Hewlett-Packard Lab ID: GC5	Chem Station Rev. A 09.03 [1417]	6890A	US00025516	1998	New
GC w/ Dual FPD	Hewlett-Packard Lab ID: GC6	Chem Station Rev. A 09.03 [1417]	6890N	US10145113	2001	New
GC w/ Dual ECD	Hewlett-Packard Lab ID: GC8	Chem Station Rev. A 06.03 [509]	6890	US00023401	1998	New
GC w/ Dual ECD	Hewlett-Packard Lab ID: GC10	Chem Station Rev. A 09.01 [1206]	6890N	US10145114	2001	New
GC w/ Dual ECD	Hewlett-Packard Lab ID: GC12	Chem Station Rev. A 09.01 [1206]	6890N	US10237038	2002	New
GC w/ Dual ECD	Hewlett-Packard Lab ID: GC14	Chem Station Rev. A 07.01 [682]	6890	US00026141	2005	Used
GC w/ Dual ECD	Hewlett-Packard Lab ID: GC15	Chem Station B.04.03 (16)	7890	CN10441121	2011	New
GC w/ Dual ECD	Hewlett-Packard Lab ID: GC16	Chem Station D.02.00.275	6890N	CN10435031	2013	Used
Balance	AND Lab ID: 14234838	N.A.	GR120	14234838	2010	Used
Hydrogen Generator	Parker Balston	N.A.	H2-800	H2800104C	2006	New
GC/MS	Hewlett-Packard Lab ID: HP3	Enviroquant Chem Station G1701BA Version B.01.001	6890 (GC) 5973 (MSD)	US00009844 (GC) US72020964 (MSD)	1997	New
Concentrator	OI Analytical	N.A.	Eclipse	D617466100P	2006	New

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS	Hewlett-Packard Lab ID: HP4	Enviroquant Chem Station G1701BA Version B.01.001	6890 (GC) 5973 (MSD)	US00010799 (GC) US72821085 (MSD)	1998	New
Concentrator	OI Analytical	N.A.	Eclipse	D616466032P	2006	New
GC/MS	Hewlett-Packard Lab ID: HP5	Enviroquant Chem Station G1701BA Version B.01.001	6890 (GC) 5973 (MSD)	US00023292 (GC) US82322212 (MSD)	1998	New
Concentrator	OI Analytical		Eclipse	D616466026P	2006	New
GC/MS	Hewlett-Packard Lab ID: HP6	Enviroquant Chem Station G1701BA Version B.01.001	6890 (GC) 5973 (MSD)	US00030465 (GC) US92522786 (MSD)	1999	New
Concentrator	OI Analytical		Eclipse	B414466952P	2006	New
GC/MS	Hewlett-Packard Lab ID: HP7	Enviroquant Chem Station G1701BA Version B.01.001	6890 (GC) 5973 (MSD)	US00028345 (GC) US91411730 (MSD)	2005	Used
Concentrator	OI Analytical	N.A.	Eclipse	D617466098P	2006	New
Autosampler	EST Analytical	N.A.	Centurion: CENT WS	CENTS136020 110	2010	New
GC/MS	Hewlett-Packard Lab ID: HP8		6890 FID	US00001295 (GC) 3526I01420 (Headspace)	2001	New
GC/MS	Hewlett-Packard Lab ID: HP9	HP Chem Station 275 Version 0.20	HP6980N (GC) 5973 (MS)	CN10505083 (GC) US44831792 (MS)	2012	Used
Concentrator	Encon	N.A.	ENCO	372061604E	2012	Used
Autosampler	EST Analytical	N.A.	Archon 8100	14178	2012	Used
Oven	Fisher Scientific Lab ID: VOA Glassware Oven	N.A.	625G	503N0042	2005	New
Balance	AND Lab ID: 14234771	N.A.	GR120	14234771	2010	
GC/MS	Hewlett-Packard Lab ID: 71	EnviroQuant Chem Station G1701BA Version B.01.00	6890 (GC) 5973 (MSD)	US00029391 (GC) US91422511 (MSD)	1999	New
GC/MS	Hewlett-Packard Lab ID: 722	EnviroQuant Chem Station G1701BA Version	6890 (GC) 5973 (MSD)	US00029396 (GC) US91922512	1999	New

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
		B.01.00		(MSD)		
GC/MS	Hewlett-Packard Lab ID: 731	EnviroQuant Chem Station G1701BA Version B.01.00	6890 (GC) 5973 (MSD)	US00031329 (GC) US93112052 (MSD)	2000	New
GC/MS	Hewlett-Packard Lab ID: 732	MSD Chem Station D.01.02.16 06/15/2004	6890N (GC) 5973 (MSD)	CN10426047 (GC) US41746674 (MSD)	2004	New
GC/MS	Hewlett-Packard Lab ID: 733	EnviroQuant Chem Station G1701BA Version B.01.001	6890 (GC) 5972 (MSD)	US65135379 (MSD) US00028233 (GC)	2005	Used
GC/MS	Hewlett-Packard Lab ID: APEX	EnviroQuant Chem Station G1701BA Version B.01.001	6890 (GC) 5973 (MSD)	US 71410457 (MSD) US00007984 (GC)	2002	Used
GC/MS	Hewlett-Packard Lab ID: MSD7	EnviroQuant Chem Station G1701BA Version B.01.001	6890 (GC) 5972 (MSD)	US80210935 (MSD) DE00020249 (GC)	2002	Used
ICP	Thermo Fisher Lab ID: 6500-2	ITEVA	6500	ICP-20074713	2014	Used
ICP	Thermo Fisher Lab ID: 6500	ITEVA	6500	ICP-20074812	2008	New
ICP/MS	Thermo Electron Lab ID: ICPMS	Plasma Lab	X-Series ICPMS	X0225	2003	New
ICP/MS	Thermo Electron Lab ID: ICPMS2	Plasma Lab	X Series ICPMS	X0344	2006	Used
Mercury Analyzer	Leeman Labs Lab ID: HGHYDRA	WIN HG	Hydra	3009	2003	New
Mercury Analyzer	Leeman Labs	ENVOY	Hydra II	0024	2010	New
Metals Digestion Block	Environmental Express Lab ID: H_2O #1	N.A.	Hot Block		2003	New
Metals Digestion Block	Environmental Express Lab ID: H_2O #3	N.A.	Hot Block		2000	New
Metals Digestion Block	Environmental Express Lab ID: H_2O #4	N.A.	Hot Block		2003	New
Metals Digestion	Environmental Express	N.A.	Hot Block		2003	New

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
Block	Lab ID: H_2O #5					
Metals Digestion Block	Environmental Express Lab ID: H_2O #6	N.A.	Hot Block		2000	New
Metals Digestion Block	Environmental Express Lab ID: Soil #10	N.A.	Hot Block		2003	New
Metals Digestion Block	Environmental Express Lab ID: Soil #9	N.A.	Hot Block		2003	New
Metals Digestion Block	Environmental Express Lab ID: Soil #8	N.A.	Hot Block		2003	New
Metals Digestion Block	Environmental Express Lab ID: Soil #7	N.A.	Hot Block		2003	New
Mercury Digestion Block	Environmental Express Lab ID: Soil #35114	N.A.	Hot Block		2003	New
Mercury Digestion Block	Environmental Express Lab ID: Soil #35113	N.A.	Hot Block		2003	New
Mercury Digestion Block	Environmental Express Lab ID: Soil #35112	N.A.	Hot Block		2003	New
Mercury Digestion Block	Environmental Express Lab ID: Soil #35111	N.A.	Hot Block		2003	New
Balance	AND Lab ID: P1856710	N.A.	EK-610I	P1856710	2008	New
Balance	OHAUS Lab ID: B417532814	N.A.	SP602	B417532814	2015	New
Ion Chromatograph (IC2100A)	Dionex	Chromeleon Client 6.80 SP4 Build 2361 (130805) 58031	ICS 2100	11050879	2011	New
Ion Chromatograph (IC25)	Dionex	Chromeleon Client 6.80 SP4 Build 2361 (130805) 58031	IC 25	00040396	2000	New

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
Ion Chromatograph (IC2000)	Dionex	Chromeleon Client 6.80 SP4 Build 2361 (130805) 58031	ICS 2000	08050561	2008	New
Ion Chromatograph (ICS2100B)	Dionex	Chromeleon Client 6.80 SP4 Build 2361 (130805) 58031	ICS 2100	11050258	2011	New
Astoria 2 Analyzer system	Astoria Pacific International	FASPAC II Flow Analyzer Software Version 2.1.2	200-A100-03	200231	5/21/2010	New
Astoria 2 Analyzer Sampler 311, XYZ, Diluter	Astoria Pacific International	FASPAC II Flow Analyzer Software Version 2.1.2	311-A100-03	4940A14695	5/21/2010	New
Astoria 2 Analyzer 322 Two Channel Auxiliary Pump	Astoria Pacific International	FASPAC II Flow Analyzer Software Version 2.1.2	322-A100-00	322199	5/21/2010	New
Astoria Analyzer Diluter Module 312-M2, 5 ml Syringe	Astoria Pacific International	FASPAC II Flow Analyzer Software Version 2.1.2	312-A200-5ML	4803A12911	5/21/2010	New
Diluter Module: Valve Module, 312 Diluter	Astoria Pacific International	FASPAC II Flow Analyzer Software Version 2.1.2	312-B002-00	300971	5/21/2010	New
Autoanalyzer (ALPKEM1)	OI Analytical (Test: 353.2)	WINFLOW 4.03	Alpkem Flow Solution IV	928893438	1998	New
UV/VIS	Milton Roy	Spectronic	Genesys5	3V08239002	2003	Used
UV/VIS	Thermo Electron Corp. (Test: MBAS)	N.A.	GENESYS 10 335900-000	2D5K278001	2007	New
UV/VIS	Thermo Electron Corp. (Test: 3060A/7196A)	N.A.	GENESYS 10SVIS	2D9P070001	2011	New
Midi Distillation Blocks	Westco Scientific	N.A.	Easy Dist		2000	New
Midi Distillation Blocks	Westco Scientific	N.A.	Easy Dist		2001	New
Midi Distillation Blocks	Westco Scientific	N.A.	Easy Dist		2005	New
pH meter	Fisher Scientific	N.A.	AR25	AR93315378	2004	New

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
pH meter	Fisher Scientific	N.A.	XL15	XL 94102132	2012	New
pH meter	Oakton	N.A.	pH110 Series	927836	2012	New
MultiMeter	Myron L Co.	N.A.	Ultrameter II 6Psi	6205309		New
Oven	Thermolyne	N.A.	6000			New
Oven	Blue M Electric Co. Lab ID: Oven #2	N.A.	OV-18A	OV1-15300		New
Oven	Fisher Scientific Lab ID: OV02	N.A.	Isotemp 630G	001O0035		New
Oven	Precision Scientific Lab ID: OV08	N.A.	18EG	10AV-9		New
Oven	Fisher Lab ID: ZHE Oven	N.A.	Isotemp Oven Model 301			
COD Reactor A	HACH	N.A.	45600	8911011228		New
COD Reactor B	Bioscience	N.A.	COD T432	163-4667	2013	Used
TOC Analyzer	OI Analytical Lab ID: 1030	N.A.	Aurora 1030	E717730273	2007	New
TOC (Lloyd Khan Method) Analyzer	Thermo Electron Corp.	Eager 300 Version 2.2 9/2004	Flash EA 112 MAS 200R NC Soil Analyzer	20057159-20057135	2006	New
Autoanalyzer	Thermo Clinical Labsystems Lab ID: KONELAB-1 (Tests:9012/420.2/420.4/9066/SM 4500 CL E/410.4)	KoneLab Workstation Software	Aqua 200	A0619933	2005	New
Method 1677 Autoanalyzer (ALPKEM3)	OI Analytical FS3000	WINFLOW v 4.03	A0001604 A000989 (autosampler)	135804017 135898017 (autosampler)	2007	Used

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
Method 1677 Autoanalyzer (ALPKEM2)	OI Analytical FS3000	WINFLOW v 4.03	A0001604 A000952 (autosampler)	120804293 120898293 (autosampler)	2001	New
BOD Meter - Automated	YSI	BODAssay PLUS V. 3.0	52	03L0794	2004	New
BOD Meter - Manual	YSI	N.A.	50B	91K033593	2003	New
Flashpoint Tester	Rapid Tester Lab ID: SETA-1	N.A.	RT-00001	024149	2002	New
Flashpoint Tester	Petrotest Pensky Martin	N.A.	PMA-4	0741043006	2004	New
Flashpoint Tester	Fisher Scientific	N.A.	K-16200	2501		
Turbidimeter	HF Scientific Inc.	N.A.	Micro 100	105034		
Speed Vap II	Horizon	N.A.	Speed Vap # 9000	01-0333	2001	New
Speed Vap II	Horizon	N.A.	Speed Vap # 9000	01-0332	2001	New
Hotplate	Thermolyne Lab ID: #3	N.A.	Cimarec 3	1073390872634		Used
Hotplate	Thermolyne Lab ID: #1	N.A.	Cimarec 3	1073010868578	2005	New
Hotplate	Thermolyne Lab ID: #14	N.A.	Cimarec 3	1073010868561		New
Hotplate	Thermolyne Lab ID: #4B	N.A.	Cimarec 3	1073980593891		New
Waterbath	Thermo Electron Corp.	N.A.	Precision 2872	202471	2007	New
Centrifuge	Damon/IEC Division Lab ID: CENT-3	N.A.	CU-5000	33473227		
Balance	Mettler Lab ID: 1126472457	N.A.	PB602	1126472457	2005	New
Balance	Mettler Lab ID: G76383	N.A.	AE240	G76383		
Balance	Fisher Lab ID: 25606	N.A.	S-400	25606		
Balance	Mettler Lab ID: AB204S	N.A.	AB204S	1126020829	2005	New
Balance	A & D Lab ID: GR-200	N.A.	GR-200	14224939	2007	New
Balance	A & D Lab ID: FZ1200i	N.A.	FZ1200i	15900520		

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
Sonicator	Fisher Scientific	N.A.	550 Sonic Dismembrator	F2099	1985	
Concentrator	Meyer	N.A.	N-Evap 112	5376		
Concentrator	Meyer	N.A.	N-Evap 115	9217		
Concentrator	Horizon Lab ID: 1	N.A.	Dry Vap	227253	2006	New
Concentrator	Horizon Lab ID: 2	N.A.	Dry Vap	227254	2006	New
Concentrator	Horizon Lab ID: 3	N.A.	Dry Vap	227255	2006	New
Concentrator	Horizon Lab ID: 4	N.A.	Dry Vap	227256	2006	New
Soxtherm Extractor	Gerhardt Lab ID: 1	N.A.	SE-3A/S306A	4012404	2002	New
Soxtherm Extractor	Gerhardt Lab ID: 7	N.A.	SE-3A/S306A	4012399	2002	New
Soxtherm Extractor	Gerhardt Lab ID: 6	N.A.	SE-3A/S306A	4012398	2002	New
Soxtherm Extractor	Gerhardt Lab ID: 5	N.A.	SE-3A/S306A	4012403	2002	New
Soxtherm Extractor	Gerhardt Lab ID: 4	N.A.	SE-3A/S306A	4012402	2002	New
Soxtherm Extractor	Gerhardt Lab ID: 2	N.A.	SE-3A/S306A	4012401	2002	New
Soxtherm Extractor	Gerhardt Lab ID: 3	N.A.	SE-3A/S306A	4012400	2002	New
Soxtherm Extractor	Gerhardt Lab ID: 8	N.A.	SE-3A/S306A	4002039	2002	New
Electric Kiln	Cress	N.A.	FTX-27P	46053	1992	
Electric Oven	Wilt Industries	N.A.	A85		1999	
TCLP Tumbler	Associated Design & Manufacturing Co. Lab ID: T-8	N.A.	6004-0590	1788		
ZHE Rotator	Associated Design & Manufacturing Co. Lab ID: Z1	N.A.	3740-8-BRE	1223		
ZHE Rotator	Bodine (Associated Design) Lab ID: Z2	N.A.	362RA9018			

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
ZHE Rotator	Bodine Electric Co. Lab ID: Z3/Z5	N.A.	42R5BFC1-E3			
ZHE Rotator	Bodine (Associated Design) Lab ID: Z4	N.A.	34R4BFC1-5R			
TCLP Tumbler	Environmental Express Lab ID: T6	N.A.		3209-12-466		
TCLP Tumbler	Environmental Express Lab ID: T7	N.A.		3209-12-467		
TCLP Tumbler	Environmental Express Lab ID: T9	N.A.		3209-12-463		
TCLP Tumbler	Dayton (motor) Lab ID: T1	N.A.	2Z794D			
TCLP Tumbler	Dayton (motor) Lab ID: T2	N.A.	5K939E			
TCLP Tumbler	Dayton (motor) Lab ID: T3	N.A.	5K939B			
TCLP Tumbler	Dayton (motor) Lab ID: T5	N.A.	5K939B			
pH Meter	Thermo Orion	N.A.	STARA1115	J00634	2012	New
Balance	A & D Lab ID: 14628771	N.A.	GF6000	14628771		
Balance	A & D Lab ID: 11684	N.A.	GX4000	14536813		
Balance	Mettler Lab ID: 1120122641	N.A.	PB8001S	1120122641		
Hot Plate	Thermodyne Lab ID: TCLP Hot Plate	N.A.	2200			
Centrifuge	Beckman	N.A.	J6-M	8749	2007	New
Centrifuge	Beckman	N.A.	J6-M	8551	2007	New
Centrifuge	Thermo Electron Corp. Lab ID: Cent-1	N.A.	K	71654833		
Centrifuge	Thermo Electron Corp Lab ID: Cent-2	N.A.	K	71654125		
Method 1664A UCT Cartridge	Enviro-Clean	N.A.	ENUCNIOGXF	UCT #1	2009	New

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
Oil-Less Vacuum Pump for UCT Cartridge System	Rocker (110V, 60 Hz)	N.A.	400	1R400TLTA076	2012	New
Oil-Less Vacuum Pump for UCT Cartridge System	Rocker (110V, 60 Hz)	N.A.	400	1R400TLTA069	2012	New
GPC – AccuPrep (GPC2)	J2 Scientific	N.A.	MPS	GPC-1022-1.0-DI	2009	New
GPC - AccuVap Concentrator System	J2 Scientific	N.A.	FLX	AVM-251-2.5-F	2009	New
GPC – Autosampler Module	J2 Scientific	J2 Software	PrepLinc AS4	ASA-1045-1.3	2009	New
Freezer	Kenmore by Sears	N.A.	253.28052803	WB91633867	2009	New
Digital Barometer	Fisher Scientific	N.A.	02-401	91116011	2009	New
Digital Buret	Brand	N.A.	4761161 TM (catalogue #)	11G38510	2010	New
IR Thermometer	EXTECH Instruments	N.A.	42511	SR IR#1	2010	New
IR Thermometer	EXTECH Instruments	N.A.	42511	SR IR#2	2010	New
IR Thermometer	EXTECH Instruments	N.A.	42511	WC IR#1	2010	New
IR Thermometer	EXTECH Instruments	N.A.	42511	OP IR#1	2010	New
Gel Permeation Chromatograph – GPC1	J2 Scientific	J2 Software	Prep Linc GPC	GPC-1089-1.0 4340A1855 PLH-1126-1.1	2010	New
Autosampler	Hewlett-Packard GC15	N.A.	7693	10390085	2010	New
Freezer	Kenmore Lab ID: WC Freezer #2	N.A.	253.28052806	WB02643189	2010	New
Freezer	Frigidaire Lab ID: Tissue	N.A.	FKFH21F7WB	WB02442941	2010	New

Instrument Type	Manufacturer	Instrument Software	Model Number	Serial Number	Year Put into Service	Condition When Received
	Freezer #3					
Freezer	Frigidaire Lab ID: Tissue Freezer #4	N.A.	FKCH17F7WC	WB02851917	2010	New
Freezer	Frigidaire Lab ID: Tissue Freezer #5	N.A.	253.28092801	WB92436406	2010	New
Freezer	Frigidaire Lab ID: Tissue Freezer #9	N.A.	LFFH20F3QWA	WB43855947	2014	New
Freezer	Frigidaire Lab ID: Tissue Freezer #10	N.A.	LFFH21F7HWK	WB43160650	2014	New
Muffle Furnace	Thermo Fisher	N.A.	F6010	0152978801110621	6/21/2011	New
pH Meter	Fisher Scientific	N.A.	Accumet XL15	XL94102132	4/4/2012	New
Refrigerator	Thermo Scientific	N.A.	3566-10A	2017081210718	2012	New
pH Meter	Accumet	N.A.	XL15	XL94102803	2012	New
Balance	AND Lab ID: 14239603	N.A.	GR-200	14239603	2013	New
pH Meter	Fisher Scientific	N.A.	Accumet AR20	AR81206163	2013	Used
Discrete Analyzer	SEAL Analytical	AQ2 Series Software V4	AQ2	090364	2013	Used
Discrete Analyzer	SEAL Analytical	AQ2 Series Software V4	AQ2	090413	2013	Used
Oil-less Vacuum Pump		N.A.	400	167400-11-AB2363	2013	New
Method 1664A UCT Cartridge		N.A.	ENUCN10GXF	UCT#2	2013	New
Incubator	Fisher Scientific	N/A	6500	60300058	2014	Used (Dayton)
BOD2 – BOD autosampler	Gilson	PC-BOD version 3.0.0.142	AutoMax122 PBM-1000-688	261E4N113	2014	New
Stirrer & Level Sensor Control Box	Gilson	PC-BOD version 3.0.0.142	PB-10030	MT-1C4-174	2014	New
BOD autosampler Interface	Gilson	PC-BOD version 3.0.0.142	PC-1085-00	MT-1H4-219	2014	New
DO Meter	YSI		5100	13J101062	2014	New

Tables 20-2
Example - Schedule of Routine Maintenance
(See SOP No. PT-QA-022 for more instrument specific information)

<table>
<tr><th>Daily</th><th>Weekly</th><th>Monthly</th><th>Quarterly</th><th>Annually</th><th>As Needed</th></tr>
<tr><td>Check sample waste container level.</td><td>Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, and condition of drain tubing.</td><td>Clean all filters and fans.</td><td rowspan="5">Replace oil in roughing pumps.</td><td rowspan="5">Replace oil in turbo-molecular pump.</td><td rowspan="5">Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics, CEM, deflector voltage.</td></tr>
<tr><td>Check quartz torch condition.</td><td>Check condition of sampler and skimmer cones.</td><td rowspan="4">Check recirculator water level.</td></tr>
<tr><td>Measure quartz torch for proper alignment.</td><td rowspan="3">Check and drain oil mist eliminator on roughing pumps.</td></tr>
<tr><td>Clean spray chamber and nebulizer.</td></tr>
<tr><td>Check oil level of roughing pumps.</td></tr>
</table>

SECTION 21

MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Microsyringes can be verified at least semi-annually or disposed of after 6 months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights And Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program) or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia – Pacific Laboratory Accreditation Cooperation). A certificate and scope of accreditation is kept on file at the laboratory.

Additional details can be found in SOP No.'s PT-QA-008 – Thermometer and Barometer Verification and Temperature Monitoring, and PT-QA-012 – Selection and Calibration of Balances and Weights.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, ISO 9001:2000, ISO 17025. All reference standards from commercial vendors shall be accompanied by a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated

- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis.

The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory contingency procedures for re-verifying expired standards is documented in SOP No. PT-QA-006 – Procurement of Standards and Materials, Labeling and Traceability.

21.4 Documentation And Labeling Of Standards, Reagents, And Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. (Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.)

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in the QA public drive L:\QA\Facility_QA_Documents\Certificate_of_Analysis. Standard certificates are maintained by each department and a copy should be scanned into LIMS Reagent log.

Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and SOP No. PT-QA-006, Procurement of Standard and Materials; Labeling and Traceability.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

21.4.1 All standards, reagents, and reference materials that may affect quality must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded within LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from electronic standard log in LIMS)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in standard/reagent log. Health and safety warning are in the MSDS (Material Safety Data Sheets) which is accessed through company intranet site.

21.4.3 In addition, the following information may be included:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22

SAMPLING

22.1 Overview

The laboratory provides sampling services for the following matrices:

- Groundwater
- Wastewater
- Potable Water
- Wastes
- Soil and Sediment

The laboratory also offers the following services:

- Flow Monitoring
- Field Parameter Analysis

Field Analyses are address in TestAmerica Pittsburgh SOP No.s:

- PT-FS-001 – Field Measurement of Dissolved Oxygen (DO)
- PT-FS-002 – Field Measurement of Total Residual Chlorine
- PT-FS-003 – Field Measurement of pH

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. For detailed information regarding container/bottle order, refer to laboratory SOP No. PT-SR-002 - Bottle Order Preparation and Shipping.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – AR Select (ACS) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – AR Select (ACS), Trace-Metals Grade or equivalent
- Sodium Hydroxide – AR Select (ACS) or equivalent
- Sulfuric Acid – AR Select (ACS) or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent
- Sodium Bisulfate – ACS Grade or equivalent

22.3 Definition Of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in “days” (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in “hours” (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the SOPs are derived from the source documents for the methods. If method required holding times as specified in the SOPs or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines for subsampling are located SOP No. PT-QA-024.

SECTION 23

HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain Of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in the project folder.

23.1.2 Legal / Evidentiary Chain-of-Custody

The laboratory may, upon special request, adhere to legal/evidentiary chain of custody requirements. If TestAmerica agrees to such procedures the samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal (Figure 23-4), retain the shipping record with the COC, and initiate an internal COC (Figure 23-5) for laboratory use by analysts and sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

23.2.1 Laboratory Receipt

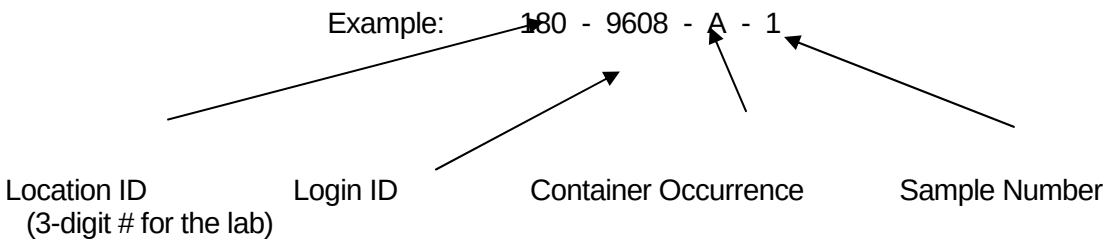
When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented via the Sample Receipt application, Sample Receipt checklist (Figure 23-3). and brought to the immediate attention of the Project Manager who will, in turn, contact the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record. This procedure is further described in SOP No. PT-QA-027, Sample Receiving and Chain-of-Custody.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This

system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Pittsburgh Laboratory (Location 180). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 180 - 9608 - A - 1 - **A** ←—— **Secondary Container Occurrence**

Example: 180-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method ;
- sample holding times must be adhered to ;
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;

- Efforts should be made to minimize any air bubbles in aqueous volatile samples. Air bubbles also the escape of volatile organics. This is especially important because air bubbles tend to form in iced samples. Volatile vials containing air bubbles larger than a pea will be treated as non-conformances;
- Samples that require chilling must be received at >0 to ≤6℃; Samples for Microbiological testing must be received at ≥1 to ≤10°C.
- If Matrix Spikes are required for the project, separate sample volumes must be available for the requested analyses;
- the project manager will be notified if any sample is received in damaged condition

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.1.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation on the chain-of-custody will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.
- If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction or agreement with the client, the sample will be rejected by the laboratory.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. PT-SR-001 – Sample Receipt and Login.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

Refrigerators and other units used to store samples must not be used for standard, reagent or other types of storage.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated cold room or refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the cold room or refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. Raw samples requiring cold storage are kept in the cold room for approximately 30 days after reported. Volatile samples are stored in the VOA refrigerator. All sample extracts are kept in the refrigerators for approximately two to four weeks after analysis, which meets or exceeds most sample holding times. After this time the sample extracts are moved to cold room, where they are stored for an additional three to six months before they are disposed of. This holding period allows samples to be checked if a discrepancy or question arises. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 Hazardous Samples And Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous, for any sample that is known to be hazardous at the time of receipt a cautionary email communication should be sent to all applicable laboratory personnel by the project manager or designee. All hazardous samples are disposed of appropriately through a hazardous waste disposal process. Foreign soil samples are sent out for incineration by an USDA-approved waste disposal facility. Analysts will notify Sample Control of any sample determined to be hazardous after completion of analysis by sending an email. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-

custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP No. PT-HS-001 and Chemical Hygiene Plan). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1

Example: Chain of Custody (COC)

[redacted]
Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL-4142 (0907)

Client	Project Manager	Date	Chain of Custody Number 364410
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page _____ of _____
City / State / Zip Code	Site Contact	Lab Contact	Analysis (Attach list if more space is needed)
Project Name and Location (State)	Carrier/Waybill Number		Special Instructions/ Conditions of Receipt
Contract/Purchase Order/Quote No.	Matrix / Containers & Preservatives		

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Air	Aqueous	Sed.	Soil	Unpres.	H_2SO_4	HNO_3	HCl	NaOH	ZnAc/ NaOH

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months (A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other_____

QC Requirements (Specify)

1. Relinquished By	Date	Time	1. Received By	Date	Time
2. Relinquished By	Date	Time	2. Received By	Date	Time
3. Relinquished By	Date	Time	3. Received By	Date	Time

Comments

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

Figure 23-2

Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - *Client name, address, phone number and fax number (if available)*
   - *Project name and/or number*
   - *Unique sample identification*
   - *Date, time and location of sampling*
   - *The collectors name*
   - *The matrix description*
   - *The container description*
   - *The total number of each type of container*
   - *Preservatives used*
   - *Analysis requested*
   - *Requested turnaround time (TAT)*
   - *Any special instructions*
   - *Purchase Order number or billing information (e.g. quote number) if available*
   - *The date and time that each person received or relinquished the sample(s), including their signed name.*
   - **Information must be legible**

2) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - **Information must be legible**

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

4) Samples must be preserved according to the requirements of the requested analytical method. (See Sampling Guide)

5) Most analytical methods require chilling samples to 4^{o} C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6^{o} C and above freezing (0^{o}C). For methods with other temperature criteria (e.g. some bacteriological methods require $\leq$ 10 ^{o}C), the samples must arrive within $\pm$ 2^{o} C of the required temperature or within the method specified range. **Note:** Samples that are hand delivered to the laboratory immediately after

collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

5i.) Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 5. In these cases, the samples shall be considered acceptable if the samples were received on ice.

5ii.) If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.

5iii.) Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

- **FOR WATER SAMPLES TESTED FOR CYANIDE (Method OIA-1677)**
  - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample is treated in the field with lead carbonate or if the client requests the sample to be treated at the lab it will be filtered and treated at the lab with Cadmium Chloride.

  - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered and qualify the results in the final report.

- It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.

- The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).

- Water samples that require ortho-phosphorus must be filtered in the field within 15 minutes of sampling. Samples received without indication of filtration in the field will have results flagged for improper preservation.

- Samples for coliform analysis must be in sterile containers and must be free of residual chlorine. The bottle must be filled to <u>above</u> the 100mL mark.

6) *Matrix Spikes* are required for your project, separate sample volumes must be available for the requested analyses.

7) For Volatile Organic analyses: Efforts should be made to minimize any air bubbles in aqueous volatile samples. Air bubbles also the escape of volatile organics. This is especially important because air bubbles tend to form in iced samples. Volatile vials containing air bubbles larger than a pea will be treated as non-conformances.

8) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply a blank with the bottle order.

9) Sample Holding Times

- TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (working days) remaining on the holding time for us to ensure analysis.

- Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. Samples analyzed in the laboratory will be qualified on the final report to indicate holding time exceedance.

10) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis. The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation.

11) Recommendations for packing samples for shipment.

- Pack samples in Ice rather than "Blue" ice packs.

- Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.

- Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.

- Fill extra cooler space with bubble wrap.

12) If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction or agreement with the client, the sample will be rejected by the laboratory.

Figure 23-3

Example: Sample Receipt Checklist

Login Sample Receipt Checklist

Client: Cardno ENTRIX — Job Number: 180-264-1

Login Number: 264
List Number: 1
Creator: Gamber, Tom

List Source: TestAmerica Pittsburgh

Question	Answer	Comment
Radioactivity either was not measured or, if measured, is at or below background	True	
The cooler's custody seal, if present, is intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the sample IDs on the containers and the COC.	True	
Samples are received within Holding Time.	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

Figure 23-4

Example: Custody Seal

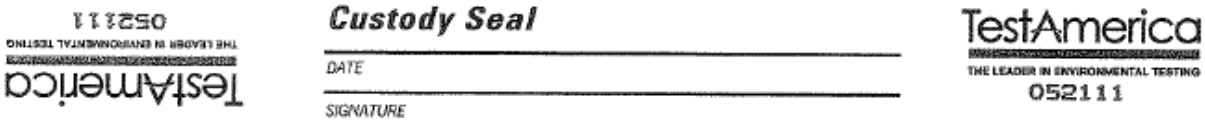


Figure 23-5

Example: Internal Chain-of-Custody (COC) Form

Historical Internal Chain of Custody

Login	Smp	Customer Sample ID	Matrix	Container ID	Lab Sample ID	Container Type	Location	Custody User	I/O	COC ID	COC Date
280-2756	1	J19WL7	Solid	280-126041	280-2756-A-1	Plastic 250ml - unpreserved	AH66	Chavez, Lawrence	I	280-13864	05/25/10 12:38
280-2756	1	J19WL7	Solid	280-126041	280-2756-A-1	Plastic 250ml - unpreserved	128	Berry III, Paul B	I	280-9241	04/24/10 10:24
280-2756	1	J19WL7	Solid	280-126041	280-2756-A-1	Plastic 250ml - unpreserved	WC Dpt	Berry III, Paul B	I	280-9234	04/24/10 08:52
280-2756	1	J19WL7	Solid	280-132543	280-2756-A-1-A						
280-2756	1			280-132544	280-2756-A-1-B						
280-2756	1			280-132545	280-2756-A-1-C						
280-2756	1	J19WL7	Solid	280-134154	280-2756-A-1-D						
280-2756	1	J19WL7	Solid	280-155231	280-2756-A-1-E						
280-2756	1	J19WL7	Solid	280-126042	280-2756-B-1	Soil jar 4oz	AH66	Chavez, Lawrence	I	280-13864	05/25/10 12:38
280-2756	1	J19WL7	Solid	280-126042	280-2756-B-1	Soil jar 4oz	128	Johnson, Aaron S	I	280-12354	05/12/10 22:18
280-2756	1	J19WL7	Solid	280-126042	280-2756-B-1	Soil jar 4oz	OP Dpt	Johnson, Aaron S	I	280-12286	05/12/10 15:24
280-2756	1	J19WL7	Solid	280-165103	280-2756-B-1-A						
280-2756	1	J19WL7	Solid	280-126043	280-2756-C-1	Soil jar 4oz	AH66	Chavez, Lawrence	I	280-13864	05/25/10 12:38
280-2756	1	J19WL7	Solid	280-126043	280-2756-C-1	Soil jar 4oz	128	Johnson, Aaron S	I	280-10311	04/30/10 01:16
280-2756	1	J19WL7	Solid	280-126043	280-2756-C-1	Soil jar 4oz	OP Dpt	Johnson, Aaron S	I	280-10172	04/29/10 15:27
280-2756	1	J19WL7	Solid	280-126043	280-2756-C-1	Soil jar 4oz	128	Skrip, Sean P	I	280-9923	04/29/10 15:52
280-2756	1	J19WL7	Solid	280-126043	280-2756-C-1	Soil jar 4oz	OP Dpt	Skrip, Sean P	I	280-9812	04/29/10 09:27
280-2756	1	J19WL7	Solid	280-134359	280-2756-C-1-A						
280-2756	1	J19WL7	Solid	280-138785	280-2756-C-1-B						
280-2756	1	J19WL7	Solid	280-165318	280-2756-C-1-C						
280-2756	1	J19WL7	Solid	280-170514	280-2756-C-1-D						
280-2756	2	J19WL8	Solid	280-126044	280-2756-A-2	Plastic 250ml - unpreserved	AH66	Chavez, Lawrence	I	280-13864	05/25/10 12:38
280-2756	2	J19WL8	Solid	280-126044	280-2756-A-2	Plastic 250ml - unpreserved	128	Berry III, Paul B	I	280-9241	04/24/10 10:24
280-2756	2	J19WL8	Solid	280-126044	280-2756-A-2	Plastic 250ml - unpreserved	WC Dpt	Berry III, Paul B	I	280-9234	04/24/10 08:52
280-2756	2	J19WL8	Solid	280-132546	280-2756-A-2-A						
280-2756	2	J19WL8	Solid	280-134155	280-2756-A-2-B						
280-2756	2	J19WL8	Solid	280-155232	280-2756-A-2-C						
280-2756	2	J19WL8	Solid	280-126045	280-2756-B-2	Soil jar 4oz	AH66	Chavez, Lawrence	I	280-13864	05/25/10 12:38
280-2756	2	J19WL8	Solid	280-126045	280-2756-B-2	Soil jar 4oz	OP Dpt	Pottruff, Erma J	I	280-12627	05/14/10 16:02
280-2756	2	J19WL8	Solid	280-126045	280-2756-B-2	Soil jar 4oz	OP Dpt	Pottruff, Erma J	I	280-12624	05/14/10 15:45
280-2756	2	J19WL8	Solid	280-126045	280-2756-B-2	Soil jar 4oz	OP Dpt	Decker, Susan H	I	280-12603	05/14/10 14:11
280-2756	2	J19WL8	Solid	280-126045	280-2756-B-2	Soil jar 4oz	128	Johnson, Aaron S	I	280-12354	05/12/10 22:18
280-2756	2	J19WL8	Solid	280-126045	280-2756-B-2	Soil jar 4oz	OP Dpt	Johnson, Aaron S	I	280-12286	05/12/10 15:24
280-2756	2	J19WL8	Solid	280-165104	280-2756-B-2-A						
280-2756	2	J19WL8	Solid	280-126046	280-2756-C-2	Soil jar 4oz	AH66	Chavez, Lawrence	I	280-13864	05/25/10 12:38
280-2756	2	J19WL8	Solid	280-126046	280-2756-C-2	Soil jar 4oz	128	Johnson, Aaron S	I	280-10311	04/30/10 01:16
280-2756	2	J19WL8	Solid	280-126046	280-2756-C-2	Soil jar 4oz	OP Dpt	Johnson, Aaron S	I	280-10172	04/29/10 15:27
280-2756	2	J19WL8	Solid	280-126046	280-2756-C-2	Soil jar 4oz	128	Skrip, Sean P	I	280-9923	04/29/10 15:52
280-2756	2	J19WL8	Solid	280-126046	280-2756-C-2	Soil jar 4oz	OP Dpt	Skrip, Sean P	I	280-9812	04/29/10 09:27
280-2756	2	J19WL8	Solid	280-134360	280-2756-C-2-A						

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SECTION 24

ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, solvent extraction, sonication, acid digestion, filtration, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

Table 24-1. Negative Controls

Control Type	Details
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis. Also further detail is provided in SOP No. PT-QA-021.

24.3.1 **Negative Controls for Microbiological Methods** – Microbiological Methods utilize a variety of negative controls throughout the process to ensure that false positive results are not obtained. These controls are critical to the validity of the microbiological analyses. Some of these negative controls are:

Table 24-2. Negative Controls for Microbiology

Control Type	Details
Sterility Checks (Media)	are analyzed for each lot of pre-prepared media, ready-to-use media and for each batch of medium prepared by the laboratory.
Filtration Blanks	blanks are run at the beginning and end for each sterilized filtration unit used in a filtration series. For pre-sterilized single use funnels a sterility check is performed on at least one funnel per lot.
Sterility checks (Sample Containers)	are performed on at least one container per lot of purchased, pre-sterilized containers. If containers are prepared and sterilized by the laboratory, one container per sterilization batch is checked. Container sterility checks are performed using non-selective growth media.

Sterility Checks (Dilution Water)	are performed on each batch of dilution water prepared by the laboratory and on each batch of pre-prepared dilution water. All checks are performed using non-selective growth media.
Sterility Checks (Filters)	are also performed on at least one filter from each new lot of membrane filters using non-selective growth media.

Negative culture controls demonstrate that a media does not support the growth of non-target organisms and ensures that there is not an atypical positive reaction from the target organisms. Prior to the first use of the media, each lot of pre-prepared selective media or batch of laboratory prepared selective media is analyzed with at least one known negative culture control as appropriate to the method.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

24.4.1.1 The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

24.4.1.2 The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard may be reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

24.4.1.3 Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

24.4.1.4 The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-3 Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.

Table 24-3 Sample Matrix Control

Control Type	Details	
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

24.6.1 As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

24.6.2 Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

24.6.3 Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

24.6.3.1 Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

24.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

24.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

24.6.3.4 The maximum acceptable recovery limit will be 150%.

24.6.3.5 The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

24.6.3.6 If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no effect on laboratory ability to meet the existing limits.

24.6.4 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. Refer to laboratory SOP No. PT-QA-021.

24.6.4.1 The Reference Data Summary generated from LIMS shows the precision and accuracy acceptability limits for analyses performed. This summary includes an effective date, is updated each time new limits are generated and is located in LIMS. Unless otherwise noted, limits are laboratory generated. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Team Leader/Area Supervisor and QA Manager) and entered into the Laboratory Information Management System (LIMS). Further details are described in Pittsburgh SOP No. PT-QA-021.

24.6.5 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

24.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

24.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit. For further detail refer to SOP PT-QA-021 and method specific SOPs. For DoD requirements refer to SOPs PT-QA-25 and PT-QA-029.

24.6.5.3 For TNI work, there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Though marginal exceedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.6 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated parent sample are reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in SOP No. PT-QA-021 – Laboratory Quality Control Program, analytical method SOPs and in Section 12 of this document.

24.6.7 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

24.7.1 The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

24.7.2 A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

24.7.3 Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

24.7.4 Selection of appropriate reagents and standards is included in Section 9 and 21.

24.7.5 A discussion on selectivity of the test is included in Section 5.

24.7.6 Constant and consistent test conditions are discussed in Section 18.

24.7.7 The laboratories sample acceptance policy is included in Section 23.

SECTION 25

REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7. A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report) on the cover page with a "Result" column header on the sample result page.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. job Number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ## at the bottom of the page. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) and time of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Reporting Limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits are included unless the client specifies they do not require reporting the QC.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda). The temperature is documented on the sample receipt checklist and noted in the report case narrative.

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator .

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.22 If applicable, the laboratory includes a cover letter.

25.2.23 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.24 When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

25.2.25 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.26 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary report). A complete report must be sent once all of the work has been completed.

25.2.27 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory's accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

Note: It is required by the PA DEP that non-accredited parameters be clearly identified on the sample results.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level Or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above.
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services in addition to the test report as described in section 25.2. When NELAP accreditation is required and both a test report and

EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. Data qualifiers appearing on the test report must be included in the EDD.
Pittsburgh offers a variety of EDD formats including Excel, Equis, Giskey, CSV or others as requested by the client.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information For Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

25.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as ‘estimated’.

25.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

25.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client’s instructions so require.

25.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.
Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the Manager(s)/Team Leaders or as assigned by the lab Director. This is the only form of “interpretation” of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP # CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer.

25.7 Format Of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments To Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the data server, as is the original report. The revised report is stored in the data server under the job number followed by "Rev (n)" where 'n' is the revision number. The revised report will have the words "Revision (n)" on the report cover page beneath the report date. Additionally, a section entitled "Revised Report" will appear on the Case Narrative page. A brief explanation of the reasons of the re-issue will be included in this section.

25.9 Policies On Client Requests For Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

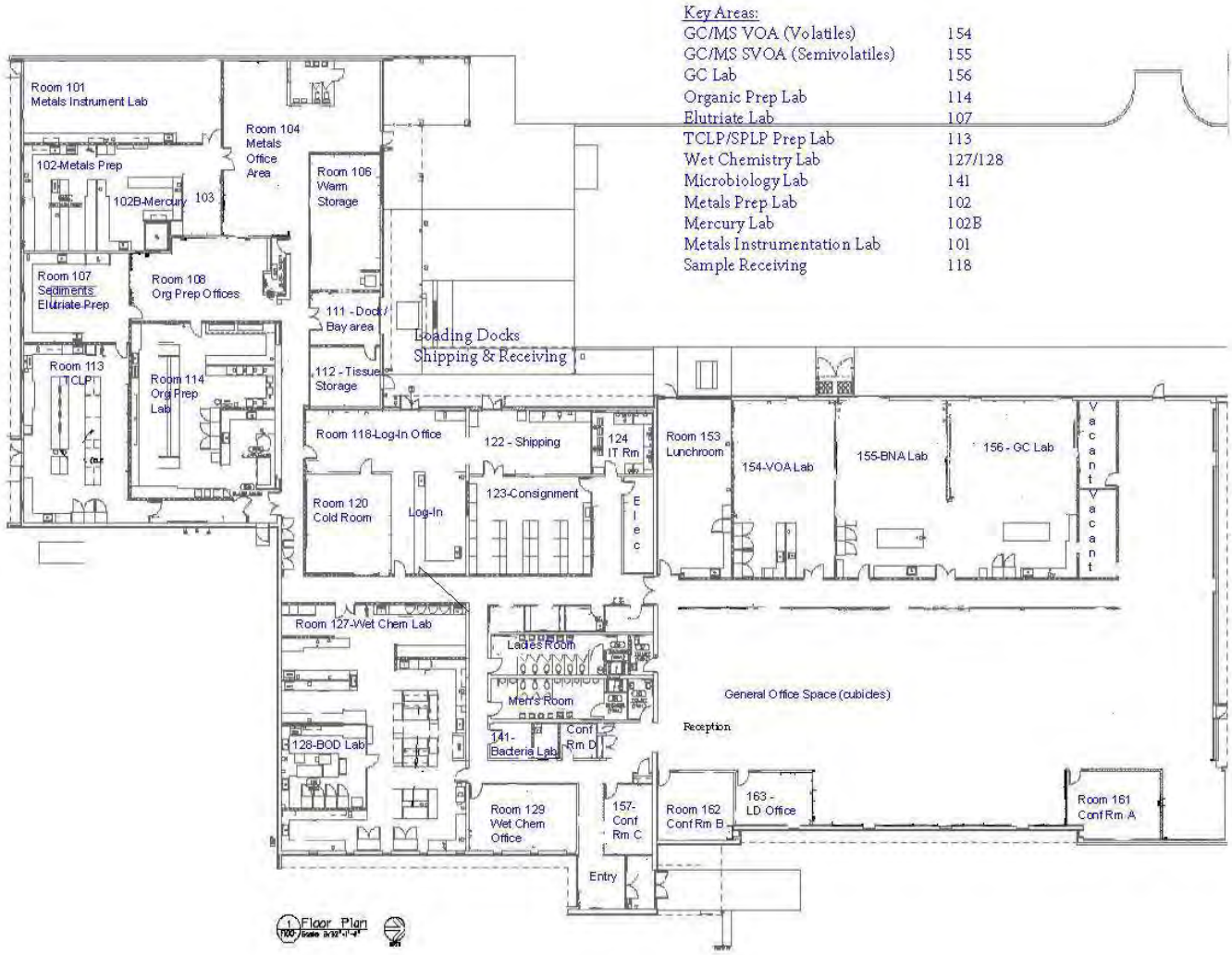
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels)
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same job number where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1
Laboratory Floor Plan



Appendix 2
Glossary/Acronyms

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (TNI)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form**:** Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction*:* Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (TNI)

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times : The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method, or more frequently if so required by the reference method, except for analytes for which spiking solutions are not available such as, total volatile solids, odor, temperature, or dissolved oxygen. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD): [a.k.a., Method Detection Limit (MDL]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification [a.k.a., MDL Verification]**:** A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air *& Emissions*: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.
Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance**:** An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): 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. (TNIS)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment,~~,~~ reporting and quality improvement to ensure that a process, item, ~~product~~ or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting NELAC and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank:
A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP: Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3

Laboratory Certifications, Accreditations, Validations

Pittsburgh maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation / certification / licensing with the following organizations:

Organization	Certificate Number Or Laboratory ID Number
Arkansas	88-0690
California ELAP	2891
Connecticut	PH-0688
Florida	E871008
Illinois	002602
Kansas	E-10350
Louisiana	04041
New Hampshire	203010
New Jersey	PA005
New York	11182
North Carolina	434
Pennsylvania	02-00416
South Carolina	89014002
Texas	T104704528
Utah	STLP
USDA	P330-10-00139
USDA	P-Soil -01
Virginia VELAP	460189
West Virginia	142
Wisconsin	998027800

The certificates and parameter lists (which may differ) are available, upon request, from a laboratory representative. They may be found on the corporate web site, the laboratory's public server and in the QA web page.

Appendix 4
Pittsburgh Laboratory SOP List

CA- and CW- indicate TestAmerica Corporate documents
PT- documents are specific to TestAmerica Pittsburgh

Document No.	Title
CA-C-S-001	Work Sharing Process
CA-C-S-002	Complaint Handling and Service Recovery
CA-I-P-002	Electronic Reporting and Signature Policy
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CA-L-S-002	Subcontracting Procedures
CA-L-S-001	Internal Investigation of Potential Data Discrepancies & Determination for Data Recall
CA-Q-M-002	Corporate Quality Management Plan
CA-Q-QM-002	Policy on GC/MS Tuning for Full Scan Volatile and Semi-Volatile Methods
CA-Q-QM-003	Technical Guidance on Reporting of Multi-Component Organochlorine Analytes
CA-Q-QM-004	Technical Guidance on Checking for Spectral Interferences in Optical ICP analysis
CA-Q-QM-006	Technical Guidelines for Analysis of Complex GC/ECD Chromatograms
CA-Q-QM-007	Guidance on the Digestion and Final Volumes for CVAA Mercury Methods
CA-Q-S-001	Solvents and Acids Lots Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Internal Auditing
CA-Q-S-005	Calibration Curves (General)
CA-Q-S-006	Detection Limits
CA-Q-S-007	Remote Data Processing
CW-Q-S-004	Management Systems Review
CA-Q-WI-015	Work Instruction for Electronic Chromatography File Surveillance using Mint Miner Manual Integration Data Mining Tool
CA-T-P-001	Qualified Products List

Document No.	Title
CA-T-P-002	Selection of Calibration Points
CA-T-P-003	Reporting Results for Methods that Require Second Column Confirmation
CW-L-P-002	Subpoenas Policy
CW-L-P-003	Organizational Conflicts of Interest
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure
PT-GC-001	Gas Chromatographic Analysis of Herbicides, SW-846 Method 8151A
PT-GC-002	Analysis of Organochlorine Pesticides and PCBs by Method 608
PT-GC-004	1,2-Dibromoethane(EDB) and 1,2-Dibromo-3-Chloropropane(DBCP) in Water by Microextraction and Gas Chromatography, Method 8011
PT-GC-005	Polychlorinated Biphenyls (PCBs) and PCBs as Congeners by GC/ECD - Method: SW-846 8082 and 8082A
PT-GC-006	Chlorinated Pesticides - Method: SW-846 8081A/B
PT-GC-007	Organophosphorus Pesticides by Gas Chromatography - Method: SW-846 8141A and 8141B
PT-GC-009	Determination of Inorganic Anions by Ion Chromatography EPA Method 300 SW-846 Method 9056A
PT-GC-010	TOC Analysis for Solids by Lloyd Kahn Method
PT-GC-011	Ethylene Glycol – 8015C/D
PT-GC-WI-001	IC Dilution Table Based on Conductivity
PT-GC-WI-002	GC Data Review Checklist
PT-GC-WI-003	IC Data Review Checklist
PT-GC-WI-004	Safety Kleen Waste Dilution (GC-ECD)
PT-GC-WI-005	Silica Gel Cleanup Work Instruction for PCB Extracts – Method 3630C
PT-HS-001	Pittsburgh Facility Addendum to TestAmerica Corporate Environmental Health & Safety Manual (CW-E-M-001)
PT-HS-002	Bloodborne Pathogen Exposure Control Path
PT-IP-002	Acid Digestion of Soils, SW-846 Method 3050B
PT-IP-003	Acid Digestion of Aqueous Samples by SW-846 Methods 3005A, 3010A and EPA Methods 200.7 and 200.8
PT-IP-W-001	Metals Prep Guide - TA Pittsburgh
PT-IP-W-002	Metals pH Check

Document No.	Title
PT-IT-001	Data Backup Procedures
PT-MS-001	GCMS Analysis Based on Method 8270C and 625
PT-MS-002	Volatile Organics by GC/MS Based on Methods 8260B, 624
PT-MS-005	VOA Holding Blanks
PT-MS-007	GCMS Volatile Organic Analysis by EPA CLP SOW OLM04.2
PT-MS-008	GC/MS Analysis, Method: SW-846 8270D
PT-MS-010	Determination of Volatile Organics by GC/MS Method: SW-846 8260C
PT-BNA-WI-001	BNA ICAL Review Checklist
PT-BNA-WI-002	BNA Data Review Checklist
PT-VOA-WI-001	GCMS VOA Backflushing OI Eclipse
PT-VOA-WI-002	GCMS VOA Encore Prep Procedure
PT-VOA-WI-003	GCMS VOA Low Level Soil Extraction Logsheet
PT-VOA-WI-004	GCMS VOA Medium Level Soil Extraction Logsheet
PT-VOA-WI-005	Soil Volatiles – Training Aid and Work Instruction for Data Entry
PT-MT-001	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW-846 Method 6010B, 6010C and EPA Method 200.7
PT-MT-002	Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICPMS) for Methods 200.8, 6020, 6020A & ILM05.2
PT-MT-005	Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1
PT-MT-007	Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, SW846 7471A & 7471B
PT-MT-WI-001	Mercury Data Review Checklist
PT-MT-WI-002	HYDRA II Operating Instructions
PT-MT-WI-003	ICP Data Review Checklist
PT-MT-WI-004	ICPMS Data Review Checklist
PT-MT-WI-005	6800 Data Review Checklist
PT-MT-WI-006	Metals Calculated Methods Data Review Checklist
PT-OP-001	Extraction and Cleanup of Organic Compounds from Waters and Solids, Based on SW-846 3500 Series, 3600 Series, 8151A and 600 Series
PT-OP-002	Simplified Laboratory Runoff Procedure (SLRP)

Document No.	Title
PT-OP-003	Standard Elutriate Test (SET)
PT-OP-004	Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure
PT-OP-005	Modified and Effluent Elutriate Tests (MET and EET)
PT-OP-006	Long Tube Column Settling Test
PT-OP-007	Illinois Resuspension Tests
PT-OP-008	Dredging Elutriate Test (DRET)
PT-OP-009	Sequential Batch Leach Test (SBLT) for Freshwater Sediments
PT-OP-011	Extractable Residue (Lipids) from Animal Tissue
PT-OP-015	Modified Multiple Extraction Procedure Method: SW-846 1320
PT-OP-016	Porewater Generation
PT-OP-017	Liquid-Solid Partitioning as a Function of Extract pH in Solid Materials using a Parallel Batch Procedure SW-846 Method 1313
PT-OP-018	Liquid-Solid Partitioning as a Function of Liquid-to-Solid Ratio in Solid Materials using a Parallel Batch Procedure SW-846 Method 1316
PT-OP-019	Mass Transfer Rates of Constituents in Monolithic or Compacted Granular Materials using a Semi-Dynamic Tank Leaching Procedure SW-846 Method 1315
PT-OP-020	Standard Test Method for 24-h Batch-Type Measurement of Contaminant Sorption by Soils and Sediments ASTM D4646-03
PT-OP-021	Liquid-Solid Partitioning as a Function of Liquid-to-Solid Ratio for Constituents in Solid Materials using an Up-Flow Percolation Column Procedure SW-846 Method 1314
PT-OP-022	Low Volume Extraction and Cleanup of Organic Compounds from Waters Method: SW846 3500 Series
PT-OP-023	Measurement of the Leachability of Solidified Wastes by a Short Term Test Procedure Modified ANSI/ANS-16.1-2003
PT-OP-025	Soil/Sediment Amendment Procedure
PT-LP-W-001	TCLP Amber Glass Extraction Blank Vessel Tracking Logbook
PT-LP-W-002	TCLP Zero Headspace (ZHE) Extraction Blank Vessel Tracking Logbook
PT-LP-WI-003	TCLP/ZHE Tumbler RPM Logsheet
PT-PM-001	Project Information Requirements
PT-PM-W-001	Bottle Kit Preservation Guide
PT-PM-WI-002	QAPP Review Checklist
PT-PM-WI-003	QAPP Review Instructions

Document No.	Title
PT-PM-WI-004	Shipping Reports
PT-PM-WI-005	Report Completeness Checks
PT-QA-001	Employee Orientation & Training
PT-QA-M-001	Pittsburgh Laboratory Quality Assurance Manual
PT-QA-002	Internal Auditing
PT-QA-003	Glassware Clean-up for Organic/Inorganic Procedures
PT-QA-004	Quarantine Soil Procedure
PT-QA-005	Measurement Uncertainty
PT-QA-006	Procurement of Standards and Materials; Labeling and Traceability
PT-QA-007	Detection Limits
PT-QA-008	Thermometer Calibration and Temperature Monitoring
PT-QA-009	Rounding and Significant Figures
PT-QA-010	Tracking, Review and Revision of SOPs
PT-QA-011	Data Recording Requirements
PT-QA-012	Selection and Calibration of Balances and Weights
PT-QA-013	Independent QA Data Review
PT-QA-014	Reporting Limits
PT-QA-015	Maintaining Time Integrity
PT-QA-016	Nonconformance & Corrective Action System
PT-QA-017	Aqueous Pipette Calibration – Gravimetric Method
PT-QA-018	Technical Data Review Requirements
PT-QA-019	Records Information Management
PT-QA-020	Report Production
PT-QA-021	Quality Assurance Program
PT-QA-022	Equipment Maintenance
PT-QA-024	Subsampling

Document No.	Title
PT-QA-026	Container Accuracy Verification – Gravimetric
PT-QA-030	Sample Management and Tracking for Cold and Warm Storage
PT-QA-031	Internal Chain of Custody
PT-QA-WI-002	Master Document List
PT-QA-WI-003	BNA Dilution Calculation Table
PT-QA-WI-004	VOA Dilution Calculation Table
PT-QA-WI-005	GC Dilution Calculation Table
PT-QA-WI-006	Metals Dilution Calculation Table
PT-QA-WI-007	Wet Chem Dilution Calculation Table
PT-QA-WI-008	Work Instruction for Vial Calibration for Sample Extracts
PT-QA-WI-009	RSK Dilution Calculation Table
PT-QA-WI-010	Work Instruction for the MiniiiIR Thermometer
PT-QA-WI-011	Work Instruction for the BASF Monaca PA Facility
PT-QA-WI-012	Work Instruction for the Ergon Newell WV Facility
PT-QA-WI-013	Work Instruction for the Mountain State Carbon Follansbee WV Facility
PT-QA-WI-014	Work Instruction for the Ohio Coatings Yorkville OH Facility
PT-QA-WI-015	Work Instruction for the Ormet Reduction Mill Hannibal Ohio Facility
PT-QA-WI-016	Work Instruction for the TIMET Toronto Ohio Facility
PT-QA-WI-017	Work Instruction for Hotblock Thermomemter Rotation - Diagram 1
PT-QA-WI-018	Work Instruction for Hotblock MB Rotation - Diagram 2
PT-QA-WI-019	Work Instruction for Hotblock MB Rotation - Diagram 3
PT-QA-WI-020	Work Instruction for Hotblock MB Rotation - Diagram 4
PT-QA-WI-021	Work Instruction for Temperature Probe Rotation - Sulfide Distillation Block Diagram
PT-QA-WI-022	Analytical SOP Review Checklist
PT-QA-WI-023	SOP Change Form (Amendment Form)
PT-QA-WI-024	PT Corrective Action/Preventative Action Narrative Checklist

Document No.	Title
PT-QA-WI-025	Non-Analytical SOP Review Checklist
PT-QA-WI-026	Sample Receiving Short-Hold Parameter
PT-QA-WI-027	Temperature Logbook
PT-QA-WI-028	Management of Change Request form
PT-QA-WI-029	Spreadsheet Validation Form
PT-QA-WI-030	Signature Log
PT-QA-WI-031	Corrective Action Report (for external use)
PT-QA-WI-032	Work Instruction for Data Recall Notification
PT-QA-WI-033	DOC Certification Statement (No Spikes)
PT-QA-WI-034	Work Instruction: Annual Working Weight Calibration Report
PT-QA-WI-035	QA Technical Data Review
PT-QA-WI-036	IDOC Limits
PT-QA-WI-037	QA Orientation Checklist
PT-QA-WI-038	QA Training Acknowledgement Form
PT-QA-WI-039	Employee Record of Training Form
PT-QA-WI-040	One on One Cross-Training Form
PT-QA-WI-041	Employee Record of SOP Training Review Form
PT-QA-WI-042	Training Attendence Roster
PT-QA-WI-043	Thermocouple Calibration Form
PT-QA-WI-044	DOC Form with LCS
PT-QA-WI-045	TestAmerica Pittsburgh Equipment List
PT-QA-WI-046	Instrument Information Form
PT-QA-WI-047	Proficiency Testing Procedures
PT-QA-WI-048	QA Data Package Review Form
PT-QA-WI-049	Non-Conformance & Corrective Action Tracking
PT-QA-WI-050	Organics "D" Qualifier Dilution Levels

Document No.	Title
PT-QA-WI-051	Analytical Balance Calibration Verification Limits
PT-QA-WI-052	Top-Loader Balance Calibration Verification Limits
PT-QA-WI-053	MDL/MDLV Login
PT-QA-WI-054	Meeting Attendance Roster
PT-QA-WI-055	Control Chart Creation
PT-QA-WI-056	Manual Integration Training Quiz
PT-QA-WI-057	Monthly Balance Verification
PT-QA-WI-058	Manual Data Qualification in TALS AD
PT-QA-WI-059	Glassware Clean-Up for Organic Prep Procedures
PT-QA-WI-060	Glassware Clean-Up for VOA's
PT-QA-WI-061	Glassware Clean-Up for Metals
PT-QA-WI-062	Glassware Clean-Up for General Chemistry
PT-QA-WI-063	Glassware Clean-Up of ZHE's
PT-SR-001	Sample Receipt & Login
PT-SR-002	Bottle Order Preparation and Shipping
PT-SC-WI-001	VOA Freezing Log
PT-SR-WI-001	Cooler Temperature
PT-SR-WI-002	Work Instruction for Bottle Order Creation in TALS
PT-SR-WI-003	Sample Send Outs
PT-SR-WI-004	Pike-Up/Delivery Request Form
PT-SR-WI-005	TestAmerica Pittsburgh Sample Acceptance Policy
PT-SR-WI-006	Metals Analyte List
PT-SR-WI-007	Short Hold Parameters
PT-SR-WI-008	Wet Chemistry QC – MS/MSD NOT Required
PT-SR-WI-009	How to log in Safety Kleen Recycle
PT-SR-WI-010	How to log in Safety Kleen Recharacterization

Document No.	Title
PT-SR-WI-011	International Shipping Guidance Documents
PT-SR-WI-012	Work Instruction for Screening Samples for Radioactivity
PT-SR-WI-013	PM to Login Job notes
PT-WC-001	Determination of Total and Total Volatile Solids in Waters and Wastes (Methods EPA160.4 and SM 2540B & 2540E)
PT-WC-002	Color, Method 110.2
PT-WC-003	Alkalinity, SM Method 2320B
PT-WC-004	Total Hardness (mg/L as CaCO3) by Method SM 2340C; and Hardness by Calculation SM 2340B
PT-WC-005	Turbidity by Method 180.1
PT-WC-006	Determination of Chlorine Contamination in Used Oil Using CLOR-D-TECT 1000 Used Oil Screening Kit, SW-846 Method 9077 and ASTM Method
PT-WC-007	Nitrate/Nitrite, Nitrite, EPA Method 353.2
PT-WC-008	Acid Volatile Sulfides (AVS) and Simultaneously Extracted Metals (SEM) in Sediment
PT-WC-009	Performance Checks on Spectronic 21 and Model 1001 Spectro-Photometers
PT-WC-010	Total Sulfide as Acid Soluble Sulfide, Method 9030B/9034, Standard Method 20th Ed. 4500S-2-F
PT-WC-012	pH, Specific Conductance and Alkalinity (Automatic Titrator)
PT-WC-013	Specific Conductance by 120.1, 2510B, and 9050A
PT-WC-014	Nitrogen, Ammonia (Automated), Method 350.1
PT-WC-015	Chromium, Hexavalent (Colorimetric) by SM3500-Cr-B, SW846 3060A/7196
PT-WC-016	Biochemical Oxygen Demand (BOD) and Carbonaceous Biochemical Oxygen Demand (CBOD) by Dissolved Oxygen Probe - SM5210B
PT-WC-017	Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC), Methods SM 5310C and SW-846 9060/9060A
PT-WC-018	Cyanide – Semi-Automated, Pyridine-Barbituric Acid For Total and Amenable, Cyanide in Water (Methods 335.4) and Soil Analyses (Method
PT-WC-020	Percent Moisture, Ash, Organic Matter and Total Solids in Soil Samples - SM 2540G and ASTM D297-84
PT-WC-021	Flash Point by Pensky-Martens Closed Tester, SW-846 Method 1010A and ASTM D93-08
PT-WC-022	Ignitability of Solids for Waste Characterization EPA SW-846 Chapter 7, Section 7.1
PT-WC-023	Chemical Oxygen Demand, Low Level, Method 410.4
PT-WC-025	n-hexane extractable material (HEM) in Sludge, Sediment and Soil samples - 9071B
PT-WC-026	PH Electrometric by SM 4500 H+B and SW-846 Methods: 9045C/D and 9040B/C

Document No.	Title
PT-WC-027	Salinity by Calculation, Electrical Conductivity Method SM 2520B
PT-WC-028	Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated Hexane Extractable Material (SGT-HEM; TPH), Method 1664A and 9070A
PT-WC-029	Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA) Method 1677
PT-WC-031	Cyanide Extraction Procedure for Solids and Oils, SW-846 Method 9013
PT-WC-032	Total Organic Carbon Analysis for Solid Matrices by Walkley Black
PT-WC-033	DI-Leachate Procedure for Solids (1 Hour Routine DI Leachate and 18 Hour ASTM DI Leachate Procedure)
PT-WC-034	Paint Filter Liquids Test, SW-846 Method 9095B
PT-WC-035	Acidity of Water and Waste Water, SM Method 2310B
PT-WC-036	Flash Point of Liquids by Setaflash (Small Scale) Closed-Cup Apparatus, SW-846 Method 1020B and ASTM Standard D 3278-96
PT-WC-037	Oxidation Reduction Potential, SM 2580B (20th Ed)
PT-WC-038	Phenolics (Automated), Method 420.1/420.2, SW-846 9065/9066
PT-WC-039	Screening Apparent Specific Gravity and Bulk Density of Waste - Method: ASTM D 5057-90
PT-WC-040	Anion Surfactants as MBAS, Standard Methods 5540C
PT-WC-041	Compatibility of Screening Analysis of Waste, Method: ASTM D5058 Test Method C – Water Compatibility
PT-WC-042	Acid Titration of Samples for Bechtel Bettis (NaOH Solution for Caustic and Na2CO3 Concentration), Bechtel-Bettis SOP - WAPD-MT(CAC)-2141,
PT-WC-043	Determination of Total and Volatile Suspended Solids in Waters and Wastes - EPA 160.4 and SM 2540D & 2540E
PT-WC-044	Determination of Settleable Solids in Water SM 2540F
PT-WC-045	Determination of Total Dissolved and Volatile Dissolved Solids in Water and Wastes, SM 2540C & 2540E
PT-WC-WI-001	UV-Absorbing Constituents Method SM5910B
PT-WC-WI-002	ORP Form - Eh/pH Phase Diagram
PT-WC-WI-003	Manual BOD Log Sheet Method: SM 5210B
PT-WC-WI-004	MBAS ICAL Extraction Worksheet
PT-WC-WI-005	MBAS Daily Extraction Worksheet
PT-WC-WI-006	Wet Chemistry Data Review Checklist
PT-WC-WI-007	Waste Visual Compatibilty Test @ Ambient Temperature Worksheet
PT-WC-WI-008	UV/Vis Spectrophotometer Performance Validation Worksheet

Document No.	Title
PT-WC-WI-009	OIA-1677 Instrument Stability Check Worksheet
PT-WC-WI-010	Gravimetric-Solids Methods Data Review Checklist
PT-WC-WI-011	Gravimetric-Oil & Grease Data Review Checklist
PT-WC-WI-012	Automated BOD Log Sheet
PT-MICRO-WI-001	Incubator Temperature Log
PT-FS-001	Field Measurement of Dissolved Oxygen (DO) Method: SM 4500-O G
PT-FS-002	Field Measurement of Total Residual Chlorine Method: SM 4500-Cl G
PT-FS-003	Field Measurement of pH Method: SM 4500 H+B
PT-FS-WI-001	Work Instruction for Courier Service

Note: The SOPs are subject to change, refer to PT-QA-WI-002 for current list of SOPs and revision numbers.

Appendix D
Laboratory Standard Operating Procedures

Contents

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

QUALITY SYSTEMS: QS10 **REVISION #: 26** **EFFECTIVE DATE: 20170524**

LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

APPROVALS:

Lab Director: _____ Date: **20170524**

Director of Finance and Administration: _____ Date: **20170524**

Data Quality Manager: _____ Date: **20170524**

QS10_R26_20170524_RCVG

- Updated sections 2.4, 2.5, 3.3, 3.3.2.6, 3.3.4, 5.1.23 and 12 for use of cooler receipts documentation in LIMS replacing manual form with issues documented in sample, analysis or container comments.
- Update section 4.0 for change in metals storage of samples from walk-in cooler to dry rack storage.
- Updated tables for quarantined areas of the U.S. in appendix IV (a,b,c).
- Updated cooler custody form in appendix V.

QS10_R25_20161121_RCVG

- Update sections 3.3.5-3.3.7 for residual chlorine testing including addition of 625 samples and updated test strips.
- Update cooler receipt form to add residual chlorine testing for 625.

QS10_R24_20161108_RCVG

- Update cooler receipt form to add a space for "Therm. ID".
- Update section 3.3 to allow temporary VOC vial storage in the Receiving VOA refrigerator.
- Update signature page to exchange Director of Finance and Administration for Group Leader.

QS10_R23_20151202_RCVG

- Section 2.7 and 3.0 updated to indicate removal of all non-volatile samples while volatile samples are to remain in the cooler until ready to label.
- Section 11 updated to reflect pulling samples using sub-out COC and second check of samples against sub-out COC with initial/date.

QS10_R22_20150924_RCVG

- All references to section supervisor updated to reflect group leader.
- Section 2.7.1 updated to remove reference to alternate sample lineup.
- Section 3 updated to reflect LIMS login and sample labeling.
- Section 3.2.3 inserted to indicate separate sample number for dissolved or TCLP with total, too.
- Section 3.3.5 updated for residual chlorine.
- Section 3.3.6 inserted to require pH and residual chlorine check for EPA 608.
- Section 6 updated for COC distribution and 6.5.2 removed – redundant.
- Section 10 updated to reflect QS14.
- Added "Notes on any corrections:" to section III of CRF form.

QS10_R21_20140729_RCVG

- Section 5.2 updated to reference expanded attachment II.
- Section 5.4 added to indicate requirement for PM to complete attachment III and add to COC documentation.
- Attachment II expanded to include checklist for LIMS data entry.
- Attachment III inserted for LIMS data entry verification by PMs.
- Attachments IV and V renumbered from III and IV.

Revision 20, 09/03/2013:

- Sections 2.5.3.4 and 7.3 referencing Aquatic Toxicology samples removed.
- Excluded oil and grease from pH checks in section 3.3.1.1 and removed reference to oil and grease in section 3.3.2.3
- Added section 3.3.6 requiring that any sample adjustment be noted within the LIMS.
- Updated section 4.1.1 to reflect VOC Water Refrigerator, added listing for segregated VOC Soil area and updated section 8.2.3 to indicate segregated VOC soil area.
- Placed all VOC storage prior to water and soil walk-in except for VOC Dry Storage Rack. Added Storage Blank reference to all VOC cold storage areas.
- Added Truck Maintenance Log to list of log books maintained by SR.
- Updated Attachment references throughout document and updated references to Sample Change Form to indicate e-mail notification.
- Removed list of holding times and preservations - added reference to table in QS11 in section 2.6.2.
- Removed Container Codes Attachment for the LIMS and "Green Sheet" for LIMS corrections (Old System).
- Updated Soil Movement Map and Custody Log. Added maps specific to fire ants and nematodes.

Revision 19, 09/17/2012

- Removed section 3.3.2.7

- Updated section 5.2 and 9.1.2 to reflect second checking of primary login information with update to SRC form.
- Updated personnel references to reflect Sample Receiving Personnel, Group Leader, Section Supervisor, Project Manager and Lab Director.
- Removed information for thermometer calibration requirement in section 2.4 and container preservation/traceability in section 3.3.3, as they are covered in other SOPs.
- Removed redundant statements from section 2.1, 3.3 and 3.4.
- Reference to sample number reset at the beginning of the year removed from section 3.2.
- Removed reference to pH paper method 9041A in section 3.3.3.2.
- Added section 3.3.4.5.3 requirement to note date/time of preservation in comment section.
- Removed reference to Na2S2O3 preservation in section 3.3.6.
- Updated refrigerator tolerance range to 0°C-6°C in section 4.5.
- Removed reference to back up compressor in section 4.6.
- Updated sample retention to 45 days from sample receipt as is consistent with all proposal notes.
- Section 5.1.2 updated to add "**Note – correct/accurate selection is absolutely critical to the successful analysis, reporting and invoicing of all samples."**
- Section 5.1.19 updated to make sample receiving personnel contact PM when appropriate test code is not available in the selected project.
- Reference to temperature monitoring of the walk-in coolers has been updated in section 4 to include TempAlert system.
- Section 6.4 updated to remove items associated to previous LIMS system.
- Log book IDs removed from section 7.4.
- Sample Receiving Custody and Disposal Form removed from SOP as Custody is maintained in the LIMS system and disposal is handled separately.
- Attachment identifications updated and Short Holding Time Parameters attachment replaces with List of Holding Times/Preservations.

Revision 18, 09/16/11
- The cooler receipt form was revised.
- Added requirement that samples must be refrigerated within 2 hours of opening cooler.
- Added cooler receipt form completion requirement upon opening cooler.
- Updated procedure for pH measurement.
- Changed default time to 00:01 in the case a time is not listed on COC.
- Removed reference to ATSD in SOP 187.

Revision 17, 05/16/11
- The list of employees has been removed from section 8.

Revision 16, 11/17/10
- Added requirement "Notes to sample analytical comments indicating date/time/initials preserved." for metals samples preserved within house.

Revision 15, 10/13/10
- Updated CRF on page 23, also added at the beginning of page 6 statement about recording the final temperature value.

Revision 14, 09/07/10
- The SOP combines SOPs 404, 406, 410, 415 and 432 into one SOP with updated naming.

Table of Contents

1.0 Sample Acceptance Criteria

This SOP lists, in as much detail as possible, our daily procedures for sample receiving, log in and storage of laboratory samples. Keep in mind that there may be project specific requirements that are more strict or different than our routine procedures. In these instances, the project specific requirements must be met and followed. Although a few project specific requirements are detailed in this SOP, i.e. DoD requirements, not every situation can be addressed. If there is ever any uncertainty on what procedures must be followed, please see the Project Manager or your Group Leader immediately. If ever in doubt, always go with the more stringent requirements.

1.1 A sample may be rejected for compliance purposes if it does not meet the following criteria. Analyses may only proceed after notification and approval to proceed from the client or from the laboratory director.

1.1.1 Sample must be properly preserved and in the proper container for the requested analysis.

1.1.2 Sample integrity must be maintained. The container shall be intact without cracks, leaks, or broken seals.

1.1.3 Adequate sample volume must be received for the requested analysis, including volume for any requested QA/QC (MS/MSD).

1.1.4 The sample ID on the bottle label must match the sample ID listed on the chain of custody.

1.1.5 The sample container label and the chain of custody must be completed with indelible ink. The sample label must be intact and list all necessary information; to include: sample date, sample time, and sample ID/location. The chain of custody shall also indicate sample date and time, requested analyses, and all necessary client information.

1.1.6 Sample temperature must be less than 6°C or received on ice.

1.1.7 Sample must be within holding time for the requested analysis.

1.1.8 Samples should be refrigerated within 2 hours of being unloaded.

These issues are discussed in more detail below under the "Sample Receiving" section of this document.

2.0 Sample Receiving

2.1 Samples are received at the Empirical Laboratories on 621 Mainstream Drive, Suite 270 Nashville, TN 37228.

2.1.1 The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Empirical Laboratories Sample Receiving (SR) area loading dock in back of the laboratory. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we do pick up our coolers at the FedEx location and transport them back directly to the laboratory.

2.1.2 Some coolers and/or samples may be received directly by Empirical Laboratories Sample Receiving personnel. If samples are hand delivered by the client make sure that necessary paperwork is included and that you sign and date the chain of custody, as well as record the final value temperature of the samples on the chain of custody as well. If the *Empirical Laboratories Chain of Custody [Attachment I*] is used the white and yellow copy of the chain of custody is retained and the pink copy must be given to the client.

2.2 Sample receiving personnel must wear the following personal protection equipment: gloves, safety glasses and a laboratory coat.

2.3 Visually inspect all coolers for tampering, custody seals, (intact if applicable) leakage, etc. If a cooler has been damaged beyond repair, unpack the samples and discard the cooler as to not reuse it. If you suspect a cooler may be damaged or is extremely dirty this cooler must not be reused. If coolers were sent by Federal Express, examine the Federal Express air bills for the number of packages in the shipment and make sure that all the packages (coolers, boxes etc.) in a group have been received. If there are any problems the Project Manager must be contacted immediately. If anything looks unusual, take the time to check it out and document the situation and findings.

2.4 Open each cooler in order to quickly inspect the contents and to locate the chain of custody. Your signature and the date and time the samples were received must be placed onto the chain of custody. The time received must reflect the actual time the samples were received even though they may be logged into the system at a later time. Samples received late in the day during the week may be processed the next morning, however, all cooler(s) must be opened, examined (for leakage, breakage etc.), the temperature measured and the chain of custody signed and dated to reflect the actual date and time which they were received. The samples must be delivered to the appropriate analytical department or put in cold storage as soon as possible after unloading.

2.4.1 Attach any shipping receipts, work orders, documentation, etc. to the chain of custody.

2.4.2 If a chain of custody or other paperwork is not sent, the client must be contacted and the samples temporarily placed on hold in cold storage. The required information may be found on the sample containers or it may be necessary to call the client to get the missing information (i.e. sample ID, collection date and time, etc.).

2.5 The temperature of each cooler or set of samples must be measured as quickly as possible using a thermometer with 0.1°C increments.

2.5.1 To measure the temperature, point the IR gun at the cooler temperature blank (if supplied) or a direct sample and wait a few seconds for the temperature to stabilize. The IR gun should be held 6 inches away (from the temperature blank or sample) for an accurate reading. Read the temperature to the nearest 0.1 °C. The **final** temperature value must also be recorded on the chain of custody. (This value will also be recorded into the LIMS at a later point.). This is calculated by measuring the initial value temperature and adding the correction value temperature (IR temperature gun calibration factor) together, to obtain the final temperature value.

2.5.2 If the temperature exceeds 6°C for any sample, the Project Manager must contact the client immediately. There may be tighter temperature control limits for specific project requirements. The customer must make the decision to either continue with the analyses or resample. Make sure the client is aware that temperature exceedence will be narrated if the samples are analyzed. Not all samples for the project will be narrated, just those samples received above 6°C. Note: Many times we are not able to get in touch with the client quickly and the best judgment on how to handle the samples must be made after discussion with the Project Manager and/or Laboratory Director. The samples may still need to go through the log in process although it may be eventually determined that the samples will not be analyzed or the samples may temporarily be placed on hold and not logged in, with the exception of short hold and rush samples. **Above all, do not allow the samples to set out at room temperature for an extended period of time while waiting for a decision.**

2.5.3 The only exceptions to the 6°C rule are:

2.5.3.1 Water samples for all Metals, (except Chrome 6+ and mercury) that have been preserved with HNO_3 to a pH of ≤ 2. *Keep in mind that non-aqueous sample for Metals must be cooled.*

2.5.3.2 Samples for Fluoride, Chloride and Bromide.

2.5.3.3 Waste/Product samples for all parameters.

2.5.3.4 Samples collected locally by Empirical Laboratories personnel or hand delivered by local customers. In some instances these samples may not have had time to cool down; however, these samples should have been placed on ice in an attempt to cool them to the proper temperature. This exception is only applicable if the samples were collected the same day as the laboratory receives them. It should be noted if samples are "Received on Ice" (ROI).

2.6 If several coolers are received at once, they must be inspected to determine the order in which the samples should be unpacked and logged in. The following priorities should be given:

2.6.1 Hexavalent chromium water samples which have a 24 hour holding time.

2.6.2 Any analyses which have a 24-72 hour holding time (See QS11 for table).

2.6.3 Any sample which has almost exceeded its' holding time. (Especially watch for this with waters organic extractions, Solids and Sulfides, all of which have only 7 days). A list of parameters and holding times is posted in sample receiving from QS11.

2.6.3.1 If a sample is received already out of holding time, this must be documented and the Project Manager must be contacted. The sample can be analyzed at the client's request, but it will be qualified on the final report as being received out of holding time. The Project Manager will inform you of the client's decision.

2.6.3.2 If a sample is received with limited holding time remaining for any parameter, it is necessary to contact the Project Manager so that he/she can contact the client. If the sample has to be analyzed on a rush basis to meet the holding time, a rush charge may apply. Also it may not be possible to analyze the sample within the holding time due to sample load, etc.

2.6.4 Samples requiring rush turnaround.

2.6.4.1 If sample(s) require 24 or 48-hour turnaround they will take first priority. Other rush requests also have high priority.

2.6.4.2 The Project Manager and/or Group Leader must be contacted for approval concerning any unscheduled rush requests.

2.7 Unpack all non-volatile samples from the cooler. All volatile samples are to remain in the cooler until labels are ready to be applied. If there are any known or suspected hazards this must be done under a hood. It may be necessary to rinse off the outside of the containers in the sink and/or wipe them off with a paper towel.

2.7.1 Visually inspect them for tampering and custody seals (if applicable). Sort and inventory the samples against the chain of custody by arranging them in the same order as they are listed on the chain of custody. Samples are assigned log numbers in the same order as they are listed on the chain of custody.

2.7.2 Check for leakage and sample container breakage as this could compromise the sample integrity. If any spillage occurred in the cooler make sure this is noted. Also list all the other samples in the cooler as cross contamination could occur.

The Project Manager and/or the customer must to be notified in these situations. It may be necessary to resample.

2.8 Check the chain of custody information against the information recorded on the containers. If these do not agree, this must be documented and the Project Manager must be notified.

2.8.1 If major changes are made on the chain of custody received from an engineering job, then the client should submit written confirmation of these changes or make the corrections and initial them directly on the chain of custody.

2.8.2 Any error requested for correction on the chain of custody must be marked through with one line, correction written, initialed and dated with the supporting documentation attached.

2.9 Note any unusual requests, methodology, hazards (known or suspected) to the Project Manager and/or Group Leader or analysts before the samples are actually logged in. Make notes of any problems (improper containers, preservatives, temperature, or descriptions, etc.).

3.0 Sample Log In

3.1 After all non-volatile samples have been unpacked, sorted and reviewed, they are then ready to be assigned log numbers and continue through the log in process. Make sure that the parameters for the samples are clearly marked on the chain of custody. Contact the Project Manager if there are any questions, problems, etc.

3.2 Samples are then logged into LIMS per section 5.0 and assigned unique ID numbers. Labels are printed from the LIMS and applied to each container. Volatile samples are removed from the cooler, lined up and labeled after all other containers have been labeled.

3.2.1 All containers with the same description must have the same sample number even if they have different preservatives and require different tests. However, each different fraction (bottle type and/or preservative) should be designated with a letter (A, B, C, etc.).

3.2.2 Grab and composite samples from the same sample location must be considered as separate samples. It may be necessary to use "grab" or "composite" as part of the sample description to distinguish between the samples. Only assign different log numbers to them if the parameters are clearly marked as grab and as composite. Do not assume that VOC must be analyzed from grab samples so therefore the client must have taken a grab sample.

3.2.3 Due to LIMS limitations, samples with both TCLP and total or dissolved and total analyses must be logged with separate numbers.

3.3 Check the following items and record this information into LIMS to further ensure sample integrity. If any of the following requirements are not met it may be necessary to contact the client. We can perform the analyses in most cases and will do so with the client's approval, however the results may be qualified in some manner on the final report.

Preserving sample integrity throughout the log in procedure must be one of our section's top priorities. This includes not only ensuring that the proper chemical preservatives have been added but also that the samples are received and maintained at the proper temperature. Samples must not sit out at room temperature if there is a delay. The samples must temporarily be placed in cold storage until you are able to complete the log in procedure. **[VOC containers may be temporarily stored in the Receiving VOA Refrigerator.]**

3.3.1 Check to determine if the proper chemical preservatives were added to adjust the sample to the correct pH. Any regulatory compliance sample received from North Carolina that does not meet the preservation requirement will be segregated

and the client will be notified of non-compliance. The samples will not be analyzed until notification to proceed with analyses is received from the client. A list of parameters and the required chemical preservatives is posted in the log-in room. The verification of this preservation will be recorded on the Cooler Receipt Form for all projects.

3.3.1.1 The pH of each container (except VOA vials or Oil and Grease samples) which requires pH preservation must be checked. Do not open and check the pH of VOA vials or Oil and Grease containers in sample receiving/log-in. This information is then documented on the project cooler receipt form.

3.3.1.2 The pH of preserved samples is checked and confirmed using pH narrow range indicator paper.

3.3.1.3 When taking the pH reading, DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Use capillary tube to measure pH. For some samples (wastes) the indicator paper may not be accurate due to interferences. The observation of the appropriate color change is a strong indication that no interferences have occurred. If it appears as if there is interference, the pH must be measured using the pH meter. [See SOP187 pH, Electrometric.]

3.3.2 The following guidelines must be followed to check pH preservation:

3.3.2.1 Water samples for Cyanide analyses must be preserved to a pH of >12.0 with NaOH upon collection. If the pH of these samples is <12.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to >12.0.

3.3.2.2 Water samples for Metals analyses must be preserved to a pH of <2.0 with HNO_3 upon collection. If the pH of these samples is >2.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to <2.0.

3.3.2.3 Samples requiring analyses which are preserved with H_2SO_4 (i.e., Nitrogen compounds, Total Phenolics, Total Phosphorus, etc.) should be preserved to have a pH of <2.0. If the pH of these samples is >2.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to <2.0.

3.3.2.4 Samples for sulfide analysis must have a pH >9.

3.3.2.5 If a sample is not properly preserved, the client must be contacted before preserving or adding additional preservative to the sample unless previously instructed. Log-in personnel must notify the Project Manager/Lab Director to contact the client and determine which of the following is appropriate:

3.3.2.5.1 If the client instructs us to add chemical preservatives to a sample, preservation information including reagent, lot# and amount used must also be included in the LIMS container comments.

3.3.2.5.2 All metals samples preserved upon receipt must be held 24 hours before proceeding with analysis - **record date, time and initials in test code comment section**. Preservation information including reagent, lot# and amount used must also be included in the LIMS container comments.

3.3.2.6 In some instances it may not be possible to adjust the sample to the proper pH due to matrix problems which cause excessive foaming or require an unusually large amount of acid. Do not continue to add acid if a few mL's of acid does not lower the pH. Notify the Project Manager, Metals Group Leader and/or analyst. They will make the decision if the sample will be diluted, not analyzed, etc. Make sure you note in the LIMS test code comments that the sample is not at the proper pH as well as any useful information (i.e., foaming, strong odor, etc.).

3.3.3 Check to make sure samples are in proper containers and that there is adequate volume for all the parameters requested and no leakage.

3.3.4 If VOA vials are present, each vial must be inverted and checked for head space. "Pea-sized" bubbles (i.e. bubbles not exceeding 1/4 inch or 6 mm in diameter) are acceptable and should be noted. Large bubbles or head space are not acceptable and this information must be documented in LIMS container comments. If this occurs, the client must be contacted. The samples can be analyzed with their approval, however the report will be qualified and the data may be questionable.

3.3.5 All Cyanide samples indicated as "Effluent" must be checked for residual (free) chlorine. AquaCheck Total Chlorine/Free Chlorine indicator paper (Hach Cat. 27450-50, or equivalent) will be used for detecting the presence of residual chlorine. DO NOT PUT THE TEST PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Use capillary tube to test and dispose of this volume after the sample is checked. If the test paper turns purple, the sample must be treated for residual chlorine. Add ascorbic acid approximately 0.6g at a time and recheck the sample until there is no residual chlorine present. If the sample required this treatment, information including reagent, lot# and amount used must also be included in the LIMS container comments. This must be done by log-in personnel before leaving the receiving area.

3.3.6 All 608 Pesticide/PCB samples must be checked for residual chlorine (following section 3.3.5 above) and pH (using wide range pH paper). If the sample contains residual chlorine, add sodium thiosulfate until the residual chlorine is removed. If the pH is less than 5, increase with the slow addition of NaOH. If the pH is greater than 9, reduce with the slow addition of HCl. These additions must be noted in the LIMS container comments including reagent, lot# and amount used.

3.3.7 All 625 Semi-volatile (BNA) samples must be checked for residual chlorine (following section 3.3.5 above). If the sample contains residual chlorine, add sodium thiosulfate until the residual chlorine is removed. Treatment information including reagent, lot# and amount used must be included in the LIMS container comments.

3.3.8 **Details (IDs of reagents used, volumes, etc.) of any sample adjustments MUST be noted within the LIMS.**

4.0 Sample Storage

4.1 After samples have been correctly logged in, they are then transferred to one of the following cold storage areas and arranged in numerical order by the assigned log in/LIMS sample number. ***Note that solid and aqueous VOC samples must be segregated from all other samples.***

4.1.1 VOC Water Refrigerator: All aqueous VOC's must be stored in this refrigerator. Storage blanks consisting of organic free water from the laboratory may be

required for specific projects. These will be analyzed for VOCs only. ***Storage blanks are required for all DOD projects.***

4.1.2 VOC Soil Freezer: All soil samples requiring VOC analysis with short hold prep times (Encores, Organic Free Water Terracores, etc.) must be stored in this freezer. ***Storage blanks are required for all DOD projects.***

4.1.3 VOC Soil Refrigerator: All bulk or chemically preserved soil samples requiring VOC analysis must be stored in this area. ***Storage blanks are required for all DOD projects.***

4.1.4 Metals Dry storage rack: All water HNO3 preserved samples for metals analysis are stored in the ICP metals area.

4.1.5 Walk in Refrigerator: All aqueous samples for all other analyses must be stored in this refrigerator.

4.1.6 Soil Walk-In Refrigerator: All quarantined and non-quarantined soil samples for all analyses must be stored in this refrigerator.

4.1.7 VOC Dry Storage Rack: All water VOCs that have exceeded double holding time can be stored on this rack. These samples are stored here segregated alone to ensure no cross contamination occurs between VOC samples and other non-VOC aqueous samples.

4.2 Quarantined soils are those quarantined by the US Department of Agriculture. A separate disposal log must be maintained for these soils including the location, date and quantity of the soil received and processed. Soil residues from quarantined samples must be treated according to regulations after testing. Quarantined soils are defined as:

4.2.1 Soil taken from much of the southeastern US and parts of New York and Maryland at a depth of three feet or less. *Soils from three feet or more are not regulated provided they are stored separately.* A map of the regulated areas in the United States entitled ***Soil Movement Regulations [Attachment IV]*** is posted in the log-in room.

4.2.2 All soils taken from foreign sources, US. Territories and Hawaii.

NOTE: All soils are treated as quarantined soils and are disposed of in accordance with USDA regulations. Above for information purposes only.

4.3 All samples must be stored in one of the four refrigerators detailed above with the following exceptions:

4.3.1 Matrices that may be adversely affected by the cold temperature. (E.g. surfactant samples, multi-phase samples).

4.3.2 Highly contaminated waste or product type samples that could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Project Manager for other options.

4.4 The temperature of each sample refrigerator is monitored and recorded each day. A Mercury thermometer or digital min/max thermometer with 1° increments must be used. Each thermometer must be calibrated against a NIST certified thermometer once a year **(digital thermometers quarterly)**. The thermometers must be tagged with a unique identification, the date calibrated and the correction factor.

4.5 The tolerance range for all refrigerators is 0°C to 6°C. If the temperature exceeds this range, corrective action measures must be implemented immediately. The Group Leaders, Data Quality Manager and Laboratory Director will be notified in order to assess the situation. It may be necessary to put a service call in to the refrigeration repair service.

4.6 All personnel removing samples from any refrigerator must sign them in and out. This is done by completing the ***Sample Custody Form [Attachment V]*** which is attached to the

door of each refrigerator. These completed forms are kept on file. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the samples are returned and signed back into the appropriate storage area. It must be noted if the entire sample volume was used and that the container was discarded.

4.7 The water and soil walk in refrigerators are the largest refrigerators and store a large majority of the samples. There are digital min/max thermometers, which monitors the temperature 7 days a week. There are also TempAlert temperature sensors connected to each of the refrigerators for round the clock monitoring. The TempAlert sensors are set up to notify management of any exceedence of the temperature requirements.

4.8 A temperature maintenance record book is kept for each refrigerator. Pay close attention to these readings and watch for signs of possible problems (change batteries at first sign of problems).

4.9 Samples must be held for a minimum of 45 days from receipt unless specified otherwise. See SOP QS14 entitled Analytical Laboratory Waste Disposal SOP for guidance on disposal of samples.

5.0 Laboratory Information Management System (LIMS)

5.1 Log the sample information into the LIMS for each sample. Every attempt should be made to get every sample logged into the LIMS by the end of the day. All information entered should be clearly stated and recorded on the COC provided. After opening the main menu of the LIMS, select the 'Work Orders' tab from the 'Sample Control' drop down menu. Now click on the 'Add' button to create a new Work Order. You will see the following:

5.1.1 Client: Select the client I.D. by clicking on the pull-down and choosing from the client list. This list is in alphabetical order. If the desired client is not on the list, a new client must be created by the Project Manager or DQM.

5.1.2 Project: Click on 'Projects' and choose the project I.D. **(Note – correct/accurate selection is absolutely <u>critical</u> to the successful analysis, reporting and invoicing of all samples.)** The projects will be client specific. After the project is chosen the "project information" areas should populate. The 'Project Name,' 'Project Number,' 'TAT,' 'Client Project Manager,' 'Lab Project Manager,' and 'Comments' information should also appear. If there are no applicable project choices, a project must be created by the Project Manager or I.T. director. There are two types of projects:

5.1.2.1 Internal – Empirical Laboratories projects;

5.1.2.2 External – direct laboratory clients.

5.1.3 Comments: This area is to be used to note any information from the Project Manager for all work orders of this project. It can also be used to list any work order specific notes; this includes but is not limited to information concerning rush turnaround, deliverables or other QC requirements, analyte concentrations, safety issues, quarantined soils, preservation or matrix problems, etc.

5.1.4 Received By: Enter the name of the person who received the samples.

5.1.5 Logged In By: Enter the name of the person who logged in the samples.

5.1.6 Received: Enter the date and time received separated by a space and using military time. Example: *08/02/2008 08:30*.

5.1.7 Project/Package Date Due: After the date and time received have been entered, the date due for both of these fields will be calculated. If this information is not

correct or needs to be amended later, check with the Project Manager before doing so.

5.1.8 Shipping Containers: Click on the 'Coolers' button and enter the temperature and condition upon receipt. If more than one cooler was received, each cooler must be assigned a different name. For example, if these came in by dedicated courier, enter the last four numbers of the Tracking Number as the name. After all of a cooler's information has been entered (received on ice, where custody seals present, preservation confirmed, COC/container labels agree, sample containers in-tact) click the 'Save' button. If more than one cooler was received, click the 'Add' button and repeat the process above, then click 'Done' after all the coolers' info has been saved.

5.1.9 COC Number: If an identifiable COC number is listed, record that ID here.

5.1.10 Shipped By: Enter the courier used to deliver the samples. If the samples were picked up by a lab employee or dropped off by the client/representative, enter 'Hand-Delivered.'

After these items have been completed, click 'Save,' then the 'Samples' button to continue. To begin entering information for a sample, click the 'Add' button on the bottom of the Samples screen.

5.1.11 Sample Name:

5.1.11.1 Only abbreviate if description is too long for the spaces allotted in the LIMS (must be discussed with Project Manager/Client). This information should come directly from the chain of custody. The sample ID entered into the LIMS will be the sample ID on the final report.

5.1.11.2 If no sample ID is provided, or is indistinguishable from other samples listed, contact the Project Manager to ascertain distinction in the samples. Include date as part of the description if this is the only way to differentiate the samples.

5.1.11.3 When logging in trip blanks that do not have an ID assigned by the client, list them as "Trip Blank # _____". This information should be on the containers. A log book must be kept in the sample kit room which lists all trip blanks and the date they were filled. This will ensure consistency with the descriptions for trip blanks.

5.1.12 Collection Date: Enter the date and time the sample was collected. You must use military time and separate by a space. Often the time collected is not given. Although this is a sampling requirement, this information may not be crucial unless a parameter with a short holding time or a data deliverables package is required. In the event that a sample collection time is not listed on the COC or the sample container, a default time of 00:01 can be used temporarily until client verification. Once verified, then the correct sample collection time must be input into LIMS. If the COC and sample containers do not list a collection date and time, this must be documented in the LIMS and the Project Manager must be notified. All attempts should be made to get all our clients to supply this information.

5.1.13 Lab/Report Matrix: Click on pull down and select matrix. Many times it is difficult to discern the matrix if it is not specified on the COC, and log-in personnel must use their best judgment with regard to analytes/methods requested. Keep in mind that the detection limits and units on the LIMS reports are linked to the matrix. In some cases it may be necessary to ask the Group Leader about the matrix selection. Log-in may do a dilution test to distinguish

water samples from oil samples if the COC does not clarify a sample matrix if need be.

5.1.14 Sample Type: This is used to differentiate between special types of samples (i.e. Field Duplicates, Equipment Blanks, Trip Blanks, etc.). If there is no definite way to determine that a sample should be classified as something else, then "SAMP-Client Sample" will be selected as the sample type. Do not list a sample as anything other than a Client Sample unless noted on the COC of are instructed by the client to do so.

5.1.15 Container: Click on the drop down list and select the appropriate bottle type. If multiple bottles are received for the same sample, move down to the next line and select all other containers as required. Repeat this process until all containers for the sample are listed. As each container is entered, an individual number is assigned to it by the LIMS system. This number is also listed on the container labels that are printed from the LIMS, and is placed on the corresponding bottle for container tracking purposes.

5.1.16 pH (Container Preservative): Use this to document the pH check information taken during sample unpacking. If no preservative was used, then nothing is required in this field.

5.1.17 Comments: Enter any information that is applicable at the sample level.

5.1.18 Field Analysis: Click on field analysis tab and enter field information when provided.

5.1.19 Work Analyses: Select all parameters requested for the sample from this list.

5.1.19.1 If the required test code is not listed, and the sample matrix is not a contributing factor, contact the Project Manager to correct the omission. **Do NOT log from "All analyses" as incorrect logins are not easily corrected.**

5.1.19.2 All preparation codes for analytes are entered and stored by the system independently of the test codes selected, except in the cases of Dry Weight analysis, and TCLP/SPLP preparation (tumbling). In the case of the TCLP/SPLP prep codes, these are entered alongside the other required analyses automatically by the LIMS when a TCLP/SPLP analyte is selected. As for Dry Weight, it is required for all solids testing except in the cases of TCLP/SPLP analysis, Explosives only analysis, and/or any pure product/non-soil based sample when specified by the client.

5.1.20 Analyses Comments: These comments should be used for any notes that only apply to that particular test code.

5.1.21 RTAT: If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.

5.1.22 Save: Once all applicable information is entered for a sample, click the save button. At this time the LIMS applies the Laboratory Sample ID to the sample. This is a five part ID code composed of the following:

5.1.22.1 A 2-digit numeral of the year. Example (**08**11248-06).

5.1.22.2 A 2-digit numeral of the month. Example (08**11**248-06).

5.1.22.3 A 3-digit numeral of the work order number. This number reset to 001 at the beginning of each month. Example (0811**248**-06).

5.1.22.4 A 2-digit numeral of the sample number separated by a dash. Example (0811248-**06**). This number is different for each sample in a work order.

5.1.22.5 A container ID is also associated to every container in the form of a letter starting with "A".

5.1.23 Add/Edit/Copy: Use these selections to add more samples to the work order, or to change existing information prior to label printing.

Once all the tests have been selected and all samples have been added in the work order, a cooler receipt form (example as Attachment II) and all container labels are printed. Labels are checked for accuracy against the containers while being labeled. At this point log-in of this group of samples is complete.

5.2 After log-in of a work order is complete and verified, the COC/CRF can then be scanned into the system and attached to the work order on the Work Order screen. Scanning the COC/CRF and saving the workorder for listing as "Received" is log-in's certification that all steps required per the SOP have been completed accurately.

5.3 The work order is then listed as Received and the PM is notified for review. The PM verifies accuracy of items listed in attachment III then updates to Available status so as to be seen by the analysts. **Updating to "Available" is PM certification that all checks have been completed.**

6.0 Daily Follow Up for Sample Receiving/Log In

6.1 Wipe out the inside of coolers and return all Empirical Laboratories coolers to the sample kit room.

6.2 At the end of the day organize all paperwork received and generated for the day. All the information from the day will be reviewed as soon as possible.

6.2.1 If any corrections or changes are required, all laboratory personnel will be notified by email distribution. E-mail notification by the Project Manager will also be sent out if a client adds or deletes any parameters, changes sample IDs, etc.

6.3 Sample Receiving will provide the original (white copy) chain of custody and cooler receipt forms to the Reporting Department.

7.0 Miscellaneous

7.1 All projects which require deliverables or other QC requirements should be listed in the notes section of the LIMS.

7.2 If samples are received from a new client or a new job number that is not in the LIMS, a new client code must be set up. This information should be on the chain of custody or it may be necessary to contact the customer if the information is incomplete.

7.3 All log books used in the Sample Receiving and Sample Storage Areas are numbered. The following log books are presently maintained. All log book pages must be closed out ("Z"ed out) before proceeding to the next page if unused lines remain. The Sample Receiving Group Leader will review the log books to check for completeness.

Log Book Description
Trip Blank Prep Log
Soil Treatment/Disposal Log
Acid Neutralization Log
Aqueous Disposal Log
Kit Room Preservation Preparation Log
Truck Maintenance Log

8.0 Sample Storage, Secure Areas and Sample Custody

8.1 Empirical Laboratories, LLC is located at 621 Mainstream Dr. suite 270 Nashville, TN 37228 on the first floor. This building is locked and monitored by an alarm system after

normal business hours. No unauthorized personnel are permitted within the facility without a proper escort and a visitor's badge. Outside business hours, all doors to the building are locked and secured by an alarm system. All front and back doors are locked and only Empirical Laboratories, LLC personnel have a key to access the building. Upon unlocking the door and entering into the laboratory, then the employee is to deactivate the alarm system using the assigned 4 digit alarm code assigned to them by Human Resources. Each employee is assigned their own designated alarm code, with no code being assigned twice. There is a buzzer at the door to Login to allow entry for sample and supply deliveries.

8.2 Sample extracts and digestates are stored in the following areas:

8.2.1 All metals digestates are stored in the metals instrument laboratory. The transfer from the digestion analysts to the ICAP analysts is documented in the metals digestion log book.

8.2.2 Non - ZHE TCLP extracts are returned to the refrigerator in which the original samples are stored. For ZHE samples, the extract is returned to the refrigerator in which the original VOC sample containers are stored.

8.2.3 Extracts from medium level VOC analyses are stored in the VOC Soil Segregated Area or VOC sample freezer in the VOC Lab, depending on the associated low-level vials.

8.2.4 All Organic extracts are stored in a Beverage Air side by side refrigerator in the organic extraction laboratory.

8.3 The generation of all sample extracts/digests and their movement through the laboratory will also be tracked on a laboratory custody sheet or in a log book. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the extracts are returned and signed back into the appropriate storage area. The metals digestates are not removed from the metals instrument laboratory.

8.4 After the analytical results have been reported, the original samples, sample extracts, and digestates will remain in secure storage until they are disposed of in accordance with the Waste Disposal Standard Operating Procedure (SOP QS14).

9.0 Sample Receiving Personnel Duties and Responsibilities

9.1 The sample receiving personnel are responsible for all duties associated to receiving, logging, subcontracting and disposing of samples. Although other laboratory personnel may assist with the duties, this is done under supervision and direction of sample receiving personnel. Sample receiving personnel are responsible for the following:

9.1.1 Receive all samples for the analytical laboratory and maintain chain of custody. This includes documenting the validated time/date of receipt.

9.1.2 Maintain the flow of samples through the log in process and make them available to the analysts on a timely manner. This includes prioritizing samples/projects based on turnaround requests, holding times, and second checking of primary logged sample.

9.1.3 Assign the correct laboratory ID sample numbers and validate that this information is properly labeled on the containers and entered into the Laboratory Information Management System (LIMS).

9.1.4 Validate that every sample proceed through all steps of the log in process. This includes checking the following to determine that the sample integrity has been upheld from the time the sample is collected until it is received in the laboratory: proper containers with ample sample volume, correct preservation, sample dates/times to ensure that holding times can be met, condition of the sample

containers, headspace of vials for VOC analysis, sample ID discrepancies and completeness of the chain of custody.

9.1.5 Communicate any information or specific requests by the client that are listed on the chain of custody, i.e., method information, detection limits, specific analytes, reporting information, turnaround information, potential hazards etc. They are also responsible for forwarding any additional information that may be received along with the samples, i.e. permit or regulatory information, letters, etc. to the laboratory managers.

9.1.6 The sample receiving personnel are personally responsible for continuing to uphold the sample integrity throughout the log in procedure and until the time when the samples are properly stored and disposed.

9.1.7 Ensure that samples are transferred into the proper storage area and that these secure areas are locked after hours.

9.1.8 Maintain all log books used in the section. These must be kept up to date, complete, neat and orderly.

9.1.9 Maintain the sample receiving and sample disposal areas in a clean, orderly and safe manner.

9.1.10 Follow good laboratory practices and safety procedures.

9.1.11 Communicate all problems, discrepancies, etc. to the Project Manager, Group Leader and Laboratory Director.

9.1.12 In situations where the client cannot be contacted, the sample receiving personnel along with the Group Leader must apply the best judgment on how to handle the samples or situation.

9.1.13 Complete all the necessary paperwork and section forms including Cooler Receipt Forms, LIMS daily print outs, Sample Receiving Custody and Disposal Form, etc. in a timely manner.

9.1.14 Dispose of all samples in a manner that is safe, cost efficient, timely, meets project requirements and is in accordance with hazardous waste regulations.

9.1.15 The sample receiving personnel are responsible for compliance of all procedures outlined in this SOP and the following SOPs.

9.1.15.1	SOP QS10	Laboratory Sample Receiving, Login and Storage
9.1.15.2	SOP QS11	Health and Safety Plan & Chemical Hygiene Plan
9.1.15.3	SOP QS13	Field Sampling & Bottle Kit Preparation
9.1.15.4	SOP QS14	Analytical Laboratory Waste Disposal

10.0 Waste Handling

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability. Please see Waste Disposal SOP QS14, for instruction of proper disposal of waste generated from this area.

11.0 Subcontracting Laboratory Samples

11.1 Sample receiving is responsible for handling all aspects of shipment of subcontracted samples. Once samples have been confirmed as sub-outs, login then notifies the Project Manager that subout samples are in house. The Project Manager then generates a purchase order number for the specific subout samples. Once the purchase order is generated by the Project Manager, then login prints out a subcontracted chain of custody from LIMS that will accompany the subout samples during transit and pulls the samples listed on the subout COC and gets verification of IDs/containers from another lab employee who initials/dates

the subout COC. Then login packs up the samples into a cooler, ices them down (if necessary) to keep the samples chilled during transit, and then the cooler is shipped to the subcontracted laboratory.

11.2 Chain of Custody/Shipping Requirements

11.2.1 When the samples are sent out, a completed sub chain of custody must be sent with the samples. Make sure to include the following information:

11.2.1.1 Be specific in your analyses request. List the method number if applicable and/or any specific analytes required. This should already have been discussed with the laboratory.

11.2.1.2 List the name of sub contract laboratory and the date shipped or delivered.

11.2.1.3 List the Empirical Laboratories; LLC LIMS log # as the sample description on the chain of custody. Do not list the actual client name or actual project information.

11.2.1.4 Record the date and time that the samples were sampled on the chain of custody.

11.2.1.5 Results and invoice should be sent to the Project Manager.

11.2.2 Two copies of the sub contract chain of custody should be retained. One copy should be stapled to the original chain of custody received from the client and the other should be stapled to the copy in log in.

11.2.3 Make sure samples are second checked against the COC and packed well so they will not break or spill in shipment. Ice must be packed in the cooler to keep the samples cold if chilling is required.

11.2.4 A P.O. must be completed and approved by the Project Manager prior to sample shipment. Sample receiving should then keep a copy of this P.O. for their records.

12.0 Attachments to QS10

I Chain of Custody Record
II Cooler Receipt Form (example)
III DATA ENTRY VERIFICATION LIST – PROJECT MANAGEMENT
IV A) Map of Quarantined Soil Areas in the U.S./ B) Map for Fire Ants/ C) Map for Nematodes
V Laboratory Sample Custody Form for Refrigerators

I Chain of Custody Record

EMPIRICAL LABORATORIES, LLC - CHAIN OF CUSTODY RECORD

SHIP TO: 621 Mainstream Drive, Suite 270 ♦ Nashville, TN 37228 ♦ 877-345-1113 ♦ (fax) 866-417-0548

Send Results to:	**Send Invoice to:**	**Analysis Requirements:**	**Lab Use Only:**
Name _____ Company _____ Address _____ City _____ State, Zip _____ Phone _____ Fax _____ E-mail _____	Name _____ Company _____ Address _____ City _____ State, Zip _____ Phone _____ Fax _____ E-mail _____		VOA Headspace Y N NA Field Filtered Y N NA Correct Containers Y N NA Discrepancies Y N NA Cust. Seals Intact Y N NA Containers Intact Y N NA Airbill #: _____ CAR #: _____
Project No./Name:	**Sampler's (Signature):**		

Lab Use Only Lab #	Date/Time Sampled	Sample Description	Sample Matrix	Comments	No. of Bottles	*Lab Use Only* Containers/Pres.

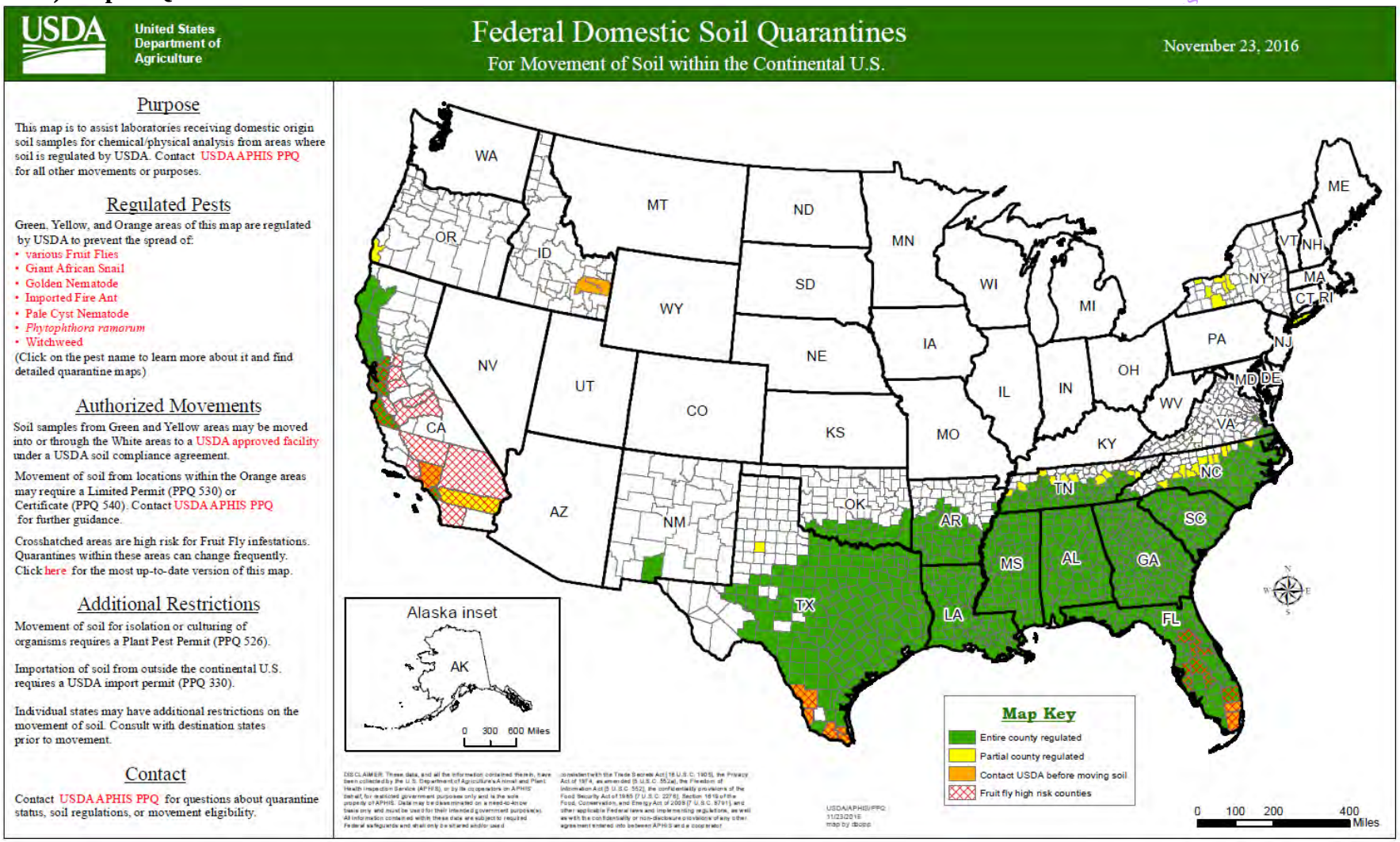
Sample Kit Prep'd by: (Signature)	Date/Time	Received By: (Signature)	REMARKS:	Details:
Relinquished by: (Signature)	Date/Time	Received By: (Signature)		Page _____ of _____
Relinquished by: (Signature)	Date/Time	Received By: (Signature)		Cooler No. ____ of _____
Received for Laboratory by: (Signature)	Date/Time	*Temperature*		Date Shipped _____ Shipped By _____ Turnaround _____

Distribution: Original and yellow copies accompany sample shipment to laboratory; Pink retained by samplers.

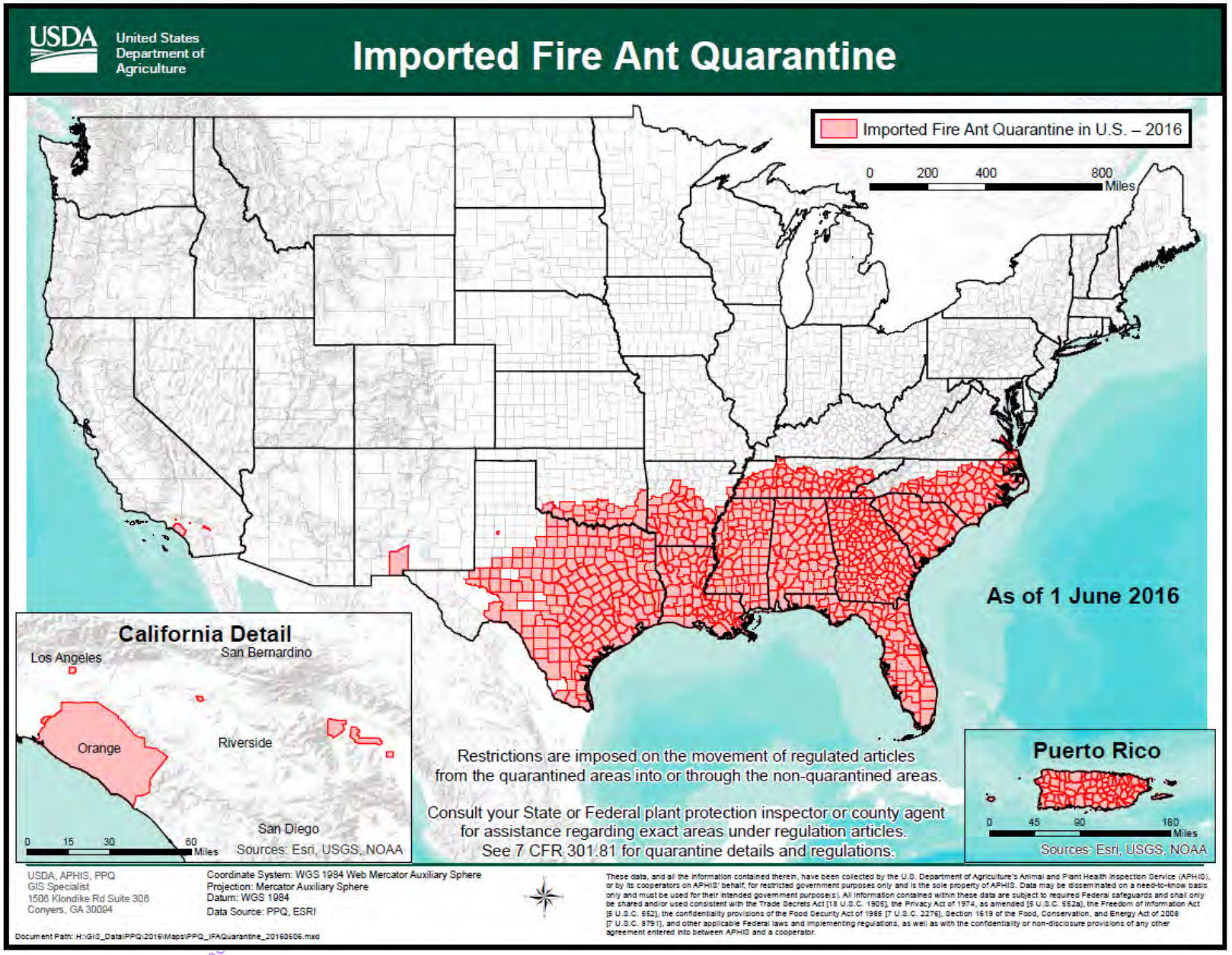
III. Project Management Veri	**Verification Item**

fication List	
1.	**Cooler Receipt Form Issues reviewed and communicated to client**
2.	**Element/ Project Screen/items verified to match the COC/CRF:**
a.	**Client/Project**
b.	Comments requiring laboratory reminder?
b.	Client and/or Project Memo requiring laboratory reminder?
3.	**Receipt Screen items verified to match the COC/CRF:**
a.	Received Date/Received By
b.	Workorder Due Date
c.	Package Due Date
d.	TAT
e.	SDG Identifier Populated
4.	**Sample Information verified against COC for each sample:**
b.	Name
c.	QC Source
d.	Matrix
e.	Sample Type
f.	Sampled Date/Time (Correct Time Zone)
h.	Work Analyses/Versions
a.	Sample Issues included in comments
i.	Unpreserved VOA holding time set to 7 days
5.	**Containers consistent with tests requested**
6.	**Field data entered and matching COC, if applicable**

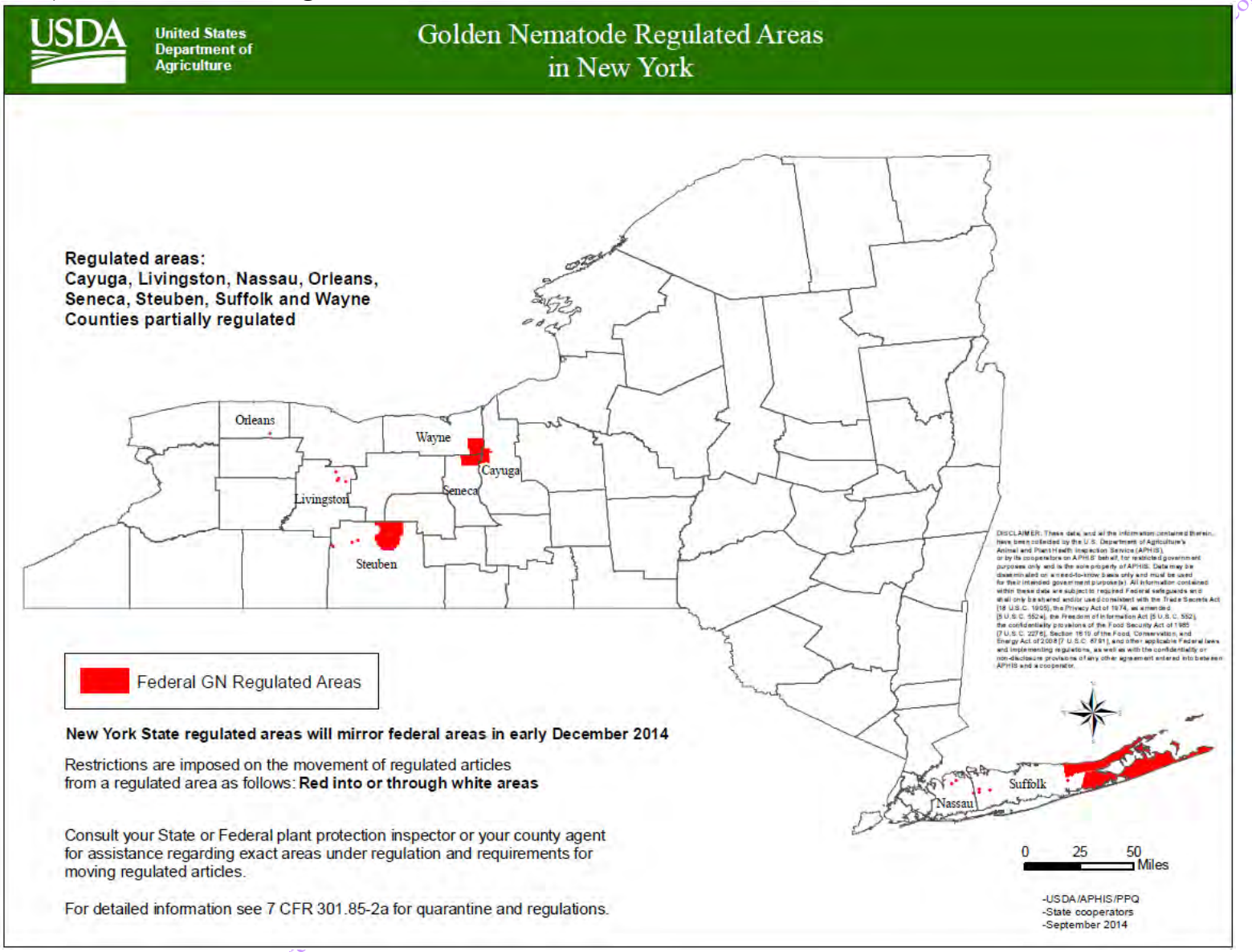
IV.A) Map of Quarantined Soil Areas in the U.S.



IV.B) Imported Fire Ant Quarantine Map



IV.C) Golden Nematode Regulated Areas in New York



V. Custody Log Example

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

QUALITY SYSTEMS: QS14 REVISION #: 11 EFFECTIVE DATE: 20160223

ANALYTICAL LABORATORY WASTE DISPOSAL

APPROVALS:

Lab Director: _____ Date:**20160223**

Data Quality Manager: _____ Date: **20160223**

Sample Receiving Group Leader: _____ Date: **20160223**
Tiana Hutchings

QS14_R11_20160223_WSTE

- Updates based on USDA Soil Permit – Sections B and C with previous section C-E advanced to D-F – review thoroughly.

QS14_R10_20150923_WSTE

- Updates based on TDEC evaluation – review thoroughly.
- Attachment C, compliance declaration added.
- All references to supervisor updated to reflect group leader.
- Section IV.B. updated to indicate primary and backup procedures.

QS14_R09_20140729_WSTE

- Watermark update to include proprietary reference.
- Section IV updated – complete review required.

Changes to this Revision - R08 20130123

- Additional information added to section IV.B. concerning handling of soil samples for disposal.
- Labeled laboratory floorplan added to the end of the SOP as Attachment A and referenced in section IV.B.

Changes to this Revision – R07 20120430

- Revision to QS14 R06 dated 08/31/2010.
- Updates have been made to all sections of this SOP. Training covering all aspects is required.

Changes to this Revision – R06 08/31/2010

- Revision to SOP405 R05 dated 6/23/2009.
- Changed the document control and named this as QS14 R06.
- Minor cosmetic/grammatical changes made.

Analytical Laboratory Waste Disposal
Standard Operating Procedure

I. SCOPE AND APPLICATION:

Laboratory waste includes excess client sample waste and waste that is generated while performing an array of analytical services, some of which are hazardous. These wastes must be disposed of in a manner that is safe, cost efficient and in accordance with hazardous waste regulations.

A. Wastes can be broken down into the following categories:

1. Unused portions of actual samples received from outside clients
   a. Unused aliquots of completed water samples
   b. Unused aliquots of completed non-aqueous samples
2. Soils from quarantined areas
3. All other soils, sediments, building debris, wipes, oils, etc.
4. Hazardous waste generated within the laboratory as part of numerous analytical procedures.

II. SUMMARY OF PROCEDURES:

A. There are four options for disposing of unused sample portions:

1. Return completed samples and any generated waste from these samples to the client
2. Disposal of samples after confirming that they are non-hazardous.
3. Disposal through a waste vendor in either a sealed drum or lab pack.
4. Treat the sample to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

B. There are two options for disposing of laboratory generated waste:

1. Disposal through a waste vendor in either a sealed drum or lab pack. The waste must be stored properly until transported off site.
2. Treat the waste to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

III. EQUIPMENT/APPARATUS:

A. Proper safety equipment should be in good working condition. This includes gloves, lab coats, safety glasses/goggles, and face shields.
B. USDOT approved drums for storing and shipping hazardous waste.
C. Fume hoods.

D. 5 gallon waste containers

IV. PROCEDURE

Waste disposal is done under the management and coordination of the Sample Receiving Manager, Group Leader, Safety Coordinator and Lab Director. Also, please see Attachment C. This is a declaration to Metro Nashville Industrial Wastewater Department that we are in compliance with their regulations.

A. Disposal of completed aqueous samples:

Completed samples are kept in cold storage for a minimum of 45 days from receipt and sample extracts are held for 90 days minimum from receipt. Engineering support projects involving CLP work, litigation cases, etc. may be saved for longer than 45 days at the request of the project manager.

1. The majority of the water samples (ground, surface and drinking) are non-hazardous and are disposed of by pouring them down the sink in the hooded area in sample receiving as follows:
   a. Make sure that the sash is closed far enough to produce sufficient ventilation.
   b. Turn on the tap water to supply copious rinse for sample disposal.
   c. Wear proper safety equipment including safety glasses (face shield, if necessary), lab coat, and gloves.
   d. Be alert to potential problems: for example, separate cyanide waste from acid waste. Neutralize acid waste that will be poured down the drain and don't mix waste/samples. Also, look for things such as phase separation, odd color, odor, etc. Check with the Group Leader or Safety Coordinator before disposing of any questionable samples.
   e. Leave tap water running when samples are poured out and for approximately 10 minutes following disposal in order for sufficient flushing and dilution to take place.
   e. Rinse all containers and discard into the trash.
   f. Document disposal in the bound aqueous sample disposal logbook with date, initials, workorder and any appropriate comments.

2. If water samples are hazardous, one of the following steps must be taken:
   a. Samples may be returned to the client. If you plan to ship the unused portion back to the client, check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT

regulations. If the samples are not returned to the client they must be stored properly until picked up by a waste vendor.

b. Treat the sample to make it non-hazardous. One example of this is if the sample is highly corrosive, the pH may be adjusted.
c. Store the sample properly until either a sealed drum or lab pack is sent out.
d. Document disposal in the bound aqueous sample disposal logbook with date, initials, workorder and any appropriate comments concerning treatment, lab-pack, etc.

B. Disposal of completed non-aqueous samples:

Note – for purposes of the USDA permit, see attachment A for a floor plan of the laboratory located at 621 Mainstream Drive, Suite 270, Nashville, TN, 37228 (GPS coordinates N36° 11.9094' and W086° 48.9253'). Emergency contacts are located on the laboratory phone list which is to be posted on the outside of the hood for the oven used to treat soils.

The majority of non-aqueous samples are soils and sediments. Although there may also be building debris, wipes, oils, and occasionally product type samples.

1. Soil samples taken at a depth of three feet or less from areas which have been quarantined by the US Department of Agriculture (USDA) and all imported foreign soil must first be treated at the laboratory to prevent the spread of any plant pests. For simplicity, all soils are treated as quarantined soils. The USDA has detailed treatment options from which we selected the following:
    a. For simplicity, Empirical Laboratories, LLC is disposing of all non-hazardous soils in a USDA recognized quarantine area. The drums are picked up by a certified waste vendor and carried for incineration then disposal to a landfill in South Carolina which is located in a USDA recognized quarantined area.
    b. An alternative to certified vendor incineration/disposal in a USDA recognized quarantine area, the samples may be heated to 180°C (356°F) in a vented oven for a minimum of two hours. USDA requirement is 121°C-154°C (250°F-309°F) for 2 hours or 154.4°C-192.6°C (310°F-379°F) for 30 minutes. We choose higher temperature with longer bake times for added certainty.
    c. All samples disposed of in this manner must be documented in the bound soil treatment/disposal logbook with date, initials, workorder(s), oven ID, temperature, quantity (oz) and any appropriate comments.
2. If non-aqueous samples are hazardous, one of the following steps must be taken.

a. Samples should be returned to the client. If you plan to ship the unused portion back to the client, check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations.
b. If the samples are not returned to the client they must be stored and labeled properly until picked up by a waste vendor.

C. **Handling and Records associated to Foreign Soils**

While requirements for disposal of foreign soils are similar to requirements for USDA quarantined soils, handling within the laboratory is more specific. Following are the records requirements, procedure for handling spills, procedure for handling water produced during handling of the soils, area cleanup requirements and training requirements.

1. Records Requirements:
    a. Date of arrival, origin of shipment and amount of soil received.
    b. Date and amount of soil used for testing.
    c. Date and amount of disposed soil and amount, if any, remaining.
    d. Date and amount of soil (domestic) that is transferred and copies of permit and compliance agreement of the receiving party.
    e. Date and amount of foreign soil that is transferred with copies of permit and compliance of the receiving party.
2. Spillage of soil:
    a. All surfaces must be cleaned with 10% household bleach solution or 70% ethanol solution.
    b. All excess soil must be disposed of in accordance with this SOP.
3. **Water Residues and Equipment/Supplies from Foreign Soil**s
    a. Water residues produced or obtained from processing of foreign soil must be boiled dry. The vessel used to boil dry must then be treated by placing it in the furnace at 250°C for 15 minutes before allowing to cool.
    b. Equipment and supplies used to conduct operation or that have contacted the soil must be decontaminated using one of the following procedures:
        i. Soaked in 10% solution of household bleach for a minimum of 30 minutes.
        ii. Soaked in a 70% solution of ethanol for a minimum of 30 minutes.
4. A Training Program is required for all lab employees handling foreign soils and will be conducted annually or at the start of any project handling foreign soils.

D. Disposal of laboratory generated waste:

Generated waste is stored until a waste pick up occurs. These areas must be maintained properly by using satellite stations within several laboratory areas. These stations must be labeled appropriately and properly maintained.
**Note: Laboratory generated solvent waste is transferred to the appropriate Satellite Solvent Waste Drum as deemed necessary. Disposal of solvent waste is done under the direction of the Laboratory Director or Safety Coordinator. Solvent drums are filled up a day or two before the pick up day. This procedure has been put into place to assure any issue with a leaking drum is taken out of the realm of possibility.

1. Waste handling and disposal within each laboratory section:
   NOTE: Each laboratory analyst and group leader is responsible in assuring that handling operations (within their area) are being followed according to the laboratory requirements.

   a. General Chemistry/Inorganic

   Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If any questions remain unanswered regarding waste disposal within your specific area contact the Group Leader or the Safety Coordinator.

   - Acid waste is neutralized by using sodium hydroxide. Once the pH of the acid waste has been neutralized (pH 5-9), the acid waste is then poured down a sink within hooded ventilation followed by 10 minutes of flushing with tap water. The amount of acid waste treated, the amount of sodium hydroxide used to neutralize the acid waste, final pH, date performed and the date disposed of is then recorded in an acid waste neutralization log book along with the initials of the person performing the neutralization/disposal.
   - Hach Ferrous Iron vials are placed into the acid waste area for disposal. The contents of these vials are not hazardous but they are in sealed glass vials, so being very careful with respect to cuts is extremely important and the Safety Coordinator or his designee will handle this task.
   - Chemical Oxygen Demand (COD) vials have to be dealt with as a special waste due to its contents. This waste contains traced levels of mercury – 200ppb. Since we use so very few of these, we will store all of these until we have adequate volume to be handled by a designated hazardous waste vendor. The container must be clearly identified with

appropriate labels, with a description of what it is. It will take an estimated 3-4 years to complete this exercise.

- Lachat Waste Instrument: This instrument produces two separate cyanide bearing wastes. The first is a clean liquid with <12 pH, it's cyanide concentration is below 2ppm which means that it can be neutralized and discharged to the POTW. The second waste is dark green with a pH <12 and has a cyanide concentration of 50-100 ppm. It also contains "phenol" at ~ 5000 ppm. This waste is designated as hazardous and is accounted for and disposed of as such.
- TOC (water) Instrument: This instrument produces a corrosive liquid effluent. This is to be handled as a corrosive waste. Once the gallon jug is full it must be placed on the shelving for disposal and the Lab Director/ Sample Receiving must be notified via e-mail immediately.

b. Metals

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If any questions remain unanswered regarding waste disposal within your specific area contact the Group Leader or the Safety Coordinator.

- **Concentrated acid waste, aqueous sample waste digestates, and old unused calibration standards (>2% by volume)** are taken to the Acid Satellite Station located on the shelving to the right of the back Wet Chem door. The shelving has signs to identify what waste goes in each area.
- **Non-aqueous sample digestate wastes** are decanted off the soil/solid samples into the Acid Satellite Station located on the shelving to the right of the back Wet Chem door. The shelving has signs to identify what waste goes in each area.
- **Cr6 digestates as with all concentrated metal/acid waste are also placed in the Acid Satellite Waste** Station located on the shelving to the right of the back Wet Chem door. The shelving has signs to identify what waste goes in each area.
- **ICP Effluent:** This liquid must be handled with the same procedures as the TOC instrument effluent. Please see section (a).

- **Perkin Elmer Effluent Waste:** This liquid must be handled with the same procedures as the ICP effluent above.

c. Organic Extraction Laboratory Area

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If any questions remain unanswered regarding waste disposal within your specific area contact the Group Leader or the Safety Coordinator.

- **Concentrated acid waste** is taken to the Acid Satellite Waste Station located on the shelving to the right of the back Wet Chem door. The shelving is labeled to show what waste is stored in each area.
- **Non-chlorinated solvent waste** (Acetone, Ether, Hexane, and Methanol ….etc…) is poured into the Non-Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory and logged in the hazardous waste logbook.
- **Chlorinated solvent waste** (Methylene Chloride, Chloroform, chlorinated standard and spike waste) is poured into the Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory and logged in the hazardous waste logbook.
- **Aqueous sample waste** from extracted samples (after the extraction solvent has been removed) is poured down the drain and flushed with copious amount of tap water.
- **Sodium sulfate** waste is dumped into a waste container under an extraction laboratory hood and left overnight or until evaporated. The waste must be 100 percent dry. Then the waste is discarded into the trash.
- **Unused stock and working standards** are emptied into the chlorinated or non-chlorinated solvent waste bottle. The empty vials are discarded in the glassware waste container.

d. Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Laboratory:

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If any questions

remain unanswered regarding waste disposal within your specific area contact the Group Leader or the Safety Coordinator.

- GRO instrument waste effluent: This waste must be handled with the same procedures as the TOC instrument. Please see section IV-C.
- HPLC effluents: Bottles must be kept with cover on them. Once bottles are full, place label on bottle identifying its contents. Email Lab Director and Sample Receiving to advise that the solvent is ready for disposal.
- LC/MS waste products: The waste is a mixture of water, acetonitrile. It will take months, if not longer, to obtain a full gallon of this waste. At that time, it should be treated as a flammable solvent and disposed of by contracted vendor. An email should be sent to the Lab Director and Sample Receiving when the container is ready for pick-up.
- Unused stock standard, unused working standards, sample extract autosampler vials and sample extract vials are separated by the solvent and discarded into the designated 5 gallon waste container located in the GC/HPLC Laboratory.
- Acid cleaned extracts are separated and provided to the sample receiving manager. They are later combined into a separatory funnel and the acid layer separated from the solvent. The acid portion is placed in the Acid Satellite Waste area located between Wet Chem and Extractions while the solvent waste is discarded into the appropriate solvent waste bottle (chlorinated/non-chlorinated waste) located in the hood in the organic extraction laboratory.

e. Gas Chromatography/Mass Spectrometry

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If any questions remain unanswered regarding waste disposal within your specific area contact the Group Leader or the Safety Coordinator.

- **Volatile sample, standard, and reagent waste:**
  Instrument Waste - Aqueous sample waste is collected in waste bottles via waste lines from each instrument. Each bottle is considered corrosive by Metro Discharge criteria. Call Sample Receiving when the containers are ready for pick-up. Send an email to the Lab Director and Sample

Receiving concerning the need to have these picked up as soon as possible. A small amount of methanol used to clean glassware is also dumped into the waste container and poured down the drain.

Unused stock and working standards are separated by solvent and discarded appropriate designated 5 gallon waste container located in the GC/MS or GC/HPLC Laboratory for later consolidation into lab packs.

- **Semi-volatile sample and standard waste disposal:** Unused stock standard, unused working standards, autosampler vials and extraction vials are discarded into the appropriate designated 5 gallon waste container located in the GC/MS Laboratory for later consolidation into lab packs.

E. Consolidation of satellite waste for contractor disposal:

In conjunction with the Safety Coordinator, the Sample Receiving Manager and the Lab Director are responsible to coordinate waste disposal operations with outside waste disposal contractors.

1. Solvent waste from the areas discussed above is periodically consolidated into a drum. We do not transfer to 55 gallon drum till a day or two before our hazardous waste contractor is scheduled to pick up the waste. This is done to prevent any issues with the drum developing a leak while waiting for pick up. .
2. The Acid Waste drum or 5 gallon waste container is also disposed through the authorized hazardous waste contractor once the drum or 5 gallon waste container is full.
3. Consolidated autosampler and standard vials are periodically Lab-Packed in drums and disposed of through an authorized hazardous waste contractor.
4. The Safety Coordinator and the Sample Receiving Manager, in conjunction with the Lab Director, will administer the Waste Disposal Program and maintain current information to track quantities of waste generated and stored on-site.
5. It is the continuous objective of our laboratory to find ways to decrease the amount of waste generated.

F. Corrosive liquids:

These liquids must be handled to comply with city and state regulations.

1. State regulations (corrosive if pH is ≤ 2). Once samples and digestates hit their disposal date, this must be managed within 24 hours. This means they must be poured up into the neutralization units. Within 1 – 1.5 weeks, the

neutralization procedure will take place by adding increments of NaOH till a pH between 5-9 is reached. At that point, the waste can be discharged to the POTW.

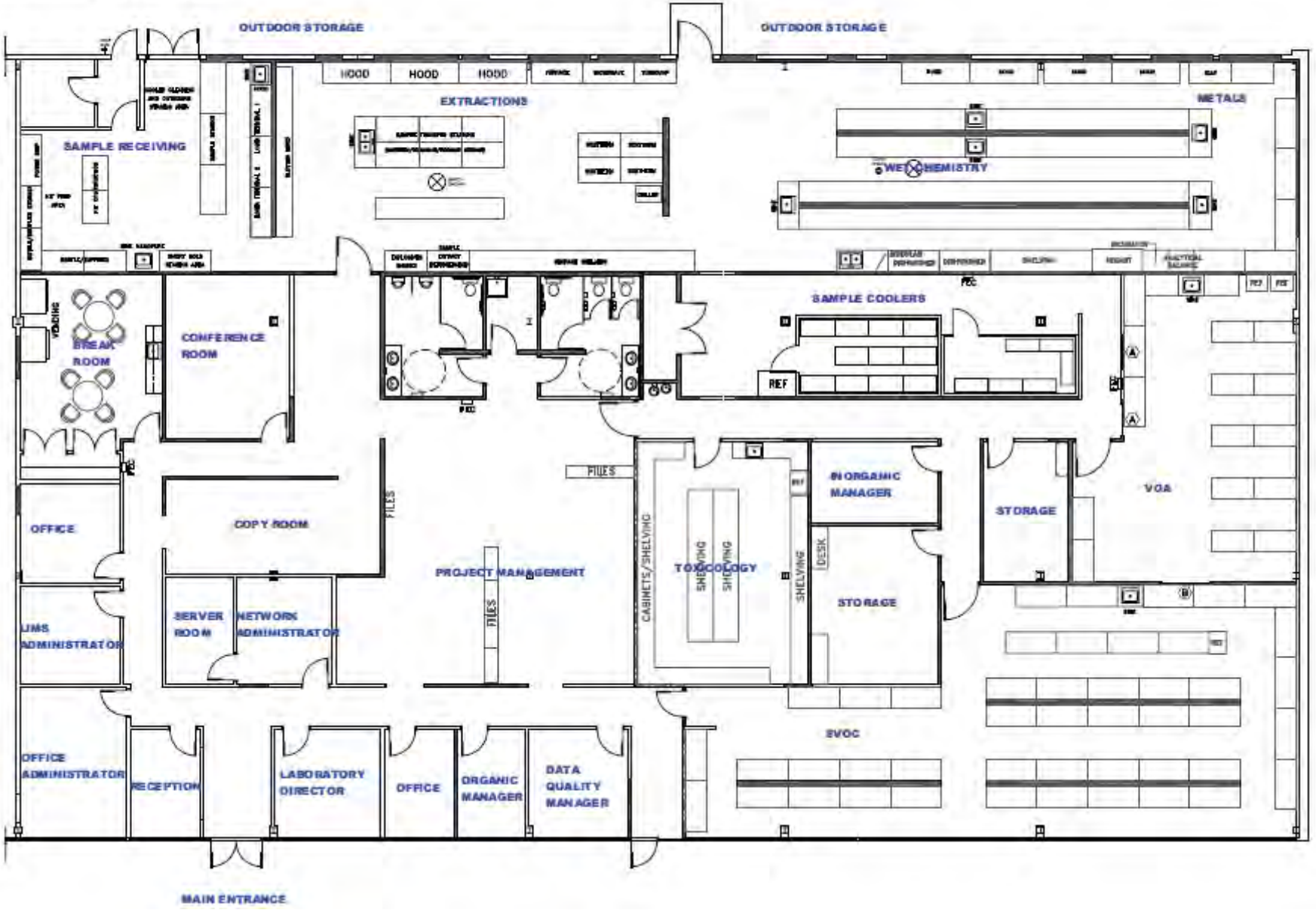
2. City regulations (pH > 2 but ≤ 9). Liquid samples and wastes must meet the above criteria before discharging to the POTW. We have analyzed these samples and we are able to meet any other discharge criteria that exists in the city discharge regulations. The liquids will be placed in 5 gallon neutralization units and NaOH will be added till acceptable pH criteria is obtained.

Attachment A: Laboratory floor plan.

Attachment B: Processing, Storage, and Treatment of Corrosive Samples

Attachment C: Total Toxic Organics (T.T.O) Declarative Statement

EMPIRICAL LABORATORIES, LLC
621 MAINSTREAM DRIVE, SUITE 270
NASHVILLE, TN 37228



ATTACHMENT B

Processing, Storage, & Treatment of Corrosive Samples

A Summary of the Five Stages

Stage	Area	Action	Notes
1	Analytical Testing Area	Sample Processing Area/Department	Sample digestates remain in the area where they are analyzed. The samples remain here for several weeks to a month or two.
2	Corrosive Sample Storage	After samples are processed and moved from (1) above	Samples are moved from stage 1 to this stage. Samples remain approximately 90-120 days, until the client required storage time has expired or client approval is received and it is acceptable to dispose of them.
3	Corrosive Waste Management Area	Samples placed into acid neutralization containers (24 Hour Management Required)	When samples are moved into this stage, they must be managed (poured into the neutralization carboys) within 24-hours. If they cannot be, the volume must be accounted for in the monthly logbook for hazardous waste generated. This stage is also where process corrosive liquids enter the system and must also be managed within 24-hours.
4	Corrosive Waste Neutralization Area	Samples neutralized with NaOH to pH 5.1-10.0	The samples that were managed in stage 3 are actually neutralized in this stage using Sodium Hydroxide. The pH must be between 5.1 to 10.0 prior to disposal into the POTW.
5	Discharge to POTW	Neutralized samples drained into sink	The neutralized samples from Stage 4 are discharged from the carboy into the lab sink.

ATTACHMENT C

TOTAL TOXIC ORGANICS (T.T.O)
DECLARATIVE STATEMENT

Based on my inquiry of the person or persons directly responsible for managing compliance with the permit limitation (or pretreatment standard) for Total Toxic Organics (T.T.O), I certify that, to the best of my knowledge and belief, no dumping of concentrated toxic organics into the wastewater system has occurred.

_____
Company Official

D. Rick Davis, Lab Director
Company Official (printed name & title)

_____
Date of Submittal

Not Applicable
Permit Number

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP100 REVISION #: 32 EFFECTIVE DATE: 20160907

METALS DIGESTION/PREPARATION

References:
Methods 3005A, 3010A, 3050B and EPA 200.7 REV. 4.4

APPROVALS:

Lab Director: _____ Date: **20160907**

Data Quality Manager: _____ Date: **20160907**

Group Leader: _____ Date: **20160907**
Betty Quillen

SOP100_R32_20160907_MDIG

- Updated section 10.2.2.1.2 and circle sheet to reflect 5mL spike for low-level waters.

SOP100_R31_20160831_MDIG

- Added Whatman#40 filter to section 9.
- Alternate digestion procedure added as 14.4.2 for possible improvement of antimony, barium, lead and silver.

SOP100_R30_20151204_MDIG

- Sections 10.1 and 12.1 updated to reference use of pre-cleaned Plastic Bead Solid Matrix for soil blanks and blank spikes.

SOP100_R29_20151111_MDIG

- EPA method updated to more accurately reflect EPA 200.7 Rev. 4.4.

SOP100_R28_20151006_MDIG

- Handling for silver greater than 100ug/L in EPA200.7 inserted as section 14.3.2.8.
- Table 26.1 updated for 1mL pipettors on TCLP and 100mL waters.

SOP100_R27_20150921

- All references to Section Supervisor updated to reflect Group Leader
- Section 10 updated to reference QS04.
- Section 12.1.2.1 clarified.
- Section 14.3.1 updated for filtration of dissolved metals.
- Section 22.3 updated for notification of sample receiving.
- 26.1 Circle Sheets updated and added reminder to verify mechanical pipettes daily at volume of use for QSM5.

SOP100_R26_20140718

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark update to include proprietary reference.
- Section 4.3.2: Requirement to filter the blank, BS, MS/MSD and DUP added.
- Section 9.17: Filter apparatus added.
- Section 10.1.1: Process for testing acids described.
- Section 10.2.2.1.1.2.1: Instructions for the BS with Sr,B,Li,Mo, Sn and Ti added.
- Section 10.2.2.1.2: The use of glass beads for BS added and teflon chips removed.
- Section 12.1.2.2: The use of glass beads for the Blank added and teflon chips removed.
- Section 16.6: Demonstration of Capability instructions added.
- Section 19: Instructions on handling difficult matrices added.
- Section 20: Instructions on actions to take when there is a QC failure added.
- Section 21: Instructions for Waste Management were updated.
- Section 22: Equipment maintenance was added.
- Section 24: Troubleshooting was added for QC problems and difficult maintenance.
- Section 25: References were updated.

Revision 25, 12/19/2013

- Section 10.2.2.1.1.1: Removed residual reference to CLP preparation.
- Section 10.2.2.1.1.2: Updated spike solution preparation information.
- Section 14: Updated information on digestion vessels and filter apparatus.
- Section 14.3.1: References to 200.7 removed.
- Section 14.3.2: Added 200.7 digestion process.

- Section 14.3.3: Previously 14.3.2 for Method 3010A.
- Attachment: Circle Sheet updated for spike SM-391-001 and separation of 3005A/200.7.

Revision 24, 09/03/2013
- Removed any references CLP and Standard Methods.
- Combined Method 3005A and EPA 200.7 digestion instructions.
- Attached Eppendorf/Solution Circle Sheets used for daily digestions.

Revision 23, 03/05/12
- The SOP is an update from Revision 22 11/17/10
- Removed any references to using watch glasses
- Added a note that the method does not require digestion for dissolved metals. Analysts digest dissolved samples only when selenium is requested.
- Changed references to Hot plate, Thermolyne Eimarec-3 to Hot block, Environmental Express
- Removed Reagents used for oil method that is no longer performed.
- Removed statement that analyst must initial and date each entry made in LIMS.
- Changed LCS to Blank Spike

Revision 22, 11/17/10
- The SOP is an update from Revision 21 dated 9/1/10
- Revised to add the need for matrix spike duplicates to be digested and analyzed for TCLP extracts.
- Requirement to hold samples 24 hours after in-house preservation was added to section 3.

Revision 21, 9/1/10
- The SOP is an update from Revision 20 dated 04/27/10
- The SOP has been found to be up-to-date with Standard Methods 21st edition.
- Reference to adjusting filtrate volume for method 3030C has been removed.
- References to bound logbooks have been replaced with LIMS references.

Revision 20, 4/27/10
- The SOP is an update from Revision 19 dated 04/20/09.
- References to oil sample preparation have been removed.
- Extraction volumes for TCLP have been updated.

Table of Contents

METALS DIGESTION/PREPARATION
References: Methods 3005A, 3010A, 3050B and EPA 200.7 REV. 4.4

1. Identification of the method

Methods 3005A, 3010A, 3050B and EPA 200.7 REV. 4.4

2. Applicable matrix and matrices

2.1. Method 3005A is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
2.2. Method 200.7 is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
2.3. Method 3010A is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.
2.4. Method 3050B is used to prepare sediments, sludges, and soil samples for analysis by ICP.
2.5. Samples for dissolved analysis may be prepared without digestion if Selenium is not required.

3. Limits of detection and quantitation

Refer to Table one in SOP 105.

4. Scope and application, including parameters to be analyzed:

4.1 AQUEOUS
4.1.1 Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
4.1.2 Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry" Revision 4.4
4.1.3 Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
4.2 SOLIDS
4.2.1 Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".
4.2.1.1 It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.
4.2.1.2 Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed and required by the client.
4.3 NOTES
4.3.1 "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.
4.3.2 "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion, when selenium is needed because sulfides in the sample are a positive interference for selenium. The method does not require digestion for dissolved metals as long as the method blank, blank spike, matrix spike and matrix spike duplicate and/or duplicate are filtered as well.

5. Summary of the method

A representative sample of water or soil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower than is customary, a four times concentration or alternate soil digestion ratio must be used in order to reach that lower level. Care must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike (not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

6. Definitions

Refer to SOP-QS08 for common environmental laboratory definitions.

7. Interferences

7.1. AQUEOUS

7.1.1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

7.2. SOLIDS

7.2.1. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

8. Safety

8.1. Normal accepted laboratory safety practices should be followed while performing this analysis.
8.2. Be certain the exhaust hood is functioning before you begin the digestion procedure.
8.3. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.
8.4. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab wide.
8.5. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of nitrile gloves and lab coats is highly recommended.
8.6. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
8.7. MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your Group Leader, lab director or data quality manager if there are any difficulties in accessing these records.

9. Equipment and supplies

9.1. Fume hood, Labconco or equivalent.
9.2. Hot block, Environmental Express or equivalent source for use at 95°C. The temperature of the hot block must be monitored via the use of a temperature blank.
9.3. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.
9.4. Vacuum pump for filtering dissolved metals- Gast or equivalent.
9.5. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.
9.6. Beckman CS-6R centrifuge.
9.7. Various class A volumetric glassware, Pyrex or equivalent.
9.8. Whatman No. 40 filter paper or equivalent.
9.9. Whatman No. 41 filter paper or equivalent.
9.10. Whatman No. 42 filter paper or equivalent.
9.11. Whatman 0.45 micron filter paper or equivalent.
9.12. Polypropylene block digester tubes and caps.
9.13. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.
9.14. Mortar & pestle thoroughly cleaned with HNO3, rinsed with deionized water and dried before use.
9.15. Clippers for cutting vegetation.
9.16. Wiley Sample Mill for grinding vegetation.
9.17. 10 mm sieve (used when specifically requested by client).
9.18. For dissolved samples filtered in house and for filtering QC associated with non-digested dissolved samples use Nalgene Rapid-Flow Filters from Thermo Scientific or Fisher, 150 mL capacity with 0.45micron filter. Part # 165-0045 or equivalent.

NOTE: All glassware should be acid washed.

10. Reagents and standard

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

10.1. REAGENTS

10.1.1. ACS grade Nitric acid (HNO_3). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used. The process to be used here is to take the same volume of acid used for digestions and add it to 100 mLs of deionized water in a clean "for metals use bottle" and submit it to receiving labeled with the Lot # and the Element # assigned to the case of acid when received. It will be given a workorder number. Then it is analyzed without digestion and the workorder is reported. The acid may not be used until this process has been followed and it is ascertained that the acid is < DL for all metals of interest.

10.1.2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <DL, then the reagent can be used. See the process above.

10.1.3. 30% Hydrogen Peroxide Reagent, ACS Grade reagent should be analyzed to determine level of impurities. If method blank is <DL, then the reagent can be used.

10.1.4. Reagent water (Deionized water).

10.1.5. Plastic Bead Solid Matrix (Environmental Express item#SC400, or equivalent) – certified by Environmental Express, pre-cleaned with nitric acid rinse then rinsed with DI water and stored in an HDPE container.

10.2. STANDARDS

10.2.1. Traceability

10.2.1.1. A LIMS record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label.

10.2.1.2. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the information is recorded in LIMS. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in LIMS. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

10.2.2. Preparation

10.2.2.1. Blank Spike

10.2.2.1.1. Aqueous

10.2.2.1.1.1. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, 0.25 mL of CLP-CAL-3 and 1 mL 1000 mg/L strontium, boron, lithium, molybdenum, tin and titanium diluted to 1 L in a volumetric flask. Use 50 mL for digestion. This solution is given a unique identifier and recorded in LIMS.

10.2.2.1.1.2. For four times concentrated samples: 5 mLs of the above solution under 10.2.2.1.1.1 is used plus 0.50 mLs of salt spike. The salt spike is made as follows: 1mL of 10000 mg/L calcium, magnesium, sodium and potassium diluted to 100 mLs with de-ionized water that has 2 mLs of HNO3 and 5 mLs of HCl. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO_3 and 5% HCl. Use 0. 5 mLs HNO_3 and 1.25 mLs HCl to each 100 mL vessel.

10.2.2.1.2. Solids

10.2.2.1.2.1. 1.0±0.02 (or 2.0 ±0.02) gram aliquot of glass beads is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier and when digested is given the descriptor. i.e. BS1 and then BS2 etc. Alternately a solid matrix standard reference material is obtained from the manufacturer.. This sample

is given a unique identifier and the weight is recorded in a bound logbook and transferred to LIMS.

10.2.2.2. Spiking Solution

10.2.2.2.1. Sample is spiked using 0.050 mL (50 uL) of CLP-CAL-1, Solution A and 0.050 mL (50 uL) of SM-391-001 for a final volume of 50 mL. These solutions are given unique identifiers. Use the circle sheet to indicate the amount spiked. Amount of spike used and the unique identifier of the standards are indicated in the bench sheets in Element as well.

10.2.2.2.2. For samples that require four times concentration, the sample is spiked using 0.025 mL of CLP-CAL-1, Solution A, 0.025 mL of CLP-CAL-1 Solution B, 0.0125 mL of CLP-CAL-2 and 0.0125 mL of CLP-CAL-3 to a 100 mL volume of sample. When required, the sample is also spiked using 0.025 mLs of B (1000 mg/L), 0.025 mL of Sn (1000 mg/L), 0.025 mLs of Mo (1000 mg/L), 0.025 mL of Titanium (1000 mg/L), 0.025 mL of Sr (1000 mg/L) and 0.025 mL of Li to a 100 mL vessel with 100 mLs of sample. The volume is lowered to less than 25 mLs and then final volume of this concentrated sample is brought back to 25mLs.

10.2.2.2.3. **For solid samples 1g to 200 mLs, the sample is spiked using 0.20 mL of CLP-CAL-1, Solution A and 0.20 mL of SM-391-001 for a final volume of 200 mL. These solutions are given unique identifiers. Use the circle sheet to indicate the amount spiked. Amount of spike used and the unique identifier of the standards are indicated in the bench sheets in Element as well.**

10.2.2.2.4. **For solid samples 2g to 100 mLs, the sample is spiked using 0.10 mL of CLP-CAL-1, Solution A and 0.10 mL of SM-391-001 for a final volume of 100 mL. These solutions are given unique identifiers. Use the circle sheet to indicate the amount spiked. Amount of spike used and the unique identifier of the standards are indicated in the bench sheets in Element as well.**

11. Sample collection, preservation, shipment and storage

11.1. AQUEOUS

11.1.1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated HNO_3 to a pH <2 immediately upon sampling. If dissolved metals are to be analyzed the sample should be filtered before the HNO_3 is added and within 72 hours of sampling unless otherwise designated in the QAPP. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

Note – samples received unpreserved and preserved in-house must be held 24 hours prior to preparation.

11.2. SOLIDS

11.2.1. Samples are taken in high density polyethylene or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

12. Quality control

12.1. Digestion

12.1.1. Temperature blank

12.1.1.1. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.

12.1.1.2. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.

12.1.2. Blanks

12.1.2.1. Prepare a blank with every batch of samples filtered and/or digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry digestion vessel and taking it through the same process as the samples.

12.1.2.2. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples. 1.0±0.02 (or 2.0 ±0.02) gram aliquot of pre-cleaned Plastic Bead Solid Matrix are used for non-aqueous blanks.

12.1.2.3. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
12.1.2.4. Sample is given a unique identifier in the digester log.
12.1.2.5. The method does not require digestion for dissolved metals as long as the method blank, blank spike, matrix spike and matrix spike duplicate and/or duplicate are filtered as well.

12.1.3. Blank Spike

12.1.3.1. For water samples, one BS is digested with every batch of samples digested (20 samples maximum.)
12.1.3.2. For water samples, a BS is digested every day for each type of digestion, every 20 samples.
12.1.3.3. For soil/sediment samples, a spiked solid matrix material must be digested per batch (20 samples maximum). 1.0±0.02 (or 2.0 ±0.02) gram aliquot of pre-cleaned Plastic Bead Solid Matrix are used for non-aqueous blank spikes.
12.1.3.4. Sample is given a unique identifier in the digestion log.
12.1.3.5. See 12.1.2.5 for information on how to handle dissolved metals.

12.1.4. Duplicates

12.1.4.1. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.
12.1.4.2. See 12.1.2.5 for information on how to handle dissolved metals.

NOTE: Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.

12.2. Sample Matrix

NOTE: Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

12.2.1. Matrix Spike

12.2.1.1. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.
12.2.1.2. See 12.1.2.5 for information on how to handle dissolved metals.

NOTE: For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Project comments or your group leader should make you aware of these projects.

12.2.1.3. For TCLP samples, a spike **and a spike duplicate** must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquots must be added to the extracts after filtration but before preservation.)
12.2.1.4. **Certain projects require that a high and a low spike be prepared and analyzed. Check project specifications. Spikes should be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.**

13. Calibration and standardization

The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. Record in temperature logbook for later transfer into LIMS.

14. Procedure

Make sure bench is clean with new bench sheet prior to starting. Also, circle sheets must be available to document All procedures below require the completion of a circle sheet indicating what procedure was used and recording the batch#. The circle sheet and bench sheet are placed with the prepared batch and the LIMS status is set to "prepared" upon completion. Make sure all dates and analyst information are correctly populated for samples and QC.

14.1. Calibrated digestion vessels are received from vendor with certificates indicating ASTM volume line calibration and lot #. Certificates are filed for future reference.
14.2. Aqueous Sample Filtration (for dissolved metals):

14.2.1. Filter apparatus are purchased from a vendor; alternatively, thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO_3 followed by a thorough D.I. water rinsing.

14.2.2. Rinse a 0.45 micron filter with 1:5 HNO_3 thoroughly, followed by D.I. water. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.

14.2.3. Filter at least 100 mLs of each sample. If the sample requires a matrix spike (MS) and Matrix spike duplicate (MSD), a minimum of 240 mLs must be filtered which will require two filter apparatus in order to get enough volume for both mercury (90 mLs) and ICAP metals (150 mLs). More will be required if concentration is needed.

14.2.4. Discard the first 50 to 100 mL.

14.2.5. A preparation blank, blank spike, matrix spike and matrix spike duplicate or duplicate must be taken through the filtration step and analyzed with the samples.

14.2.6. Preserve the sample with HNO_3 to pH<2.

14.2.7. Soluble samples that are clean and clear may not have to be digested unless the client is requesting selenium, since sulfides, which are normally found in ground waters, are a positive bias for selenium. For samples not requiring digestion, filter 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO_3 to preserve and matrix match.

14.3. Aqueous Sample Preparation:

14.3.1. **Method 3005A "Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP ", "Acid digestion procedure for total recoverable metals"**.

14.3.1.1. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration, pour 100 mLs of the well-mixed sample into the large (130 mL) digestion vessels.

14.3.1.2. Add 1.0 mL concentrated HNO_3 to the sample. For samples which require concentration, add 0.50 mL concentrated HNO_3 to the sample.

14.3.1.3. Add 2.5 mL concentrated HCl to the sample. For samples which require concentration, add 1.25 mL concentrated HCl to the sample.

14.3.1.4. Transfer the digestion vessel to a pre-heated hot plate at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the temperature logbook. Take the volume down to between 5 to 10 mL, **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot block and cool.

14.3.1.5. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

NOTE: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

14.3.1.6. Bring sample to its pre-digestion volume (or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the LIMS.

14.3.1.7. The sample is now ready for analysis.

14.3.1.8. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.

14.3.2. **Method 200.7**, **"Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP ", "Acid digestion procedure for total recoverable metals"**. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.

14.3.2.1. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel.

14.3.2.2. Add 0.50 mL concentrated HNO_3 to the sample.

14.3.2.3. Add 0.25 mL concentrated HCl to the sample.

14.3.2.4. Transfer the digestion vessel to a pre-heated hot block at 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the temperature logbook. Take the volume down to between 5 to 10 mL, **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot block and cool.

14.3.2.5. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

NOTE: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

14.3.2.6. Bring sample to its pre-digestion volume with DI water in the digestion vessel. The final volume must be recorded in the LIMS.

14.3.2.7. The sample is now ready for analysis.

14.3.2.8. **If concentration for silver is found to exceed 100ug/L, it is necessary to digest/analyze progressively smaller, well-mixed aliquots until the analysis solution contains <100ug/L silver.** This is due to the fact that silver is only slightly soluble in the presence of chloride unless there is sufficient chloride concentration to form the soluble chloride complex.

14.3.2.9. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.

14.3.3. **<u>Method 3010A</u>, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy"**.

14.3.3.1. Shake sample thoroughly and pour 50 mL (5ml diluted to 50mL for TCLP, full 50ml volume for SPLP) of the well-mixed sample into the digestion vessel.

14.3.3.2. Add 1.5mL concentrated HNO_3 to the sample.

14.3.3.3. Transfer the digestion vessel to a pre-heated hot block at 90 to 95°C. A temperature blank must be used, with the temperature being recorded in the temperature logbook. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries.** Remove the sample from the hot block and cool.

14.3.3.4. Add another 1.5 mL portion of concentrated HNO_3 to the sample.

14.3.3.5. Transfer the vessel to the hotblock. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

14.3.3.6. Evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.

14.3.3.7. Add 2.5 mL of concentrated HCl.

14.3.3.8. Reflux for an additional 15 minutes.

14.3.3.9. Bring sample to its predigestion volume in digestion vessel.

14.3.3.10. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

NOTE: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

14.3.3.11. The sample is now ready for analysis.

14.3.3.12. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

14.4. Solid Sample Preparation

It is extremely important that waste (when appropriate, soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE 1: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

NOTE 2: Vegetation samples must be ground using a Wiley Sample Mill before digestion by the 3050B method.

14.4.1. **<u>Method 3050B</u>, "Acid digestion of Sediments, Sludges and Soils"**

14.4.1.1.1. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the LIMS.

<u>NOTE:</u> To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.

14.4.1.1.2. Add 5 mL D.I. water and 5 mL concentrated HNO_3 (1:1), mix the slurry. Place the samples in a preheated hot block and reflux at 95°C for 10 to 15 minutes being certain that the sample does not boil. Record temperature in temperature logbook.

14.4.1.1.3. Allow sample to cool. Add 5 mL concentrated HNO_3, and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) over and over until <u>no</u> brown fumes are given off by the sample indicating the complete reaction with HNO_3. Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at 95°C $\pm$ 5°C for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the vessel. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.

14.4.1.1.4. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of H_2O_2 if it is still warm.) Return the sample to the hot block and heat until the bubbling subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides; then cool the digestion vessel. Add two more 3 mL portions of H_2O_2 to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2.)

14.4.1.1.5. Continue heating the acid-peroxide digestate at 95°C $\pm$ 5°C without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.

14.4.1.1.6. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling.

14.4.1.1.7. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

14.4.1.1.8. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle using a class A graduated cylinder. Invert the 50 ml sample digestion vessel several times to mix the sample and mix 50mL sample volume with the 150 mL in the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.

NOTE1: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

NOTE2: To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.

14.4.1.1.9. The sample is now ready for analysis.

14.4.1.1.10. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

14.4.2. **Method 3050B Alt, "Alternate digestion to improve solubilities and recoveries of antimony, barium, lead and silver – when necessary and client/management has approved additional cost."**

14.4.2.1.1. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the LIMS.

NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.

14.4.2.1.2. Add.2.5 mL concentrated HNO_3 and 10mL of concentrated HCl. Place the samples in a preheated hot block and reflux at 95°C+/-5°C for 15 minutes - being certain that the sample does not boil. Record temperature in temperature logbook.

14.4.2.1.3. Allow sample to cool. Pre-rinse a Whatman #40 or #41 filter with 1:6 HNO3 then filter the digestate and collect filtrate in a 100mL digestion vessel.

14.4.2.1.4. Wash the filter paper while still in the funnel with 5mL of HCl then 20mL 95°C reagent water. Collect the filtrate in the 100mL digestion vessel from above.

14.4.2.1.5. Place filter and residue in original digestion vessel and add 5 mL of concentrated HCl.

14.4.2.1.6. Heat at 95°+/-5°C until filter paper dissolves. Record temperature in temperature logbook.

14.4.2.1.7. Pre-rinse a second Whatman #40 or #41 filter with 1:6 HNO3 then filter the digestate and collect in 100mL digestion vessel from above.

14.4.2.1.8. Allow to cool. If precipitate forms, add up to 10mL concentration HCl to dissolve then bring to 100mL final volume with DI water.

14.4.2.1.9. The sample is now ready for analysis.

14.4.2.1.10. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

15. Data analysis and calculations

The analyst must record both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the bench sheet.

16. Method performance

16.1. Never take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your Group Leader or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.

16.2. Antimony (Sb) soils should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it the next day or the same day that it is digested.

16.3. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, the date digested, and the volume or mass digested.

16.4. There are several precautions that must be taken to minimize the possibility of contamination:
16.4.1. All metals glassware must be kept separate from all other laboratory glassware.
16.4.2. Metals glassware must be washed as soon as possible after it is used.
16.4.3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.

16.5. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your Group Leader must be consulted if this schedule cannot be met at a particular time.

16.6. Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples. The data is calculated for accuracy and precision requirements (see Group Leader for the form to be used). The DOC form, as listed in the Quality Control SOP QS03 under "links to forms" is completed by each analyst and then provided to the Group Leader for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM4.2 or QSM5.0, associated QSM limits will be used.**

17. Pollution prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability. Please see Waste Disposal SOP QS14, for instruction of proper disposal of waste generated from this area.

18. Data assessment and acceptance criteria for QC measures

18.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **#12 above** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Corrective actions and out-of-control data

19.1. If a sample boils during digestion, redigest another sample aliquot.

19.2. If a sample goes dry or a portion of the beaker bottom is exposed due to excess evaporation during digestion, redigest another sample aliquot, and if the glass beaker was dry for an extended period of time, discard the beaker.

20. Contingencies for handling out-of-control or unacceptable data

20.1. If a method blank is contaminated and the sample concentrations are not greater than 10 x the concentration of the method blank all samples impacted must be re-digested and reanalyzed. Try to find the reason for the contamination before starting the process over.

20.2. When matrix spike and matrix spike duplicate RPDs are not less than 20% the sample spiked and the MS/MSD should be re-digested and reanalyzed.

20.3. Samples that come in over a quarter of the way full of dirt and already preserved especially those logged for the low level test codes should be discussed with the manager before digesting.

20.4. When samples will not digest due to excessive effervescing during the hydrogen peroxide phase or even during the HNO3 phase, contact your manager and make sure that the PM knows the problem. Normally we will go back and digest with a smaller volume of sample.

20.5. Organic samples or those samples with an organic phase should not be digested on the hot block. These samples should be discussed with the manager and most likely will be separated into two samples – organic and inorganic phases. The organic phase would need to be subbed out for microwave digestion.

21. Waste Management

21.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.2 Waste acids are labeled properly as to what they are and are then placed on the acid shelf with secondary containment for proper disposal by receiving personnel.

22.3 Digestates that are more than 3 months old are labeled properly and are also placed on the acid shelf for proper disposal by receiving personnel. The sample receiving group leader is notified by e-mail when digestates are placed on the acid shelf and the amount of waste being placed there.

22. Equipment/instrument maintenance

If Hot Block or Hood is not working try turning them off and back on and if that does not reset them, inform your manager and/or send an email to maintenance@empirlabs.com.

23. Computer hardware and software

Not applicable

24. Troubleshooting.

24.1. When blanks come back as contaminated for a metal then the whole hood should be cleaned and wiped down, before re-digesting.

24.2. When duplicate spikes do not agree, unless it can be ascertained that this is due to matrix (non-aqueous samples), the pipette used to add the spike amounts should be re-calibrated before re-digesting.

24.3. If samples spill over the sides while adding H2O2 the digestion must be started again. Wait until the sample is completely cooled before adding H2O2. If the sample continues to spill over even when the sample is completely cool before adding H2O2, then a smaller sample size must be used, be sure to inform your Group Leader so that the PM can be informed and the client will be warned that they will have a higher detection limit for those samples.

25. References

25.1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 3005A, 3010A and Method 3050B.

25.2. USEPA *Code of Federal Regulations, 40, CH 1,PT 136;* Method 200.7; APX-B.

25.3. DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003. 10/25/2010

25.4. DOD and DOE consolidated Quality Systems manual for Environmental Laboratories Version 5.0. [(Based on ISO/IEC 17025:2005(E) and the NELAC Institute (TNI) Standards, Volume 1, (September 2009)].

25.5. Standard Methods – 22nd Edition.

26. Tables, diagrams, flowcharts and validation data

26.1. Circle Sheet for Eppendorfs and Solutions

26.1 Circle Sheet for Eppendorfs and Solutions

ICP Metals: Circle Applicable Digestion/Spike and pipette option then staple to bench sheet

Reminder: Make sure pipettes are verified daily at volume of use for QSM5 batches!

Soil 2g-100mL (3050B)

Amount	BS/MS/MSD Standards 6010 Soil
0.10mL	#01 CLP-CAL-1 (50-33000ug/mL)
0.10mL	SM-391-001 (125->1000ug/mL)
Circle	0.1ml Eppendorf 148897Z or 250847
	1mL FinnPipette FJ61963
Amount	**Reagents for Digestion 6010 Soil**
5mL	HNO3
5mL	HNO3
2mL	DI H2O
3mL	H2O2
3mL	H2O2
3mL	H2O2
2.5mL	DI H2O
2.5mL	Conc HCl
10mL	DI H2O

Soil 1g-200mL (3050B)

Amount	BS/MS/MSD Standards 6010 Soil
0.20mL	#01 CLP-CAL-1 (50-33000ug/mL)
0.20mL	SM-391-001 (125->1000ug/mL)
Circle	0.1ml Eppendorf 148897Z or 250847
	1mL FinnPipette FJ61963
5mL	HNO3
5mL	HNO3
2mL	DI H2O
3mL	H2O2
3mL	H2O2
3mL	H2O2
2.5mL	DI H2O
2.5mL	Conc HCl
10mL	DI H2O

TCLP 5mL-50mL (3010A)

Amount	MS/MSD Standards 6010 TCLP
5.0mL	Silver TCLP Spike
0.50mL	Main TCLP Spike
Circle	1mL Transfer 06M15889 or FinnPipette FJ61963
Circle	5mL FinnPipette KU04206 or Transfer 04M88005
Amount	**BS Standards TCLP 6010**
50mL	Water LCS (0.050-5.0ug/mL)
Amount	**Reagents for Digestion TCLP**
1.5mL	HNO3
1.5mL	HNO3
2.5mL	HCl

Water 100mL-25mL (3005A)

Amount	MS/MSD Standards 6010 Water
0.025mL	#01 CLP-CAL-1 (50-33000ug/mL)
0.025mL	#02 CAL-CAL-1 Solution B (250ug/mL)
0.0125mL	#03 CLP-CAL-2 (1000ug/mL)
0.0125mL	#04 CLP-CAL-3 (500ug/mL-1000ug/mL)
0.025mL	#05 Boron (1000ug/mL)
0.025mL	#06 Tin (1000ug/mL)
0.025mL	#07 Molybdenum (1000ug/mL)
0.025mL	#08 Titanium (1000ug/mL)
0.025mL	#09 Strontium (1000ug/mL)
0.025mL	#10 Lithium (1000ug/mL)
Circle	0.1ml Eppendorf 148897Z or 250847
Amount	**BS Standards 6010 Water**
5.0mL	Water LCS (0.050-5.0ug/mL)
0.5mL	Salt Spike (25ug/mL)
Circle	1mL Transfer 06M15889 or FinnPipette FJ61963
Circle	5mL FinnPipette KU04206 or Transfer 04M88005
Amount	**Reagents for Digestion Low Water**
0.5mL	HNO3
1.25mL	HCl

Water 50mL-50mL (3005A)

Amount	MS/MSD Standards 6010 Water
0.050mL	#01 CLP-CAL-1 (50-33000ug/mL)
0.050mL	SM-391-001 (125->1000ug/mL)
Circle	0.1ml Eppendorf 148897Z or 250847
Amount	**BS Standards 6010 Water**
50mL	Water LCS (0.050-5.0ug/mL)
Amount	**Reagents for Digestion 6010 Water**
1.0mL	HNO3
2.5mL	HCl

Water 50mL-50mL (200.7)

Amount	MS/MSD Standards 6010 Water
0.050mL	#01 CLP-CAL-1 (50-33000ug/mL)
0.050mL	SM-391-001 (125->1000ug/mL)
Circle	0.1ml Eppendorf 148897Z or 250847
Amount	**BS Standards 6010 Water**
50mL	Water LCS (0.050-5.0ug/mL)
Amount	**Reagents for Digestion 6010 Water**
0.50mL	HNO3
0.25mL	HCl

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 103 REVISION #: 27 EFFECTIVE DATE: 20161121

MERCURY ANALYSIS IN WATER
BY MANUAL COLD VAPOR TECHNIQUE
METHODS USEPA SW846 7470A and 245.1 Rev. 3.0

APPROVALS:

Lab Director: _____ Date: **20161121**

Data Quality Manager: _____ Date: **20161121**

Group Leader: _____ interim Date: **20161121**

Changes Summary

SOP103_R27_20161121_HGDIGW
- Referenced to Element updated to LIMS throughout document.
- Section 9.14 updated to include scan of blank and verification documentation to LIMS by LIMS ID.
- Section 12.3 inserted for initial linear dynamic range determination per EPA 245.1.

SOP103_R26_20151221_HGDIGW
- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.

SOP103_R25_20151111_HGDIGW
- Initial EPA method reference updated to include revision number.

SOP103_R24_20150921_HGDIGW
- All references to supervisor updated to reflect group leader.
- Section 9 updated for equipment.
- Section 10 updated to reference QS04
- Section 14.3 updated to include KI check
- Section 15.2 updated for worksheet location
- Section 22 updated with troubleshooting tips.
- Circle sheets updated.
- Added immediate analysis of 2 passing CCVs as option in table 2.
- Table 3 removed and Data Reviewer Checklist updated to reflect Table 3.

Revision 23, 20140721
- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark update to include proprietary reference.
- 9.0 Equipment and supplies updated.
- 10.2 Preparation of calibration standards specified
- 13.0 Calibration and Standardization further specified.
- Limits references updated to direct analyst to table 2 throughout the document.
- 15.0 Data Analysis and Calculations further specified.
- 22.0, 23.0 and 24.0 added/populated.
- 25.0 updated
- Table 2 updated to include QSM5.0 criteria

Revision 22, 09/03/2013
- All references to CLP have been removed.
- Detection limit table removed as Table 1 but maintained in the body of the SOP and Digestion Reference Sheets added as Table 1 and referenced in digestion procedure.
- Requirement to report time on and time off for digestion added to procedure and checklist.
- Removed reference to linear correlation coefficient requirement of 0.998.

Revision 21, 20120813
- Added 2 samples between CCV/CCB for example sequence
- Updated analyst review checklist
- Added instructions for starting/running instrument
- Added requirement for certificate of analysis on purchased standards.

Revision 20, 20110516

- Software instructions have been updated in section 13 to reflect new software.
- References to old 400 level SOPs have been removed.

Revision 19, 11/17/10

- Revised to require a matrix spike duplicate for TCLP extracts.
- Requirement to hold samples 24 hours after in-house preservation was added to section 11.1.
- Quality systems SOP references updated.

Revision 18, 04/11/10

- The SOP is an update from Revision 17 dated 03/25/10
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.2, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.
- Tables have been updated to reflect the current limits/processes.

Table of Contents

MERCURY ANALYSIS IN WATER
BY MANUAL COLD VAPOR TECHNIQUE
METHODS USEPA SW846 7470A and 245.1 Rev. 3.0

1.0 Identification of the Test Method

This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury, and is compliant with SW846 Method 7470A and USEPA Method 245.1 Revision 3.0.

2.0 Applicable Matrix or Matrices

This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

3.0 Limits of Detection and Quantitation

Method Detection Limit (MDL)/Detection Limit (DL), Limit of Detection (LOD) and Empirical Laboratories' Reporting Limit (ERL)/Limit of Quantitation (LOQ) and Analyte Wavelength:

Limits Table

Aqueous Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ)			
Mercury by EPA 245.1, 7470A	**AQUEOUS MDL/DL (ug/L)**	**AQUEOUS LOD (ug/L)**	**AQUEOUS ERL/LOQ (ug/L)**
Mercury (Wavelength 253.7)	0.080	0.16	0.20

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in the table above.

4.2 This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

4.3 In addition to inorganic forms of mercury, organic materials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenol mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system. For distilled water the heat step is not necessary.

4.4 The range of the method may be varied through instrument and/or recorder expansion. Using a 30 mL sample, a detection limit of 0.2 µg Hg/L can be achieved.

4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized and the 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 a flow injection Mercury system. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

7.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

7.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

7.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 6.25 mL in 30 mL of sample). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation at 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by using an excess of hydroxylamine sulfate reagent (6.25 mL to 30 mL of sample).

7.4 Samples containing high concentrations of oxidizable organic materials, as evidenced by high chemical oxygen demand values, may not be completely oxidized of organic mercury will be low. The problem can be eliminated by reducing the sample volume or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

8.0 Safety

8.1. Normal accepted laboratory practices should be followed while performing this procedure.

8.2. The toxicity and carcinogenicity of each reagent in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

8.3 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

8.4 The analyst should make sure that the system is vented to charcoal filter. Otherwise Hg vapors could be vented to the room.

8.5 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab wide.

8.6 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of nitrile gloves and lab coats is highly recommended.

8.7 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.8 MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9.0 Equipment & Supplies

9.1 Perkin Elmer Flow injection Mercury system. (FIMS 100)

9.2 Perkin Elmer S10 autosampler

9.3 Mercury lamp-FIMS (PE) B0510487

9.4 Yellow/blue pump tubing pk/12 Perkin Elmer (PE) B0193161

9.5 Tubing per pump 3.18mm bl/wh 40 cm pk/12 (PE) B0508310

9.6 Red/red pump tubing pk/12 (PE) B0193160
9.7 Mixing blocks (PE) B0507962
9.8 Quartz Window (PE) B0066549
9.9 PTFE tubing 1000 mm (PE) B0191060 (sample line from autosampler to intake valve)
9.10 PTFE tubing 700 mm (PE) B0191059 (for HCl and SnCl)
9.11 PTFE tubing 300 mm (PE) B0198097 (line from manifold to light tube)
9.12 500 uL sample loop (PE) B0194049
9.13 Environmental Express Mod-Block digestion block capable of holding 95+2°C for 2 hours.
9.14 Snap cap digestion polypropylene vessels for use with the mod block digester. Before use, each lot must be assigned LIMS ID and five vessels of each lot number must have blanks run in them to check for mercury with documentation scanned to LIMS ID. Unless Class A certified with documentation scanned as COA to LIMS ID, ten vessels of each lot number must be taken through volume verification as per SOP QS08 and recorded in the non-volumetric labware calibration logbook with verification scanned as COA to LIMS ID.
9.15 Polypropylene watch glasses suitable for use with the above vessels.
9.16 Millipore –filters- Fluoropore PTFE 1.0 um wh pl 25 mm 100/pk

10.0 Reagents and Standards

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST - RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards. Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Spex, Ultra Scientific and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at room temperature.

10.1 REAGENTS

10.1.1 Concentrated sulfuric acid suitable for Hg determination (ACS grade).
10.1.2 Concentrated nitric acid suitable for Hg determination.
10.1.3 Stannous chloride: In a 1000 mL volumetric flask add approximately 500 mLs D.I. water, 30 mLs concentrated HCl, add 11 grams stannous chloride crystals swirl to mix and dilute to 1000 mLs. Prepare fresh daily.
10.1.4 3% HCl Carrier Solution: Dilute 30 mLs of concentrated metals grade HCl to one liter. Prepare fresh daily.
10.1.5 Sodium chloride-hydroxylamine chloride solution: Dissolve 120 grams of sodium chloride and 120 grams of hydroxylamine hydrochloride (very high grade --Do not get from Tennessee Reagents) in D.I. water and dilute to 1 liter.
10.1.6 Potassium permanganate: 5% solution, w/v: dissolve 200 grams of potassium permanganate in 4000 mLs of D.I. water. Should have "suitable for mercury determination" written on the side of the potassium permanganate bottle. This reagent takes overnight stirring (minimum of 3 hours if absolutely necessary). Use stirring bar already in the reagent bottle for this purpose. It is very easy to contaminate with mercury.
10.1.7 Potassium persulfate: 5% solution, w/v: dissolve 100 grams of potassium persulfate in 2000 mLs D.I. water. Slight heating with stirring may be necessary to completely dissolve. The formation of crystals in this solution is not a problem.

10.2 STANDARDS

Purchased standards must be received with a Certificate of Analysis (COA). The standard must be logged into the LIMS to receive an ID which is then written on the COA. The COAs are then forwarded to the administrative department with ID indicated so PDF can be generated and associated for access within LIMS and appropriate archival.

10.2.1 Traceability

10.2.1.1 All reference materials are given a unique identifier within LIMS and labeled with the LIMS #. This record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number within LIMS as well as on the container's label.

10.2.1.2 All working standards made from reference materials shall be labeled with a unique LIMS ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded within LIMS. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

10.2.1.3. All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs). **All standards, blanks and samples are taken through the digestion process. Note**: standards for 245.1 do not require digestion and the group leader may exercise the option NOT to digest the standards. This decision must be documented.

10.2.1.4 Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique LIMS identifier.

10.2.1.5 Primary source and secondary source mercury standard solutions at 200 ug/L: dilute 2 mLs of stock solution to 1000 mLs in a 1000 mL volumetric flask, with 1.5 mLs concentrated HNO_3. This solution is recorded in LIMS and given a unique LIMS identifier.

10.2.2 Calibration Standards

Prepared from the primary source working standard. The preparation of the calibration standards, etc. is described below.

10.2.2.1 Dilute the volumes below to 30 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 10 mLs for the initial step of the digestion. From that point when 25 mLs of DI water are added to samples, 15 mLs of DI water is added to the standards.

ug/L Hg	mLs of 200 ug/L standard in 30 mLs
0.20	0.03
0.50	0.075
1.0	0.15
2.0	0.30
4.0	0.60
6.0	0.90
10.0	1.5

10.2.2.2 Appropriate reagents are added as below in the sample preparation section.

10.2.2.3 Prepare one vessel for each.

10.2.2.4 It is necessary to digest the calibration standards.

10.2.3 Calibration Verification Standards

10.2.3.1. Initial calibration verification (ICV) solution – 4.0 ug/L

10.2.3.1.1 Prepared by diluting 0.6 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel. (TV = 4.0 ug/L)

10.2.3.1.2 Appropriate reagents are added as below in the sample preparation section.

10.2.3.1.3 It is necessary to digest the ICV standards for Method 7470A. While all standards are routinely digested, standards for 245.1 do not require digestion and may be undigested with documentation of an undigested curve.

10.2.3.2 Continuing calibration verification (CCV) solution

10.2.3.2.1 Prepared from the primary source standard.

10.2.3.2.2 Prepared by diluting 0.3 mL of the primary standard at 200 ug/L to 30 mLs with reagent water in a 70 mL polypropylene vessel for 2.0 ug/L or 0.6 ml to 30 mLs for 4.0 ug/L.

10.2.3.2.3 Appropriate reagents are added as below in the sample preparation section.

10.2.3.2.4 It is necessary to digest the CCV standards for Method 7470A. While all standards are routinely digested, standards for 245.1 do not require digestion and may be undigested with documentation of an undigested curve.

10.2.4 Digestion standards

10.2.4.1 Blank Spike

10.2.4.1.1 Prepared from the secondary source standard.

10.2.4.1.2 Prepared by diluting 0.3 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel.

10.2.4.1.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.1.4 This solution should be given a unique identifier within LIMS.

10.2.1.2 Matrix Spikes

10.2.1.2.1 Prepared from the secondary source working standard.

10.2.1.2.2 Prepared by diluting 0.3 mL of the second source standard to 30 mL with sample in a 70 mL polypropylene vessel. Project specific or method specific requirements may over-ride the spiking level.

10.2.1.2.3 Appropriate reagents are added as below in the sample preparation section.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Samples are preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection, and refrigeration to 4°C. Note – samples received unpreserved and preserved in-house must be held 24 hours prior to preparation.

11.2 The holding time for the mercury digestion and analysis is 28 days from time of sampling.

12.0 Quality Control

12.1 Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2 An initial demonstration must be performed by each analyst performing this method. Four BS's are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria. **(Reference SW-846, 7470A Update III, or 245.1, Rev 3.0, 5/94 for further clarification)**

12.3 An initial demonstration of instrument linear range is required for EPA 245.1. Analyze 3 or more standards to determine highest standard providing recovery within ±10% of true value. Maintain documentation for future reference. While the method indicates the linear dynamic range should be verified annually or whenever a change in analytical performance indicates it should be re-determined, this is only applicable if it is being used to report samples outside the range of the daily calibration curve.

12.4 Daily **See table 2 for criteria associated to the following.**

12.4.1. **The instrument must be calibrated daily for all projects.**

12.4.2 Begin each analysis with an ICV (QCS) second source.

12.4.3 Analyze ICB.

12.4.4 If the ICV (QCS) is not in control a new curve must be analyzed prior to sample analysis.

12.4.5 If the IPC (initial CCV) for 245.1 is not within the limits, try preparing another undigested CCV and reanalyzing before recalibrating. If this fails then a recalibration is necessary.

12.4.6 Follow each set of 10 samples with a CCV and also end up with a CCV after the last sample.

12.4.7 A CCB must always follow a CCV. CCB must be run at the beginning and end of a sequence and after every 10 samples.

12.5 Digestion **See table 2 for criteria associated to the following.**

12.5.1 Laboratory control sample (BS) - Employ a minimum of one laboratory control sample (BS) per sample batch to verify the digestion procedure. The BS is taken through the same digestion/preparation steps as the samples being tested. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The project manager or QA Officer will make this determination.

12.5.2 BLK - Employ a minimum of one preparation blank (BLK) per sample batch to determine if contamination or any memory effects are occurring. The BLK is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be below the method detection limit. If the BLK is not within table 2 criteria, the impact upon the client data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The project manager or QA Officer will make this determination.

12.6 Sample matrix:

12.6.1 Analyze one replicate sample for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations.

12.6.2 Analyze one spiked sample and spiked sample duplicate for every batch of one to twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should meet table 2 criteria. If not, check with group leader to determine appropriate action. The final analytical report must document this situation.

NOTE: For TCLP extracts, a matrix spike (**and a matrix spike duplicate, if no other MSD in the batch)** must be performed for each matrix (soil or water). The method of standard additions must be used if the sample spike recoveries are not at least 50% and the concentration of Hg does not exceed the regulatory level and if the concentration of Hg measured in the extract is within 20% of the regulatory level.

12.6.3 The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the calculation SOP. The group leader must be notified if the control limit is not met. They will determine corrective action if required. The final analytical report must document this situation.

12.6.4 For 245.1 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.

12.6.5 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

12.7 Quarterly LOD/LOQ verifications. MDL studies are completed at setup and when a significant process change is made. Detection limits are set based upon MDL studies and validated with quarterly LOD/LOQ verifications.

13.0 Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Set up the instrument with proper operating parameters.

13.3 Perkin Elmer Flow Injection Mercury System (FIMS).

13.3.1 Prepare the instrument for calibration by the following steps:

13.3.1.1 Change or rotate pump tubing. 2 blue/yellow, 1 black/white, 1 red. Top of pump has 1 Blue/Yellow in back as 3% HCl carrier intake and red in front for SnCl intake.

13.3.1.2 Bottom of pump has Black/White in back as waste from manifold and a Blue/Yellow in front as sample waste.

13.3.2 Change manifold tube and filter. Can use nitrogen to blow tubing dry. Filter goes smooth side up. Record in maintenance logbook that tubes and filter have been changed.

13.3.3 Loosen pump tightener screws and clamp tubes.

13.3.4 Put reagent inflow tubings [SnCl (red) & 3% HCl (blue)] and Sample intake line in DI water. Make sure that instrument waste lines are in waste collection vessel.

13.3.5 Turn on PE 100 spectrophotometer

13.3.6 Wavelength = 253.7; smoothing points = 9; measurement = peak height; read time = 18 sec; BCC time = 2 sec.

13.3.7 Turn on the computer and double click on the icon "Winlab32 for AA open".

13.3.7.1 Wait for instrument to run pre-check.

13.3.8 Open FIAS.

13.3.8.1 Set pump speed to 60 rpm.

13.3.8.2 Click pump-one button so it turns green.

13.3.8.3 Pump will turn on an drum smoothly. If the pump makes a grinding noise, turn off pump and loosen screws more. Turn pump back on.

13.3.9 Upper right corner, click method. Select Hg CAL 2.

13.3.10 Upper right corner below method, click Sample Info.

13.3.10.1 Open similar old Sample Info File (SIF).

13.3.10.2 Save as current run name with water or soil designation (i.e. 071414W or 071414S)

13.3.10.3 Delete old information and put in all current information.

13.3.10.4 Make sure there is a CCV/CCB every 10 samples (SEQ-CCV, SEQ-CCB).

13.3.10.5 –OR- SIF can be created ahead of time using OFFLINE version of Winlab32 for AA and then opened when going to run.

13.3.11 Allow instrument to warm up for 10 to 20 minutes.

13.3.12 Check Nitrogen tank first for gas volume. If you run out of gas mid-run, you will have to change tank and recalibrate to finish run. Turn on nitrogen tank. Check front of instrument for confirmation of air flow. Should show between 50 to 100 psi. (floating white ball on right side above round black knob behind manifold)

13.3.13 Make 3% HCl carrier in 1L bottles x2. Make 1 bottle of SnCl.

13.3.14 Take reagent inflow tubing from DI water and put in the appropriate reagent bottle.

13.3.14.1 Put sample intake probe in auto-sampler arm. Fill small bucket with 3% HC1 rinse. To move auto-sampler to wash: Analysis: Auto-sampler: Go To Wash.

13.3.15 While instrument warms with reagents in place, load sample tray with samples according to SIF.

13.3.16 Once samples are loaded and instrument is warm: Click FIAS to bring pump control window to front.

13.3.16.1 Click pump-one to turn off.

13.3.16.2 Set speed to 120 rpm.

13.3.16.3 Turn pump back on.

13.3.16.4 Check/tighten tube screws.

13.3.16.5 Let run 2 to 5 minutes at higher setting.

13.3.17 Check Method and SIF in upper right corner to confirm they are correct.

13.3.18 Click FIAS and move box to upper left of window.

13.3.19 Click Auto and move under FIAS in lower left window.

13.3.19.1 Auto window will have SIF in left hand side and right hand side will be empty for data set.

13.3.19.2 Click **open** next to Data Set and save new data set with current run name. (wait, software is slow)

13.3.19.3 Bottom of Auto window has tab Analyze. Click for SIF to auto-populate.

13.3.20 Click Results and move box to upper right of window.13.3.21 Click Peaks and resize to fit under Results in lower right of window.13.3. 22 In place of 13.3.17 through 13.3.20 above, you can open Workspace, hg soils and waters.ffm and it will bring up all windows for you. You will have to set up SIF, Method, and Data Set in all appropriate locations. This is ok to use, but can cause software to crash, so use at own risk.

13.3.23 On Auto Window in Analyze tab, Click Calibrate.

13.3.23.1 Calibrate will run calibration only. After running it will print/show the curve info and then go into a "stand-by/ready" mode. Will not run samples.

13.3.24 After Calibration has run and is satisfactory, click Analyze Samples.

13.3.24.1 This will start with the ICV, ICB, and then the Samples.

13.3.24.2 If a sample or QC item fails, or needs to be rerun, click Analyze Samples again and click the appropriate action. Then ok.

13.3.24.2.1 Stop immediately.

13.3.24.2.2 Stop after this replicate.

13.3.24.2.3 Stop after all replicates for this sample

13.3.24.3 Once sample has stopped and auto-sampler is in the Home/Wash location, click Analyze Samples again and chose the appropriate option. Then ok.

13.3.24.3.1 Continue with next sample #,

13.3.24.3.2 Reanalyze previous seq # and continue,

13.3.24.3.3 Continue with seq # x (you fill in)

13.3.24.3.4 Restart current method.

13.3.24.4 If you reset your sequence due to an addition of a sample to the SIF or to fix a typo, it will take you to the ICV. Do not run the ICV. Click Analyze Samples, Stop Immediately, ok, click Analyze Samples again a and then tell it which sample # to run.

13.3.24.5 If you have no reason to restart the sequence or rerun any samples, the initial click of analyze samples will run smoothly until the end of the run.

13.3.25 When run is finished, turn pump on and put tube lines and sample line in DI water to rinse. Let rinse for 5 to 10 minutes. Can loosen tube screws and increase amount of DI run through instrument. After tubes are rinsed, take tubes out of DI and allow to run dry. Once waste tubes are dry, turn off pump and unclamp tubes. Do not run tubes dry any longer then necessary, it will cause undue wear on the instrument.

13.3.26 Turn off instrument.

13.3.27 Turn off Nitrogen. (verify off with instrument)

13.3.28 Export data in .csv. (procedure if automatic export does not work)

13.3.29.1 File, Utilities, Data Manager.

13.3.29.2 Once Data Manager comes up, choose/highlight the appropriate run/data set name.

13.3.29.3 Click Export in upper bar, Use Existing Design, Browse, Hg Data to be Transferred TEST, open. Finish, Export Data, Finish.

13.3.29.4 Close data manager.

13.3.29.5 Data will be in C:/Hg data to be Transferred. There is a shortcut on the desktop.

13.3.29.6 Transfer data to Hg Excel Spreadsheet and turn in data.

13.3.29 Close Winlab32 for AA

13.4 Analyze the calibration standards as below.

13.4.1 New calibration points must be analyzed when the ICV analysis is not within table 2 criteria. **A curve must be analyzed daily for all projects.**

13.4.2 The curve should be linear with a calculated intercept and meet table 2 criteria. If not, a new curve must be analyzed.

14.0 Procedure

14.1 Label the vessels indicating which sample will be in each.

14.2 Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below.

14.3 Sample preparation (see Circle Sheets added as Table 1):

Note – samples require check for residual chlorine. If KI paper turns dark blue/black, check interference section for procedure to follow.

14.3.1. Transfer 30 mLs, or an aliquot diluted to 30 mLs, of sample to the 30 mL mark on a 50 mL digestion vessel previously marked for this sample.

NOTE: Normally, an automatic dilution of 10X to 100X is performed for all TCLP extracts. All TCLP samples get a matrix spike and matrix spike duplicate, unless several come in at one time with the same matrix. Then one in ten of the same matrix gets spiked. Check with your manager.

14.3.2 Add 1.5 mLs of concentrated sulfuric acid to each vessel and mix.

14.3.3 Add 0.75 mL of concentrated nitric acid to each bottle and mix.

14.3.4 Add 4.5 mLs potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 7.5 mLs). If the purple color does not persist after the addition of 7.5 mLs $KMnO_4$ the sample must be diluted prior to digestion. Inform your manager that the minimum detection limit cannot be reached for that particular matrix.

NOTE: The same amount of $KMnO_4$ added to the samples should be present in the standards and blanks.

14.3.5 Add 2.4 mLs of potassium persulfate to each vessel and mix. Cover.

14.3.6 Record the time placed on the digestion block Heat for 2 hours in the block digester at 95±2°C. Monitor/Document the block temperature – recording observed temperature, correction factor, and the corrected temperature. Record time and temperature when the samples were put in the block and when they were removed from the digestion block. Cool.

14.3.7 Samples may be saved at this point if there is not time to run the whole set that day.

NOTE: Stannous Chloride (VII. A 5.) and 3% HCl (VII. A 8.) are added by the instrument during analysis.

14.4 Sample analysis

14.4.1 Set up the instrument as described in the calibration section above.

14.4.2 When ready to run samples, add 1.8 mLs of sodium chloride-hydroxylamine chloride to reduce the excess permanganate and dilute to 40 mLs with DI water, cap and shake until clear. Sample analysis must be preceded by the analysis of an ICV within table 2 control limits. Followed by the ICB within table 2 criteria.

14.4.3 Each set of ten samples and at the end of the analytical run must be followed by a CCV within table 2 control limits.

14.4.4 CCB must always follow the CCV within table 2 criteria.. CCB must be run at the beginning and end of a sequence and after every 10 samples.

14.4.5 The auto-sampler log is set up to analyze 106 samples at a time.

Instrument Run Log example:

AS LOC **Sample ID**

0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	SEQ-ICV
10	SEQ-ICB
11	CRL
12	BSI

AS LOC	**Sample ID**
13	BLK
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	Sample
22	Sample
23	Sample
24	SEQ-CCV
25	SEQ-CCB
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	Sample
32	Sample
33	Sample
34	Sample
35	Sample
36	MS
37	MSD
38	SEQ-CCV
39	SEQ-CCB

14.5 Data Reporting

14.5.1 Reduce data to result which will be reported.

14.5.2 Complete the data review checklist (attached). Must be completed and attached to each set of DOD data.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 The Excel file for calculations is located at: "V:\Standard Operating Procedures\Current SOP File Directory\Worksheets" or can be accessed through TOC_SOPs_Controlled_Documents spreadsheet at V:\Standard Operating Procedures. Pull up the blank spreadsheet and transfer all the information pertinent to the current analysis. Save as the date of analysis with "W" for water and/or "S" for soil at the end i.e. 071014W for waters. This information can be obtained from your mercury batch sheet.

15.3 Transfer the sample absorbance into the excel spreadsheet in the appropriate cell. The spreadsheet uses the current calibration to calculate the Hg results.

15.4 Make sure that the appropriate dilution factors are entered into the spreadsheet in the correct cells.

15.5 The spreadsheet should divide the result which is the µg Hg obtained from the sample volume by the sample volume in L. This will yield a result of µg Hg/L sample. Calculations in the spreadsheet should be checked to make sure that they are working correctly.

15.6 Report the data as µg Hg/L of sample.

16.0 Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed in Quality Control SOP QS03 is completed by each analyst and then provided to the group leader for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM, DOD limits will be used.**

DOC BS Preparation: Dilute 0.3 mL of the second source standard to 30 mLs with reagent water in a 70 mL polypropylene vessel. Follow SOP procedure for preparation and analysis steps.

DOC Accuracy and Precision Criteria: The four BS's for the DOC need to be within the methods (or DODs) recovery ranges. Duplicates should be at or below 20% relative percent difference.

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Corrective Actions and Out-of Control Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20.0 Contingencies for Handling out-of-control or unacceptable data

20.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.2 CORRECTIVE ACTIONS: INSTRUMENT RELATED

20.2.1 ICV (**QCS for 245.1**)- second source not within table 2 criteria

A. If the problem is with the solution, re-prepare, obtain new stock if necessary.

B. If the problem is with the calibration, recalibrate through analysis of appropriate standards and recheck ICV.

20.2.2 CCV not within table 2 criteria

A. If the problem is with the solution, re-prepare, obtain new stock if necessary.

B. If the problem is with the calibration, recalibrate through analysis of appropriate standards and re-prepare/reanalyze the previous ten sample according the following guidelines.

1. If the CCV was biased high, any of the previous ten samples which were below the detection limit do not require reanalysis.

2. If the CCV was biased low, the previous ten samples must be reanalyzed.

20.3 CORRECTIVE ACTION: DIGESTION RELATED

20.3.1 The preparation blank not within table 2 criteria

A. If the problem is with the instrument or stannous chloride.

Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, re-prepare the stannous chloride or determine if there are any problems with the instrument. Contact group leader immediately.

B. If the problem is with the digestion.

All associated samples which are below the RL, CRDL or have a level of mercury greater than 5X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is neither BMDL nor greater than 5 X the level found in the preparation blank, the sample must be re-digested/re-analyzed or reported as qualified. The project manager or QA manager will make this determination.

C. LCS not within not within table 2 criteria

1. If the problem is with the instrument, reanalyze when instrument is in control if further sample bottles are available.

2. Is the problem is with the digestion.

a. If biased low, associated samples must be re-digested.

b. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

20.4 CORRECTIVE ACTION: SAMPLE MATRIX RELATED

20.4.1 Replicate analysis RPD not within not within table 2 criteria

The associated sample data must be qualified on the final report.

20.4.2 Spike analysis recovery not within not within table 2 criteria

A. If the analyte level in the sample is greater than 4X the spiking level, the %recovery should not be evaluated and no action is taken.

B. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. TCLP extracts must be evaluated as in section XI.D.2 above. The associated sample data must be qualified on the final report.

20.4.3 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

21.0 Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

22. Equipment/instrument maintenance

22.1 Rotate or change tubing with each analysis. Record in maintenance log.

22.2 Change Millipore –filters- Fluoropore PTFE 1.0 um wh pl 25 mm with each analysis. Record in maintenance log.

22.3 Check line coming from the manifold that contains the above filter and goes to the Hg cell for moisture. Change or dry with nitrogen gas, if needed, before use .

23. Computer hardware and software

23.1 Instrument: Perkin Elmer Flow Injection Mercury System FIAS 100

23.2 Software: Winlabs2 for AA version 6.5.0266 @ 2007

24. Troubleshooting.

24.1 Sample Line has clog and will not pull, or sample waste line will not stay attached due to pressure. Rinse out sample probe with DI and very fine wire. Rinse sample line with DI or 3% Nitric. If this does not fix problem, unhook all tubes from FIAS Valve and disassemble using instructions from manufacturer's Maintenance book located near instrument and computer. Rinse all holes and ports with DI water to check for flow and loosen any clog or determine specifically where the clog is. Reassemble and test flow.

24.1.1 Any other tubing can get a blockage. Replacing is fastest/easiest way to fix. If replace is not an option at the time, soak in DI or 3% nitric to loosen clog. Always rinse out 3% Nitric with DI.

24.2 Mixing blocks have clog/blockage. Rinse with DI or 3% Nitric. If this does not work, soak in DI for a few hours to loosen clog. Can use fine wire to push out clog. If problem still persists, put mixing blocks in sonicator for an hour or so. If problem still persists, throw away blocks and replace with new.

24.3 If pump lines are leaking near pump, turn off pump and check tubes. May have worn a hole. Replace with new tube.

24.4 If a port leaks, tighten the center screw.

25.0 References

25.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update II); Method 7470A.

25.2 USEPA Code of Federal Regulations, 40, CH 1,PT 136; Method 245.1 Revision 3.0; APX-B.

25.3 DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003.) Dated 10/25/2010

25.4 Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories. Based on ISO/IEC 17025:2005(E) and The NELAC Institute (TNI) Standards, Volume 1, (September 2009) Dated July 2013

26.0 Tables, Diagrams, Flowcharts and Validation Data

26.1 Table 1, Eppendorf/Standard/Reagent Circle Sheet

26.2 Table 2, Method Quality Control Requirements Summary

26.3 Table 3, Data Reviewers Checklist

Table 1 - Mercury:
Circle Applicable Digestion/Spike and 0.1mL Eppendorf option then staple to bench sheet

Reminder: Make sure pipettes are verified daily at volume of use for QSM5 batches!

Water

Amount	Curve Standards 7470 Water
0.0 mL	
0.03mL	Mercury 1st Source (0.2ug/mL)
0.075mL	Mercury 1st Source (0.2ug/mL)
0.15mL	Mercury 1st Source (0.2ug/mL)
0.30mL	Mercury 1st Source (0.2ug/mL)
0.60mL	Mercury 1st Source (0.2ug/mL)
0.90mL	Mercury 1st Source (0.2ug/mL)
1.5mL	Mercury 1st Source (0.2ug/mL)
Circle	0.1ml Eppendorf 148897Z or 250847
Circle	1.0mL Eppendorf 1854694 or FinnPipette 1FJ61693
	5mL FinnPipette KU04206

Amount	BS/MS/MSD Standards 7470 Water
0.30mL	Mercury 2nd Source (0.2ug/mL)
Circle	1.0mL Eppendorf 1854694 or FinnPipette 1FJ61693
Amount	**Reagents for Digestion Water/TCLP**
1.5mL	H2SO4
0.75mL	HNO3
4.5mL	Potassium Permanganate
2.4mL	Potassium Persulfate

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Initial calibration (ICAL)	• Daily ICAL prior to sample analysis • Low standard at the RL/LOD level • Minimum 5 points and calibration blank	• r-squared $\geq$ 0.990 or r $\geq$ 0.995 • Must follow curve processing requirements from SOP QS08	• Re-run curve • Check instrument for maintenance needs Samples cannot be analyzed until there is a passing calibration
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Must be within $\pm$ 10% of true value	• Re-run ICV • Repeat ICAL
Continuing calibration verification (CCV)	• After every 10 field samples and at the end of analysis sequence.	• $\pm$ 20% of true value for 7470A and DoD QSM 4.2 • $\pm$ 10% of true value for DoD QSM 5.0	• Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful CCV. • Two passing CCVs analyzed immediately following a failed CCV can be used to report unqualified data.
Method Blank (BLK)	One per prep batch	No analytes detected > ½ LOQ or greater that 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	• Re-analysis to confirm the positive value • Notify the PM for further action • Re-prep of samples associated with the BLK • NCR will be required for data reported
ICB/CCB	Following ICV but before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-analyze calibration blank. All samples following the last acceptable calibration blank must be reanlyzed.
BS	One per prep batch	7470A/DoD QSM4.2 80%-120% DoD QSM5.0 82%-119%	• Re-analyze to confirm failed. • Re-prep and reanalyze BS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. • NCR will be required for data reported
MS	One per prep batch, if sample volume available.	7470A/DoD QSM4.2 80%-120% DoD QSM5.0 82%-119%	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	7470A/DoD QSM4.2 80%-120% DoD QSM5.0 82%-119% RPD 20%	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between within BS limits with less than or equal to a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	At setup and when a significant process change is made.	• See QS09 for calculations/criteria .	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	• Parameter must be detected • The response must be 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	• Bias Requirement at 1-2x the LOQ Recovery within BS limits. • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

Table 3, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	**Sequence ID:**
Calibration:	**NCR#:**
Method: 7470A/245.1 (Mercury)	

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard?				
(If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did BS meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation Blank (BLK) below the necessary limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was water bath temperature monitored/documented and did you apply the thermometer correction factor?				
1. Include the water bath Time ON:_____Time OFF:_____from bench sheet.				
11. Sample preparation information is correct and complete.				
12. Analytical results are correct and complete.				
13. The appropriate SOP's have been followed.				
14. "Raw data" including all manual integration's have been correctly interpreted.				
15. "Special" sample preparation and analytical requirements have been met.				
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, non-conformance reports are complete.				
17. Data has been uploaded to the LIMS with correct analyst and analysis factors included.				
18. Reagents/Standards verified accurate on bench sheets and in LIMS.				
19. Calculation is shown on the raw data for sample _____to validate final concentration in LIMS.				

Comments on any "No" response:

Analyst: Date:

Second-Level Review: Date:

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 104 REVISION #: 26 EFFECTIVE DATE: 20160831

MERCURY ANALYSIS IN SOIL/SEDIMENT BY MANUAL COLD VAPOR TECHNIQUE METHODS SW846 7471A or 7471B

APPROVALS:

Lab Director: _____ Date: **20160831**

Data Quality Manager: _____ Date: **20160831**

Group Leader: _____ Date: **20160831**
Betty Quillen

Changes Summary

SOP104_R26_20160831_HGDIGS
- Preparation of IDOC removed from section 16 other than reference to BS.
- Sample weight updated from 0.2-1.0 to 0.25g-0.35g in keeping with ½ method reference of 0.5g-0.6g.
- Consolidated sequence in 14.3.7.
- Changed 15.5.5 to read check per sequence rather than occasionally.

SOP104_R25_20151221_HGDIGS
- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.

SOP104_R24_20150921_HGDIGS
- All references to supervisor updated to reflect group leader.
- Section 9 updated for equipment.
- Section 10 updated to reference QS04
- Section 14.3 updated to include KI check
- Section 15.2 updated for worksheet location
- Section 22 updated with troubleshooting tips.
- Circle sheets updated.
- Added immediate analysis of 2 passing CCVs as option in table 2.
- Table 3 removed and Data Reviewer Checklist updated to reflect Table 3.

Revision 23, 20140721
- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark update to include proprietary reference.
- Reference to SOP193 updated to reflect QS08.
- 9.0 Equipment and supplies updated.
- 10.4.3 Preparation of calibration standards clarified.
- 13.3 Procedure clarified.
- Limit references in main text updated to reference table 2 throughout the document.
- 22.0, 23.0 and 24.0 added/populated.
- 25.0 updated
- Table 2 updated to include QSM5.0 criteria.

Revision 22, 20130903
- References to CLP and 245.5 removed.
- Detection Limit as Table 1 removed and Eppendorf/Reagent/Standard circle sheets added as Table 1.
- Requirement to report time on and time off for digestion added to procedure and checklist.
- Removed reference to linear correlation coefficient criteria of 0.998.

Revision 21, 20121031
- **"**Ten vessels of each lot must be taken through volume verification as per SOP193 and recorded in the non-volumetric labware calibration logbook." added to section 9.6.

Revision 20, 20120813
- Added 2 samples between CCV/CCB for example sequence
- Updated analyst review checklist

- Added instructions for starting/running instrument
- Added requirement for certificate of analysis on purchased standards.

Revision 19, 20100411

- The SOP is an update from Revision 18 dated 03/25/10.
- Software instructions have been updated in section 13 to reflect new software.
- References to old 400 level SOPs have been removed.

Revision 18, 20100308

- The SOP is an update from Revision 17 dated 01/29/09.
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DOD samples are analyzed.
- Numerous improvements/modifications were made to this SOP. Details/specifications were added that require evaluation from start to finish.

Table of Contents

MERCURY ANALYSIS IN SOIL/SEDIMENT
BY MANUAL COLD VAPOR TECHNIQUE
METHODS SW846 7471A or 7471B

1.0 Identification of the Test Method

1.1 This SOP is compliant with SW-846 methods 7471A and 7471B.

2.0 Applicable Matrix or Matrices

2.1 This procedure measures total mercury (organic and inorganic) in soils, sediments, bottom deposits and sludge type materials.

3.0 Limits of Detection and Quantitation

3.1 The range of the method is 0.2 to 2 µg/g. The range may be extended above or below the normal range by increasing or decreasing sample size or through instrument and recorder control.

3.2 Method Detection Limit (MDL)/Detection Limit (DL), Limit of Detection (LOD) and Empirical Laboratories' Reporting Limit (ERL)/Limit of Quantitation (LOQ) and Analyte Wavelength:

Limits Table

Soil/Solid Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ),			
Mercury by 7471A, 7471B	**SOLID/SOIL MDL/DL (mg/Kg)**	**SOLID/SOIL LOD (mg/Kg)**	**SOLID/SOIL ERL/LOQ (mg/Kg)**
Mercury (Wavelength 253.7)	0.013	0.026	0.033

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in the Table above.

4.2 This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

5.1 A weighed portion of the sample is acid digested for 2 minutes at 95±2°C, followed by oxidation with potassium permanganate and with a secondary digestion at 95°C for 30 minutes. Mercury in the digested sample is then measured by the conventional cold vapor technique.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

7.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.

7.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.

7.3 Samples high in chlorides require additional permanganate (as much as 12.5 mLs) because chlorides are converted to free chlorine during the oxidation step, which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell.

7.4 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.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab-wide.

8.2 Normal accepted laboratory practices should be followed while performing this procedure.

8.3 The toxicity and carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory safety practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

8.4 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analyses should be conducted in a laboratory exhaust hood. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

8.5 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of nitrile gloves and lab coats is highly recommended.

8.6 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.7 MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9.0 Equipment & Supplies

9.1 Perkin Elmer Flow Injection Mercury System (FIMS).
9.2 Perkin Elmer S10 Autosampler.
9.3 Mercury lamp-FIMS (PE) B0510487
9.4 Yellow/blue pump tubing pk/12 Perkin Elmer (PE) B0193161
9.5 Tubing per pump 3.18mm bl/wh 40 cm pk/12 (PE) B0508310
9.6 Red/red pump tubing pk/12 (PE) B0193160
9.7 Mixing blocks (PE) B0507962
9.8 Quartz Window (PE) B0066549
9.9 PTFE tubing 1000 mm (PE) B0191060 (sample line from autosampler to intake valve)
9.10 PTFE tubing 700 mm (PE) B0191059 (for HCl and SnCl)
9.11 PTFE tubing 300 mm (PE) B0198097 (line from manifold to light tube)
9.12 500 uL sample loop (PE) B0194049
9.13 Environmental Express Mod-Block digestion block capable of holding 95+2°C for 2 hours.

9.14 A scale or balance capable of weighing to 0.01 + 0.02 gram.
9.15 Snap cap digestion polypropylene vessels for use with the mod block digester. Before use, five vessels of each lot number must be taken through blank analysis to verify mercury levels <1/2LOQ. Ten vessels of each lot number must also be taken through volume verification as per SOP QS08 and recorded in the non-volumetric labware calibration logbook.
9.16 Polypropylene watch glasses suitable for use with the above vessels.
9.17 10 mm sieve used for special prep when requested.
9.18 Manual Sample Mill (used for chopping up vegetation)
9.19 Wiley Sample Mill (used for chopping up vegetation)
9.20 Clippers for cutting vegetation
9.21 Millipore –filters- Fluoropore PTFE 1.0 um wh pl 25 mm 100/pk

10.0 Reagents and Standards

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST - RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

10.2 Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Spex, Ultra and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at 4 ° C.

10.3 REAGENTS

10.3.1 Reagent Water: Reagent water will be interference free. All references to water in this method refer to reagent water unless otherwise specified.

10.3.2 Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3. Both HNO_3 and HCl must be ACS grade.

10.3.3 Concentrated HCl.

10.3.4 Concentrated HNO_3.

10.3.5 Stannous chloride in a one liter volumetric flask add ~500 mL D.I. H_2O, 30 mL concentrated HCl, and 11g stannous chloride crystals. Swirl to mix and dilute to 1 L. (must be prepared fresh daily).

10.3.6 Sodium chloride-hydroxylamine chloride solution: Dissolve 120 g of sodium chloride and 120 g of hydroxylamine sulfate in reagent water and dilute to 1 L. Note: this is normally made up 2 liters at a time.

10.3.7 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 200 g of potassium permanganate in 4 L of reagent water.

10.3.8 3 % HCl carrier solution: 30 mLs HCl – 1 L DI H20; Prepare fresh daily.

10.4 STANDARDS

Purchased standards must be received with a Certificate of Analysis (COA). The standard must be logged into the LIMS to receive an ID which is then written on the COA. The COAs are then forwarded to the administrative department with ID indicated so PDF can be generated and associated for access within Element and appropriate archival.

10.4.1 Traceability

10.4.1.1 All reference materials are given a unique identifier within Element and labeled with the Element #. This record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number within Element as well as on the container's label.

10.4.1.2 All working standards made from reference materials must be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded within Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

10.4.2 Preparation

10.4.2.1. **NOTE:** All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs). All Standards, blanks, and samples are taken through the digestion process.

10.4.2.2 Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique identifier.

10.4.2.3 Primary source and secondary source mercury standard solutions: dilute 2 mLs of stock solution to 1000 mLs in a 1000 mL volumetric flask, with 1.5 mLs concentrated HNO_3 (200 ug/L).

10.4.3 Calibration standards:

Prepared from the primary source standard. The preparation of the calibration standards, etc. is described below.

10.4.3.1 To prepare the calibration curve dilute the volumes below to 5 mLs in seven separate 70 mL polypropylene vessels. **(Note: The standards are diluted to 5 mLs for the initial step of the digestion.)**

ug/L Hg	mLs of 200 ug/L standard in 5.0 mLs for initial digestion step with a final volume of 50 mLs
0.20	0.050
0.50	0.125
1.0	0.25
2.0	0.50
4.0	1.0
6.0	1.5
10.0	2.5

10.4.3.2 Appropriate reagents are added as below in the sample preparation section.

10.4.3.3 It is necessary to digest the calibration standards when following all mercury

methods.

10.4.4 Calibration verification standards:

10.4.4.1. Initial calibration verification (ICV) solution – 4.0 ug/L.

10.4.4.1.1 Prepared from the secondary source mercury standard (200 ug/L).

10.4.4.1.2 Prepared by diluting 1.0 mL of the second source mercury standard to 5 mLs in a polypropylene digestion vessel.

10.4.4.1.3 Appropriate reagents are added as below in the sample preparation section.

10.4.4.1.4 It is necessary to digest the ICV standards when using all mercury methods for soil.

10.4.4.2 Continuing calibration verification (CCV) solution:

10.4.4.2.1 Prepared from the primary or secondary source mercury standard. The concentration is alternated from 2.0 ug/L to 4.0 ug/L every 20 samples.

10.4.4.2.2 Prepared by diluting 0.50 for a 2.0 ug/L and 1.0 mL for a 4.0 ug/L of the secondary 200 ug/L standard to 5.0 mLs with reagent water in a polypropylene digestion vessel.

10.4.4.2.3 Appropriate reagents are added as below in the sample preparation section.

10.4.4.2.4 It is necessary to digest the CCV standards when following all mercury methods for soil.

10.5.5 Digestion standards:

10.5.5.1 Laboratory control sample:

10.5.5.1.1 The Laboratory Control Sample (BS) is prepared by diluting 0.50 mL of the secondary mercury standard (200 ug/L) to 5 mLs in a polypropylene digestion vessel with 0.30 grams of glass beads.

10.5.5.1.2 Appropriate reagents are added as below in the sample preparation section.

10.5.5.1.3 This solution is given a unique identifier in Element.

10.5.5.2 Matrix Spikes

10.5.5.2.1 Prepared from the primary or secondary source mercury standard (200ug/L).

10.5.5.2.2 Prepared by adding 0.50 mL of the mercury standard (200ug/L) to the sample in a polypropylene digestion vessel. Project specific requirements may over-ride the spiking level.

10.5.5.2.3 Appropriate reagents are added as below in the sample preparation section.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 Because of the extreme sensitivity of the analytical procedure and the omnipresence of mercury, care must be taken to avoid extraneous contamination. Sampling devices and sample containers should be ascertained to be free of mercury; the sample should not be exposed to any condition in the lab that may result in contact with solid, liquid or airborne mercury.

11.3 Refrigerate solid samples at 0°C-6°C upon receipt until digestion and analysis.

11.4 The sample should be analyzed without drying. A separate percent solids determination is required

11.5 The holding time for digestion of mercury samples is 28 days.

12.0 Quality Control

12.1 Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2 An initial demonstration must be performed by each analyst performing this method. Four BSs are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria.

12.3 QUALITY CONTROL (see references for further clarification)

12.3.1 Daily (See table 2 for criteria)

12.3.1.1 The instrument must be calibrated daily for all projects.

12.3.1.2 Begin each analysis with an ICV (concentration at or near mid range).

12.3.1.3 Analyze ICB.

12.3.1.4 If the ICV is not in control a new curve must be analyzed prior to sample analysis.

12.3.1.5 Follow each set of 10 samples with a CCV and also must end up with CCV after last sample. If an exceedence occurs, analyze another CCV (2 passing required for DoD QSM), if the second CCV fails, then a new calibration curve should be generated and all affected samples should be reanalyzed.

12.3.1.6 Follow each CCV with a CCB.

12.3.2 Quarterly (See table 2 for criteria)

12.3.2.1 LOD/LOQ verifications. MDL studies are completed at setup and when a significant process change is made. Detection limits are set based upon MDL studies and validated with quarterly LOD/LOQ verifications. See Table 2 for criteria.

12.3.3 Digestion (See table 2 for criteria)

12.3.3.1 BS data should be maintained and available for easy reference or inspection.

12.3.3.2 BLK

12.3.3.2.1 Employ a minimum of one BLK per sample batch to determine if contamination or any memory effects are occurring. The preparation blank is taken through the same digestion/preparation steps as the samples being tested.

12.3.3.3 Laboratory control sample (BS).

12.3.3.3.1 Employ a minimum of one BS per sample batch to verify the digestion procedure. The BS is taken through the same digestion/preparation steps as the samples being tested. A BS will accompany each batch of soil samples. If the BS is not in control, the group leader must be notified immediately. Several possibilities exist at this point and a thorough investigation and data evaluation is essential. The first question is to evaluate the impact upon the data. All samples may need to be retested or flagged with the appropriate qualifier. The next question is to find out why it occurred and to proceed with a corrective action plan to prevent reoccurrence. This non-conformance is documented in a NCR.

12.3.4 Sample matrix (See table 2 for criteria)

12.3.4.1 Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations.

12.3.4.2 Analyze one spiked sample and spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process

in duplicate. Project specific requirements will take precedence in these situations. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within table 2 criteria. If results do not fall within the control limit re-digestion/reanalysis may be required. If reanalysis is not required, the associated batch of samples will be flagged accordingly. Discuss the situation with your group leader. A non-conformance report (NCR) must be filled out and attached to the data as well as emailed or sent to the group leader when the control limits are exceeded.

12.3.4.3 The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. Group leader must be notified if the control limit is not met. Group leader will determine corrective action if required. The final analytical report must document this situation. A non-conformance report (NCR) must be filled out and attached to the data as well as emailed or sent to the group leader when the control limits are exceeded.

12.3.4.4 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) may be appropriate. Section 8.6

13.0 Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Set up the instrument with proper operating parameters.

13.3 Perkin Elmer Flow Injection Mercury System (FIMS).

13.3.1 Prepare the instrument for calibration by the following steps:

13.3.1.1 Change or rotate pump tubing. 2 blue/yellow, 1 black/white, 1 red. Top of pump has 1 Blue/Yellow in back as 3% HCl carrier intake and red in front for SnCl intake.

13.3.1.2 Bottom of pump has Black/White in back as waste from manifold and a Blue/Yellow in front as sample waste.

13.3.2 Change manifold tube and filter. Can use nitrogen to blow tubing dry. Filter goes smooth side up. Record in maintenance logbook that tubes and filter have been changed.

13.3.3 Loosen pump tightener screws and clamp tubes.

13.3.4 Put reagent inflow tubings [SnCl (red) & 3% HCl (blue)] and Sample intake line in DI water. Make sure that instrument waste lines are in waste collection vessel.

13.3.5 Turn on PE 100 spectrophotometer

13.3.6 Wavelength = 253.7; smoothing points = 9; measurement = peak height; read time = 18 sec; BCC time = 2 sec.

13.3.7 Turn on the computer and double click on the icon "Winlab32 for AA" to open.

13.3.7.1 Wait for instrument to run pre-check.

13.3.8 Open FIAS.

13.3.8.1 Set pump speed to 60 rpm.

13.3.8.2 Click pump-one button so it turns green.

13.3.8.3 Pump will turn on and run smoothly. If the pump makes a grinding noise, turn off pump and loosen screws more. Turn pump back on.

13.3.9 Upper right corner, click method. Select Hg CAL 2.

13.3.10 Upper right corner below method, click Sample Info.

13.3.10.1 Open similar old Sample Info File (SIF).

13.3.10.2 Save as current run name with water or soil designation (i.e. 071414W or 071414S)

13.3.10.3 Delete old information and put in all current information.
13.3.10.4 Make sure there is a CCV/CCB every 10 samples (SEQ-CCV, SEQ-CCB).
13.3.10.5 –OR- SIF can be created ahead of time using OFFLINE version of Winlab32 for AA and then opened when going to run.
13.3.11 Allow instrument to warm up for 10 to 20 minutes.
13.3.12 Check Nitrogen tank first for gas volume. If you run out of gas mid-run, you will have to change tank and recalibrate to finish run. Turn on nitrogen tank. Check front of instrument for confirmation of air flow. Should show between 50 to 100 psi. (floating white ball on right side above round black knob behind manifold)
13.3.13 Make 3% HCl carrier in 1L bottles x2. Make 1 bottle of SnCl.
13.3.14 Take reagent inflow tubing from DI water and put in the appropriate reagent bottle.
13.3.14.1 Put sample intake probe in auto-sampler arm. Fill small bucket with 3% HC1 rinse. To move auto-sampler to wash: Analysis: Auto-sampler: Go To Wash.
13.3.15 While instrument warms with reagents in place, load sample tray with samples according to SIF.
13.3.16 Once samples are loaded and instrument is warm: Click FIAS to bring pump control window to front.
13.3.16.1 Click pump-one to turn off.
13.3.16.2 Set speed to 120 rpm.
13.3.16.3 Turn pump back on.
13.3.16.4 Check/tighten tube screws.
13.3.16.5 Let run 2 to 5 minutes at higher setting.
13.3.17 Check Method and SIF in upper right corner to confirm they are correct.
13.3.18 Click FIAS and move box to upper left of window.
13.3.19 Click Auto and move under FIAS in lower left window.
13.3.19.1 Auto window will have SIF in left hand side and right hand side will be empty for data set.
13.3.19.2 Click **open** next to Data Set and save new data set with current run name. (wait, software is slow)
13.3.19.3 Bottom of Auto window has tab Analyze. Click for SIF to auto-populate.
13.3.20 Click Results and move box to upper right of window.
13.3.21 Click Peaks and resize to fit under Results in lower right of window.
13.3.22 In place of 13.3.17 through 13.3.20 above, you can open Workspace, hg soils and waters .ffm and it will bring up all windows for you. You will have to set up SIF, Method, and Data Set in all appropriate locations. This is ok to use, but can cause software to crash, so use at own risk.
13.3.23 On Auto Window in Analyze tab, Click Calibrate.
13.3.23.1 Calibrate will run calibration only. After running it will print/show the curve info and then go into a "stand-by/ready" mode. Will not run samples.
13.3.24 After Calibration has run and is satisfactory, click Analyze Samples.
13.3.24.1 This will start with the ICV, ICB, and then the Samples.
13.3.24.2 If a sample or QC item fails, or needs to be rerun, click Analyze Samples again and click the appropriate action. Then ok.
13.3.24.2.1 Stop immediately.
13.3.24.2.2 Stop after this replicate.
13.3.24.2.3 Stop after all replicates for this sample
13.3.24.3 Once sample has stopped and auto-sampler is in the Home/Wash location, click Analyze Samples again and chose the appropriate option. Then ok.
13.3.24.3.1 Continue with next sample #,
13.3.24.3.2 Reanalyze previous seq # and continue,
13.3.24.3.3 Continue with seq # x (you fill in)

13.3.24.3.4 Restart current method.

13.3.24.4 If you reset your sequence due to an addition of a sample to the SIF or to fix a typo, it will take you to the ICV. Do not run the IC. Click Analyze Samples, Stop Immediately, ok, click Analyze Samples again a and then tell it which sample # to run.

13.3.24.5 If you have no reason to restart the sequence or rerun any samples, the initial click of analyze samples will run smoothly until the end of the run.

13.3.25 When run is finished, turn pump on and put tube lines and sample line in DI water to rinse. Rinse for 5 to 10 minutes. Can loosen tube screws and increase amount of DI run through instrument. After tubes are rinsed, take tubes out of DI and allow to dry. Once waste tubes are dry, turn off pump and unclamp tubes. Do not run tubes dry any longer then necessary, it will cause undue wear on the instrument.

13.3.26 Turn off instrument.

13.3.27 Turn off Nitrogen. (verify off with instrument)

13.3.28 Export data in .csv. (procedure if automatic export does not work)

13.3.29.1 File, Utilities, Data Manager.

13.3.29.2 Once Data Manager comes up, choose/highlight the appropriate run/data set name.

13.3.29.3 Click Export in upper bar, Use Existing Design, Browse, Hg Data to be Transferred TEST, open. Finish, Export Data, Finish.

13.3.29.4 Close data manager.

13.3.29.5 Data will be in C:/Hg data to be Transferred. There is a shortcut on the desktop.

13.3.29.6 Transfer data to Hg Excel Spreadsheet and turn in data.

13.3.29 Close Winlab32 for AA

13.3.30 Analyze the calibration standards as below.

13.3.30.1 A curve must be analyzed daily for all projects. A new curve must be analyzed when the ICV analysis is not within criteria indicated in table 2.

13.3.30.2 The curve should be linear with a calculated intercept with a minimum correlation coefficient indicated in table 2. If not, a new curve must be analyzed.

14.0 Procedure

14.1 Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below. Record the standard preparation in the standard log.

14.2 Sample preparation (see Circle Sheets added as Table 1):

14.2.1 It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

14.2.1.1 The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.

14.2.1.2 Two quarters should then be mixed to form halves.

14.2.1.3 The two halves should be mixed to form a homogenous matrix.

14.2.1.4 This procedure should be repeated several times until the sample is adequately mixed.

14.2.1.5 NOTE: Samples that are clay type materials must be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

14.2.1.2 Grinding of Vegetation Samples

14.2.1.2.1 Remove sample from shipping container and brush off dirt particles.

14.2.1.2.2 Chop sample into about half inch pieces with clippers or other cutting tool.

14.2.1.2.3 Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill.

14.1.2.2.4 Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed.

14.1.2.2.5 Place the ground sample in a container and label immediately.

14.2.2 Transfer 0.30 g (use anywhere from 0.25g to 0.35g and record the weight in the digestion log) of sample to a polypropylene digestion vessel previously marked for this sample. Record the exact sample mass on the bottle and on the Element Batch Sheet. (Note: the balance must be calibrated for the specific task. Calibrate by weighing a 0.5 and a 0.1g weight on the balance along with a digestion vessel. Record in specific balance calibration log.)

14.2.3 Add 2.5 mLs of reagent water, and 2.5 mLs of aqua regia and mix for samples. Add 2.5 mLs of aqua regia to standards and mix.

14.2.4 Cover samples and standards with watch glasses and heat for 2 minutes in the hot block at 95+2°C (The hot block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature).

14.2.5 Cool, bring to 30 ml with D.I. water.

14.2.6 Add 7.5 mLs potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 12.5 mLs).

NOTE: The same amount of KMnO4 added to the samples should be present in the standards and blanks.

14.2.7 Record Time On. Heat for 30 minutes on the hot block at 95+2°C. Monitor/Document the block temperature – recording observed temperature, correction factor, and the corrected temperature when samples are placed in the block and when the samples are removed from the block. Record time the samples are placed in the block and when they are removed from digestion block. Cool. Samples may be saved at this point if there is not time to run the whole set that day.

14.2.8 Add 3 mLs of sodium chloride-hydroxylamine chloride solution to each vessel.

14.2.9 Bring to 50 mLs with D.I. water both standards and samples. Cap mix and vent to decolor and release Cl gas. Let samples stand at least an hour after this process to allow time to settle and off gas the Cl2. The samples are now ready for analysis.

14.2.10 NOTE: Stannous Chloride (10.1.5) and 3% HCl (10.1.8) are added by the instrument during analysis.

14.3 Sample analysis

14.3.1 Set up the instrument as described in the calibration section above.

14.3.2 When ready to run samples, transfer samples and standards to autosampler tubes and load the auto sampler according to the sample information sheet set up previously. If excessive chlorides are suspected, purge the head space in the polyethylene tube for at least 1 minute to get rid of any chlorine gas collected there. After a delay of at least 30 seconds the sample is ready for step "3". NOTE: Purging the samples of chlorine is accomplished by putting a pasteur pipette on the end of some air tubing hooked to a fish pump. The pasteur pipette is then placed at an angle into the top of the polyethylene vessel without breaking the surface of the sample. It takes about one minute to purge the air above the sample of chlorine.

14.3.3 Analysis must be preceded by the analysis of an ICV (concentration at or near mid range) with control limits indicated in table 2.

14.3.4 The ICB must follow the calibration standards with specifications indicated in table 2, but not before the ICV.

14.3.5 Each set of ten samples must be followed by a CCV with specifications indicated in table 2. The run must also end with a CCV, then CCB.

14.3.6 Analyze CCB after calibration and each CCV. The CCB frequency and requirements indicated in table 2.

14.3.7 Instrument Run Log example:

AS LOC	Sample ID
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	SEQ- ICV
10	SEQ-ICB
11	BS
12	BLK
13-22	Samples 1-10
23	SEQ-CCV
24	SEQ-CCB
25-34	Samples 11-20
35	MS
36	MSD
37	SEQ-CCV
38	SEQ-CCB

14.3.8 Sample analysis:

14.3.8.1 Go to "Analyze", "select location" and type in the range of numbers needed to complete analysis. (i.e. 9-54). Press enter and the autosampler will proceed to enter the selected range. NOTE: Check standards are loaded as part of the tray.

14.3.8.2 Make sure that the sample wash beaker is filled with 3% HCl.

14.3.8.3 Dilute and reanalyze samples that are more concentrated than within 10% of the high standard. Soil sample dilutions are made from the digested aliquot. Sample concentration results that are below the calibration curve but above the MDL/DL are reported flagged as estimated, ("B" flag).

14.4 Data reporting

14.4.1 Reduce data to result which will be reported using the soil spreadsheet found on the network.

14.4.2 Complete the data review checklist (attached). Must be completed and attached to each set of DoD data.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 The Excel file for calculations is located at: "V:\Standard Operating Procedures\Current SOP File Directory\Worksheets" and can be accessed through TOC_SOPs_Controlled_Documents spreadsheet at V:\Standard Operating Procedures. Pull up the blank spreadsheet as "read only" and transfer all the information pertinent to the current analysis. Save as the date of analysis with "W" for water and/or "S" for soil at the end (i.e. 071014W for waters). This information can be obtained from your mercury batch sheet.

15.3 Transfer the sample absorbance into the excel spreadsheet in the appropriate cell. The spreadsheet uses the current calibration to calculate the Hg results.

15.4 Make sure that the appropriate dilution factors are entered into the spreadsheet in the correct cells.

15.5 The spreadsheet should divide the result which is the µg Hg obtained from the sample mass by the sample mass in grams. This will yield a result of µg Hg/g sample on a wet weight basis. Calculations in the spreadsheet should be checked each sequence to make sure that they are working correctly.

15.6 If available, divide the result by the %solids to obtain the result on a dry weight basis.

15.7 Report the data as µg Hg/g of sample (mg/kg wet or mg/kg dry when % solids are available).

16.0 Method Performance

Initial Demonstration of Capability (IDOC)/Initial Demonstration of Performance (IDP) : Each analyst must perform an IDOC/IDP prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples prepared as in 10.5.5.1 above. The data is calculated for accuracy and precision requirements. The DOC form, as listed in Quality Control SOP QS03, is completed by each analyst and then provided to the group leader for further processing and approval. See **Table 2** for acceptance criteria.

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Corrective actions and out-of-control data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details corrective actions and out-of-control data.

20.0 Contingencies for Handling out-of-control or unacceptable data

20.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.2 **CORRECTIVE ACTIONS: INSTRUMENT RELATED**

20.2.1 ICV not within table 2 limits

20.2.1.1 If the problem is with the solution, re-prepare, obtain new stock if necessary.
20.2.1.2 If the problem is with the calibration, recalibrate thru analysis of appropriate standards and recheck ICV.

20.2.2 CCV not within table 2 limits

20.2.2.1 If the problem is with the solution, reprepare, obtain new stock if necessary.
20.2.2.2 If the problem is with the calibration, recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.

20.2.2.2.1 If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit may not require reanalysis. See table 2.
20.2.2.2.2 If the CCV was biased low, the previous ten samples must be reanalyzed.

20.3 CORRECTIVE ACTION: DIGESTION RELATED

20.3.1 The preparation blank exceeds limits specified in table 2.

20.3.1.1. If the problem is with the instrument or stannous chloride.

20.3.1.1.1 Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, reprepare the stannous chloride or determine if there are any problems with the instrument.
20.3.1.1.2 If the problem was with the instrument or the stannous chloride and the situation is corrected continue analysis with a second aliquot of the preparation blank.

20.3.1.2 If the problem is with the digestion, all associated samples which meet the criteria specified in table 2 may be acceptable. See table 2 for specifications.
20.3.1.3 If the level of mercury in an associated sample does not meet the criteria specified in table 2, the sample must be redigested/reanalyzed or reported as qualified. The project manager, lab director or data quality manager will make this determination.

20.3.2 BS not within control limits.

20.3.2.1 If the problem is with the instrument, reanalyze when instrument is in control with another aliquot of the sample.
20.3.2.2 If the problem is with the digestion.

20.3.2.2.1 If biased low, associated samples must be redigested.
20.3.2.2.2 If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

20.4 CORRECTIVE ACTION: SAMPLE MATRIX RELATED

20.4.1 Replicate analysis RPD not within table 2 criteria.

20.4.1.1 The associated sample data must be qualified on the final report.

20.4.2 Spike analysis recovery not within table 2 criteria

20.4.2.1 If the analyte level in the sample is greater than 4X the spiking level, the % recovery should not be evaluated and no action is taken.

20.4.2.2If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A non-conformance report must accompany the data and be emailed or given to the group leader.

21.0 Waste Management

21.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

22.0 Equipment/instrument maintenance

22.1 Rotate or change tubing with each analysis. Record in maintenance log.

22.2 Change Millipore –filters- Fluoropore PTFE 1.0 um wh pl 25 mm with each analysis. Record in maintenance log.

22.3 Check line coming from the manifold that contains the above filter and goes to the Hg cell for moisture. Change or dry with nitrogen gas, if needed, before use.

23.0 Computer hardware and software

23.1 Instrument: Perkin Elmer Flow Injection Mercury System FIAS 100

23.2 Software: Winlabs2 for AA version 6.5.0266 @ 2007

24.0 Troubleshooting

24.1 Sample Line has clog and will not pull, or sample waste line will not stay attached due to pressure. Rinse out sample probe with DI and very fine wire. Rinse sample line with DI or 3% Nitric. If this does not fix problem, unhook all tubes from FIAS Valve and disassemble using instructions from manufacturer's Maintenance book located near instrument and computer. Rinse all holes and ports with DI water to check for flow and loosen any clog or determine specifically where the clog is. Reassemble and test flow.

24.1.1 Any other tubing can get a blockage. Replacing is fastest/easiest way to fix. If replace is not an option at the time, soak in DI or 3% nitric to loosen clog. Always rinse out 3% Nitric with DI.

24.2 Mixing blocks have clog/blockage. Rinse with DI or 3% Nitric. If this does not work, soak in DI for a few hours to loosen clog. Can use fine wire to push out clog. If problem still persists, put mixing blocks in sonicator for an hour or so. If problem still persists, throw away blocks and replace with new.

24.3 If pump lines are leaking near pump, turn off pump and check tubes. May have worn a hole. Replace with new tube.

24.4 If a port leaks, tighten the center screw.

25.0 References

25.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 7471A.

25.2 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update IV); Method 7471B

25.3 USEPA Code of Federal Regulations, 40, CH 1, PT 136; Method 245.1; APX-B

25.4 DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003.) Dated 10/25/2010

25.5 Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories. Based on ISO/IEC 17025:2005(E) and The NELAC Institute (TNI) Standards, Volume 1, (September 2009) Dated July 2013

26.0 Tables, Diagrams, Flowcharts and Validation Data

26.1 Table 1, Eppendorf/Standard/Reagent Circle Sheet

26.2 Table 2, Method Quality Control Requirements Summary

26.3 Table 3, Data Reviewers Checklist

Table 1

Mercury: Circle Applicable Digestion/Spike and 0.1mL Eppendorf option then staple to bench sheet

Reminder: Make sure pipettes are verified daily at volume of use for QSM5 batches!

Soil

Amount	Curve Standards 7471 Soil
0.0 mL	
0.05mL	Mercury 1st Source (0.2ug/mL)
0.125mL	Mercury 1st Source (0.2ug/mL)
0.25mL	Mercury 1st Source (0.2ug/mL)
0.50mL	Mercury 1st Source (0.2ug/mL)
1.0mL	Mercury 1st Source (0.2ug/mL)
1.5mL	Mercury 1st Source (0.2ug/mL)
2.5mL	Mercury 1st Source (0.2ug/mL)
Circle	0.1ml Eppendorf 148897Z or 250847
Circle	1.0mL Eppendorf 1854694 or FinnPipette 1FJ61693
	5mL FinnPipette KU04206

Amount	BS/MS/MSD Standards 7471 Soil
0.50mL	Mercury 2nd Source (0.2ug/mL)
Circle	1.0mL Eppendorf 1854694 or FinnPipette 1FJ61693
Amount	**Reagents for Digestion Soil**
2.5mL	Aqua Regia
7.5mL	Potassium Permanganate

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Usability
Initial calibration (ICAL)	• Daily ICAL prior to sample analysis • Low standard at the RL/LOD level • Minimum 5 points and calibration blank	• r-squared $\geq$ 0.990 or r $\geq$ 0.995 • Must follow curve processing requirements from SOP QS08	• Re-run curve • Check instrument for maintenance needs Samples cannot be analyzed until there is a passing calibration
ICV	Alternate source standard to be analyzed after every calibration curve	Must be within $\pm$ 10% for SW846 7471A,±20% for 7471B (±10% for DoD)	• Re-run ICV • Repeat ICAL
CCV	After every 10 field samples and at the end of analysis sequence.	• $\pm$ 20% for SW846-7471A&B • ±10% for DoD QSM 5.0	• Follow guidelines for SOP QS05 • Two passing CCVs analyzed immediately following a failed CCV can be used to report unqualified data.
BLK	One per prep batch	No analytes detected > ½ LOQ or greater that 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	• Re-analysis to confirm the positive value • Notify the PM for further action • Re-prep of samples associated with the BLK • NCR will be required for data reported
BS	One per prep batch	7471A/7471B/DoD QSM4.2 80%-120% DoD QSM5.0 80%-124%	• Re-analyze to confirm failed. • Re-prep and reanalyze BS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. • NCR will be required for data reported • Follow guidelines from SOP QS05
ICB/CCB	Following ICV but before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	• Correct problem. Re-analyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.
MS	One per prep batch, if sample volume available.	7471A/7471B/DoD QSM4.2 80%-120% DoD QSM5.0 80%-124%	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	7471A/7471B/DoD QSM4.2 80%-120% DoD QSM5.0 80%-124% RPD 20%	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Usability**
DOC Study	1. Initially per analyst prior to reporting data 2. Annually per QS08 requirements	• Average percent recovery should be between within BS limits with less than or equal to a 20% standard deviation.	Re-prep and / or re-analysis
MDL Study	At setup and when a significant process change is made.	• See QS09 for calculations/criteria .	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	• Parameter must be detected • The response must be 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	• Bias Requirement at 1-2x the LOQ Recovery within BS limits. • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

Table 3, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	**Sequence ID:**
Calibration:	**NCR#:**
Method: 7471A/7471B (Mercury)	

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard?				
(If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did BS meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation Blank (BLK) below the necessary limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was water bath temperature monitored/documented and did you apply the thermometer correction factor?				
1. Include the water bath Time ON:_____Time OFF:_____from bench sheet.				
11. Sample preparation information is correct and complete.				
12. Analytical results are correct and complete.				
13. The appropriate SOP's have been followed.				
14. "Raw data" including all manual integration's have been correctly interpreted.				
15. "Special" sample preparation and analytical requirements have been met.				
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, non-conformance reports are complete.				
17. Data has been uploaded to the LIMS with correct analyst and analysis factors included.				
18. Reagents/Standards verified accurate on bench sheets and in LIMS.				
19. Calculation is shown on the raw data for sample _____to validate final concentration in LIMS.				

Comments on any "No" response:

Analyst: Date:

Second-Level Review: Date:

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 105 REVISION #: 26 EFFECTIVE DATE: 20161108

METALS
BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY (ICP-AES) TECHNIQUE
SW846 6010C, SW846 6010D, EPA 200.7 Rev. 4.4 and SM 2340 B-2011 for Hardness Calculation

APPROVALS:

Lab Director: _____ Date: **20161108**

Data Quality Manager: _____ Date: **20161108**

Group Leader: _____ interim Date: **20161108**

Changes Summary

SOP105_R26_20161108_ICP

- Updated to lower CAL2 to LOQ or below in listing, table 2 and appended standard information.

SOP105_R25_20160815_ICP

- Updated to include 6010D references/requirements and remove 6010B references.
- Calibration blank requirements updated to indicate <LOD.
- Table 1 updated with headers defined
- MDL/LOD/LOQ references simplified in table 2.
- Updated calibration requirements to include 6010D low-standard and mid-standard read-back and added to checklist.

SOP105_R24_20151221_ICP

- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.

SOP105_R23_20151111_ICP

- Initial EPA method reference updated to include revision number.

SOP105_R22_20150921_ICP

- All references to supervisor updated to reflect group leader.
- Section 10 updated to reference QS04
- Section 24 updated with troubleshooting tips.
- Table of IECs removed as they fluctuate routinely.
- Added immediate analysis of 2 passing CCVs as option in table 2.
- Table 3 removed and Data Reviewer Checklist updated to reflect Table 3.

Revision 21, 20140718

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Section 5.1: When soluble samples are not digested, the blank, BS, MS/MSD and DUP must be filtered through 0.45 micron before analysis.
- Section 10.7.2: CAL1 blank is prepared every 3 months and stored in a teflon bottle, rather than daily.
- Section 10.8.2: RL standard preparation changed
- Section 14.5.1: The CCV for 200.7 was changed from ±5% to ±10%. ICV is ±5% only.
- Section 17.2: Instructions for how to deal with instrument waste were added.
- Section 22: Equipment and instrument maintenance were added.
- Section 23: Computer hardware and software were listed
- Section 24: Trouble shooting was added for the nebulizer and flow through the autosampler.
- Table 1 and Table 2 updated to reflect QSM5.0 criteria in addition to QSM4.2 criteria
- References to Teflon chips replaced with glass beads.

Revision 20, 20130701

- Updated standard methods reference to reflect SM 2340 B-2011 rather than SM 2340B (19th, 20th and 21st Edition).
- Removed CLP references/procedures throughout SOP.

- Standard stock concentrations corrected for Aluminum, Calcium, Iron and Magnesium in section 10.6.3.
- RL standard clarified in section 12.1.5 to indicate also known as LLICV/LLCCV with DoD QSM limits on ICV of ±20% but 6010C limits of ±30%.
- Serial Dilution in section 12.1.9 updated to indicate a 5x dilution instead of 1:4 as in 6010A or 1:5 as in 6010B/6010C.
- Updated section 12.3.2 to be consistent with QC table 2.
- Reference to USACE removed from section 14.2 and 14.4.
- LLICV and LLCCV added to QC table 2.

Revision 19, 20120813
- Software qualifier definitions added Table 1C.
- Analyst review checklist updated
- Table of metals added listing all the metals affected when another metal is present Table 1B.

Revision 18, 20120720
- Archival/PDF access of standard COAs and reagent checks added to section 10.
- Standard expiration dates addressed in section 10.
- Appendix I information added as referenced in section 10.
- Standard methods references added to section 21.
- Cadmium wavelength update in table 1A.
- Table 1 updated to reflect MDL/DL, LOD and LOQ/MRL for all metals currently calibrated.

Revision 17, 20110516
- This is an update of SOP revision 16 dated 4/11/2010.
- Change all limit statements to include "after rounding to the nearest whole number".
- Add procedure for recording digestates filtered prior to analysis within section 14.2.
- Training SOP reference updated to QS03 in section 14.6.
- References to DoD QSM 4.1 have been updated to DoD QSM 4.2.

Revision 16, 04/11/10

- The SOP is an update from Revision 15 dated 05/08/09
- The SOP is formatted to include all 22-elements required per the NELAC standards.
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

METALS
BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY (ICP-AES) TECHNIQUE
SSW846 6010C, SW846 6010D, EPA 200.7 Revision 4.4 and SM 2340 B-2011 for Hardness Calculation

1. Identification of the Test Method

This SOP is compliant with methods –SW846 6010C, SW846 6010D, EPA 200.7 Revision 4.4, and SM 2340 B-2011 for Hardness Calculation.

2. Applicable Matrix or matrices

This SOP is applicable to all matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludge samples, sediments, and other solid wastes, require digestion prior to analysis.

3. Limits of Detection and Quantitation

Detection limits are found in **Table 1** of this SOP. Sensitivity and optimum ranges of the metals may be found in the ICP method file.

4. Scope and Application, Including Parameters to be Analyzed

Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Method Detection Limit and the Reporting Limit (also defined as the Limit of Quantitation and Lowest Limit of Quantitation).

5. Summary of the Test Method

5.1. Prior to analysis, samples must be solubilized or digested using appropriate sample Preparation Methods (e.g., Methods 3005-3050). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis. When dissolved samples are not digested, the blank, BS, MS/MSD/DUP must be filtered before analysis.

NOTE: When selenium is required dissolved samples must always be digested.

5.2. This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving a measurement of the concentration of each element type in the original sample. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analytic wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Control of the spectrometer is provided by PC based *iTEVA* software.

5.3. Inductively Coupled Argon Plasma (ICAP) primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.

5.4. It is standard procedure to use an internal standard (scandium) with samples to increase the stability of the instrument as recommended by the manufacturer (Thermo Fisher). (When samples are suspected of containing scandium, internal standard cannot be used.)

6. Definitions

6.1. Laboratory Quality System QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2. **ICP or ICAP-** Inductively Coupled Plasma or Inductively Coupled Argon Plasma.

6.3. **Inter-element correction (IEC)-** Defined as a correction factor applied by the instrument when there is an overlap of the spectrum from the plasma gases or from another metal into the spectrum of another metal causing that metals concentration to either be inflated or deflated.

7. Interference

7.1. Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high-concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

7.1.1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.

7.1.2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a 200 mg/L or 500 mg/L concentration near the upper analytical range limit.

7.1.3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

7.1.4. When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that Arsenic is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Aluminum. According to Table 2 from the method, 100 mg/L of Aluminum would yield a false signal for Arsenic equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Aluminum would result in a false signal for Arsenic equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The interference effects must be evaluated for each individual instrument since the intensities will vary.

7.1.5. Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.

7.1.6. The interference effects must be evaluated for each individual instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The instrument utilizes a computer routine for automatic correction on all analyses.

7.1.7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

7.1.8. When inter-element corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions (IFA/IFB). If the correction factors or multivariate correction matrices tested

on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at ± one reporting limit from zero, daily verification is not required. All inter-element spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation-change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

7.2. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.

7.3. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized.

7.4. Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. When the instrument displays negative values, dilution of the samples may be necessary.

8. Safety

8.1. Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed lab wide.

8.2. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.

8.3. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.4. MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9. Equipment and Supplies

9.1. Inductively coupled argon plasma emission spectrometer: Thermo Scientific 6500 DUO.

9.2. Computer-controlled emission spectrometer with background correction: Thermo Scientific 6500 DUO or equivalent.

9.3. Radio frequency generator compliant with FCC regulations: Thermo Fisher or equivalent.

9.4. Auto-sampler: Thermo Fisher or equivalent.

9.5. Printer capable of printing results every 4 minutes.

9.6. Cooling Water recycler.

9.7. *Iteva* software.

9.8. Argon gas supply – Liquid Argon

9.9. Class A volumetric flasks (100, 200, 500 and 1000 mL)

9.10. Analytical balance - capable of accurate measurement to a minimum of three significant figures (0.001gm).

9.11. Variable Eppendorf Pipettes 1000µL; 5000µL with disposable tips.
9.12. Disposable beakers 10, 20 and 50 mL size.
9.13. Hood system capable of venting the heat from the system off of the instrument during analysis.

10. Reagents and Standards

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

10.1. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method. All reagents shall be made from ACS reagent grade chemicals. All reagents used for distillation and analysis are entered into Element. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the samples they were used with.

10.2. Note: Unless specifically indicated below or vendor/source standard indicates earlier expiration date, stock standards and reagents are given 12 month expiration dates, digestion spike standards are given 6 month expiration dates and working calibration/IEC standards are given 3 month expiration dates.

10.3. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.

10.4. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL/DL, then the reagent is acceptable. All reagents must be logged into Element and assigned an ID.

10.5. Hydrochloric acid (concentrated), HCl. A method blank is digested and analyzed before a new lot number of HCl is put into use, to ascertain purity. The lot # is logged into Element and the data forwarded to the administrative department with Element ID indicated so PDF can be generated and associated for access within Element and appropriate archival.

10.6. Nitric acid (concentrated), HNO3. A method blank is digested and analyzed before a new lot number of HNO3 is put into use, to ascertain purity. The lot # is logged into Element and the data forwarded to the administrative department with Element ID indicated so PDF can be generated and associated for access within Element and appropriate archival.

10.7. Calibration standards – Purchased standards must be received with a Certificate of Analysis (COA). The standard must be logged into the LIMS to receive an ID which is then written on the COA. The COAs are then forwarded to the administrative department with ID indicated so PDF can be generated and associated for access within Element and appropriate archival.

10.7.1. All standards have an acid matrix of 2% HNO3 and 5% HCl and should be prepared using class A volumetric flasks and calibrated Eppendorfs).

10.7.2. CAL1 is the calibration blank: Reagent grade water matrix matched as in 10.5.1. **Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. When stored in Teflon, this solution is stable for 3 months.**

10.7.3. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.

10.7.4. Stock QC7 solution: Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals- (50 ug/mL)- silver; (100 ug/mL)- aluminum,

boron, barium and sodium; (1000 ug/mL)- potassium; (500 ug/mL or 100 ug/mL note we use two sources of this standard and each have different concentrations for Si) –Silica.

10.7.5. Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.6. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.7.7. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.8. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.7.9. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier. Note: Two sources are needed.

10.7.10. Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.7.11. Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.7.12. Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.7.13. Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element**.**

10.7.14. Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.7.15. Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.16. Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.17. Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.18. Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.19. Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.20. Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.21. Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.22. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.23. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.24. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.25. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.26. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.27. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.28. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.29. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.30. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.31. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.7.32. Stock Scandium solution (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.8. **Calibration and Calibration Verification standards**

10.8.1. The calibration standards and calibration verification standards preparations are recorded in Element. Please find method of preparation in Appendix I. ICV (second source) and CCV standards are prepared daily.

10.8.2. The CRL solution is analyzed to check the accuracy of the instrument at the reporting limit. The stock standard solutions Stock Solution #1 and Stock Solution A are prepared from single element standards listed in 10.5 above. Please find method of preparation in Appendix I. Prepared by adding 1.0 ml of RL Stock solution 1 to deionized water with 2% HNO3 and 5% HCl matrix and diluting to 100 mLs and mix – This is RL STD 1 and 1.0 ml of RL Stock Solution A to de-ionized water with 2% HNO3 and 5% HCL matrix and diluting to 100 mLs, and mix- This is RL STD A.

10.8.3. The interference check standard solutions (IFA and IFB, also known as Spectral Interference Checks) are prepared to provide an adequate test of the IECs. A purchased solution containing 5000 ug/mL Al, Ca, Mg and 2000 ug/mL Fe is diluted 10x to prepare the IFA (Mixed Element SIC). The IFB is prepared by diluting 100x a purchased solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x.

10.9. **Digestion Standards**

10.9.1. The Blank Spike (BS) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B; and 0.50 mLs of the 1000 ug/mL single element standards for Molybdenum, Boron, Titanium and Strontium is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3 and 0.050 mLs of 10000 ug/mL Tin. 25 mL of HCl and 10 mL of HNO3 are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the BS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier within Element. The BS is prepared from a source independent from that used in the calibration standards. 50 mLs of this solution is used for digestion for normal level water samples and the sample is brought back to 50 mLs after digestion. Low level water samples start with two 50 mLs vials with only 1.0 mL of the stock blank spike solution in each taken to 50 mLs. The samples are cooked down to below 25 mLs and combined and then cooked down to below 25 mLs again and then brought back to 25 mLs. This low level BS is given a unique identifier in Element.

10.9.2. The solid BS used with soil samples is prepared by weighing up 1.0 gram of Glass beads for regular level and 2.0 grams of Glass beads for low level and spiking using the same spiking solutions used to spike the sample matrix. This standard is given a unique identifier i.e. Batch #-BS1. Note: Amount of spiking

solution used varies according to whether the samples are being digested for normal level or low level soils. See spiking solutions in 10.7.3.1 for how to prepare the BS for a solid sample, it is prepared the same way that a soil spike is prepared only the known amounts of metals are added to laboratory water.

10.9.3. The spiking solutions are prepared as follows:

10.9.3.1. Stock Multi-element Spiking Solutions: High Purity CLP-CAL-1 solution A: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; solution B: 250 ug/mL Ag; CLP-CAL-2: 1000 ug/L Sb; CLP-CAL-3: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier within Element. Add 0.050 mL for water samples and 0.20 mL for normal level soil samples and 0.10 for low level soil samples of CLP-CAL-1 solutions A and B, and 0.0125 mL for water samples and 0.05 mLs for normal level soil samples and 0.025 mLs for low level soil samples of CLP-CAL-2 and 3 to 50 mL of sample for water samples and 1gram of sample for normal level soils and 2 grams of sample for low level soils for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.

10.9.3.2. TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion: 2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL . Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. This solution is assigned a 3 month expiration date.

10.9.3.3. TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion: 0.50 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store this solution in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 250 ug/L. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. This solution is assigned a 3 month expiration date.

11. Sample Collection, Preservation, Shipment, and Storage

11.1. Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been pre-filtered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).

11.3. Sample digestates are stored at room temperature for at least 3 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. All metal samples are neutralized before disposal in the receiving section of the laboratory.

11.4. **The appropriate SOPs should be consulted regarding sample preparation.** The following is a brief summary of the methods we use for metals preparation.

11.4.1. Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and HNO3 prior to metal determination.

11.4.2. Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.

11.4.3. Standard Methods Method 3030C prepares ground-waters and surface water samples for acid extractable metals: (lead and chromium.) This preparation has a holding time of 72 hours. The samples are preserved at collection with 5mL/L of HNO3, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. Once cooled, the sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.

11.4.4. Method 3050B prepares wastes samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

12. Quality Control

Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.1. Daily run and batch QC – See table 2 for criteria

12.1.1. Calibration is required daily. Either a blank and a high standard or a client specific three standard concentration points and a blank calibration is required daily.

12.1.2. Low-level and Mid-level standards are to be back calculated to the curve for validation (6010D).

12.1.3. IEC correction standards for aluminum and iron are required daily.

12.1.4. ICV – second source following calibration.

12.1.5. The ICB/CCB must immediately follow the ICV/CCV.

12.1.6. RL standard - also known as low-level ICV or CCV (LLICV or LLCCV)

12.1.7. IFA (Mixed element SIC)/IFB analyzed daily.

12.1.8. CCV must be analyzed every ten field samples and at the end of the analysis.

12.1.9. CCB must be analyzed every ten samples immediately following the CCV or at the end of the analysis

12.1.10. *The following should be analyzed with each preparation batch containing a matrix spike.*

12.1.10.1. Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 5x dilution (volumetric glassware must be used). If not within criteria, a chemical or physical interference effect should be suspected. The analyst and or group leader must note this situation on the final analytical report.

12.1.10.2. Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, and is required especially if the pre-digestion matrix spike is outside of control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. Run all associated samples in the preparatory batch by method of standard additions (MSA) or apply "J" qualifier. The analyst and or group leader must note this situation on the final analytical report.

12.2. Quarterly and/or every six months – See table 2 for criteria

12.2.1. Linear range standards must be analyzed at a frequency no less than once every six months. The linear range standard is required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. This standard can be analyzed as the linear dynamic range.

12.2.2. The inter-element correction factors (IEC) should be verified at the time the linear range standards are analyzed or whenever there is any question about whether an IEC is correcting correctly.

12.2.3. MDL studies are completed at setup and when a significant process change is made. Detection limits are set based upon MDL studies and validated with quarterly LOD/LOQ verifications.

12.3. **Digested Batch QC – See table 2 for criteria**

12.3.1. All quality control data should be maintained and available for easy reference or inspection.

12.3.2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank (BLK), sometimes referred to as the preparation blank, is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. If criteria are exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The extracted blank associated with TCLP batches must be less than 100 X the regulatory limit for barium.

12.3.3. Employ a minimum of one blank spike (BS) for aqueous samples or one glass bead spiked sample per sample batch to verify the digestion procedure. These blank spikes are taken through the same digestion/preparation steps as the sample being tested. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be re-digested. Consult your group leader for further action. Qualifying the associated data may not be permissible for some clients.

12.4. **Sample – See table 2 for criteria**

12.4.1. Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. Group leader must be notified if the control limit is not met. Group leader will dictate corrective action if required. The final analytical report must document this situation.

12.4.2. Analyze a minimum of one spiked sample and/or spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. Project specific requirements will take precedence in determining whether a matrix spike duplicate is employed in these situations.

13. Calibration and Standardization

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

13.2. Operating conditions - **The instrument settings can be found in method file within the iTEVA software.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

13.3. Auto-peak when some change has been made to the introductory system and calibrate the instrument according to the instrument manufacturers recommended procedures, using the specified calibration standard solutions.

Flush the system with 2% HNO_3 / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ($r \geq 0.998$). If a three point calibration curve is not required for the client samples being analyzed by Empirical Laboratories may use a blank and one standard as referenced in the method.

13.4. Before beginning the sample run, analyze single element Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs using the iTEVA software. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV ± 10% (for 200.7 - ± 5%) and ICB (< ±2xMDL/DL, <±LOD-DOD or ±RL/CRDL for others, first, then analyze a reporting limit standard (a standard at the concentration of the reporting limit). This standard must be within ±20% for DOD projects and within ±30% for samples analyzed for 6010C. Note: Group leader must be notified if the control limit is not met. Group leader will dictate corrective action if required. The final analytical report must document this situation.

13.5. Verify the inter-element and background correction factors at the beginning of the sequence in the specific order of IFA, IFB, CCV and CCB (IFA criteria: non-spiked analytes < ±2xMDL or <±LOD for DOD beginning of sequence**.** Do this by analyzing the interference check solution IFA and IFB. Absolute value of concentration for all non-spiked analytes in the IFA must be <LOD (unless they are verified trace impurity from one of the spiked analytes) for DOD. Results must be within ±20% of the true value for IFB. If corrective action fails, apply Q-qualifier to all results for specific analyte(s) in all samples associated with the ICS.

Note: Group leader must be notified if the control limit is not met. Group leader will dictate corrective action if required. The final analytical report must document this situation.

13.6. The instrument must be calibrated once every 24 hours.

13.7. Instrument Autosampler Report example:

Calibration Rack (used by instrument software to insert QC)

1) Cal Std 1 (blank)
2) Cal Std 2 (Low Cal – at or below the LOQ)
3) Cal Std 3 (Mid Cal)
4) Cal Std 4 (Ba @ 5000 ppb, Ag @ 2000 ppb, Mn @ 10,000 ppb)
5) Cal Std 5 (QC5)
6) Cal Std 6 (QC 21)
7) Cal Std 7 (NAK 100)
8) Cal Std 8 (QC3)
9) Al IEC-(correction using ITEVA software)
10) Fe IEC-(correction using ITEVA software)

Sample Sequence RACK 1

1) SEQ-ICV
2) SEQ-ICB
3) SEQ-CRL1-reporting limit standard 1
4) SEQ-CRL2-reporting limit standard 2

5) Rinse
6) SEQ-IFA1
7) SEQ-IFB1
8) Rinse
9) SEQ-CCV
10) SEQ-CCB
11) Method Blank (*Batch #* -BLK1)
12) Blank Spike (*Batch #* -BS1)
13) Sample 1
14) Sample 2
15) Sample 3
16) Sample 4
17) Sample 5
18) Sample 6
19) Sample 7
20) Sample 8
21) Sample 9
22) Sample 10
23) SEQ-CCV
24) SEQ-CCB
25) Sample 11
26) Sample 12
27) Sample 13
28) Sample 14
29) Sample 15
30) Sample 16
31) Sample 17
32) Sample 18
33) Sample 19
34) Sample 20
35) Sample matrix spike (*batch#*- MS1)
36) Sample matrix spike duplicate (*batch#* -MSD1)
37) Sample post digestion spike (*batch#* -PS1)
38) Sample serial dilution (*batch#* -DUP1)
39) SEQ-CCV
40) SEQ-CCB
41) Preparation Blank (*batch#* -BLK1)
42) Blank Spike (*batch#* -BS1)
43) Sample 1
44) Sample 2
45) Sample 3
46) Sample 4
47) Sample 5
48) Sample 6
49) Sample 7
50) Sample 8

51) Sample 9
52) Sample10
53) SEQ-CCV
54) SEQ-CCB
55) Sample 11

RACK 2

1) Sample 12
2) Sample 13

Etcetera…

Each rack holds 60 samples and there are 4 racks that are used for samples, CCVs and CCBs and run QC.

14. Procedure

14.1. Once the instrument has been calibrated, begin the analysis of samples.

14.2. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. The sample requiring filtration must be recorded on the bench sheet and added to the bench sheet comments in the LIMS.

14.3. Flush the system with 2% HNO_3 / 5% HCl for at least 1 minute before the analysis of each sample.

14.4. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7, $\pm$ 10% of the linear range standard.

14.5. Verify calibration every 10 field samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

14.5.1. The results of the CCV are to agree within ±10% for 6010 and 200.7. If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run with the understanding that, after conferring with the group leader, it may be necessary to reanalyze a group of samples. The analyst must notify the group leader within 24 hours and prepare a non-conformance report as indicated.

14.5.2. The results of the calibration blank (this is not the method/preparation blank) are to be < 2x ±MDL, for **DOD no analytes detected >LOD.** If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank < 1/10 the concentration of the action level of interest and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).

15. Data Analysis and Calculations

15.1. Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2. Total hardness is reported from HNO_3 preserved sample. The final concentration is calculated from the calcium and magnesium results as follows: Ca mg/L x 2.5 + Mg mg/L x 4.1 = total Hardness in mg/L as $CaCO_3$.

15.3. The instrument will generate data results in mg/L or µg/L (labeled appropriately). Each result represents an average of three individual readings per metal channel.

15.4. For aqueous samples, if a post/pre-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.

15.5. For solid samples, if a post-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

SR (ug/g or mg/kg) = IR*DF*FED/SM

SR	=	Sample result
IR	=	Instrument result (µg/L)
DF	=	Dilution factor (post digestion)
FED	=	Final volume of digestate (L)
SM	=	Sample mass digested (g)

16. Method Performance

Initial Demonstration of Capability (DOC)/Performance (IDP): Each analyst must perform an IDOC/IDP prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples (prepared from same source as calibration). The data is calculated for accuracy and precision requirements. The IDOC form is completed by each analyst and then provided to the group leader for further processing and approval. See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

16.1 Method Detection Limit Study or Detection Limit Determination
16.2 Limit of Detection Verification
16.3 Limit of Quantitation, Lower Limit of Quantitation or Reporting Limit Verification
16.4 Initial Demonstration of Capability (IDOC)/Performance (IDP)
16.5 PT Studies

17. Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria. Table 2 of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Corrective Actions and Out-of-Control Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

19.1. Instrument Related
19.1.1. ICV
19.1.1.1. Is the problem with the solution?
19.1.1.1.1. Re-prepare or obtain new stock.

19.1.1.2. Is the problem with the calibration?

19.1.1.2.1. Recalibrate through analysis of appropriate standards and recheck ICV.

19.1.2. ICB

19.1.2.1. Is the problem with the solution?

19.1.2.1.1. Re-prepare.

19.1.2.2. Is the problem with the calibration?

19.1.2.2.1. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.

19.1.3. Check Standards

19.1.3.1. Is the problem with the solution?

19.1.3.1.1. Re-pour, re-prepare or obtain new stock.

19.1.3.2. Is the problem with the calibration?

19.1.3.2.1. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.4. IFA

19.1.4.1. Is the problem with the solution?

19.1.4.1.1. Re-prepare or obtain new stock.

19.1.4.2. Is the problem with the calibration?

19.1.4.2.1. Recalibrate through analysis of appropriate standards. Restart analysis with the ICV.

19.1.5. IFB

19.1.5.1. Is the problem with the solution?

19.1.5.1.1. Re-prepare or obtain new stock.

19.1.5.2. Is the problem with the calibration?

19.1.5.2.1. Recalibrate through analysis of appropriate standards. Restart analysis with the ICV.

19.1.6. CCV

19.1.6.1. Is the problem with the solution?

19.1.6.1.1. Re-prepare or obtain new stock.

19.1.6.2. Is the problem with the calibration?

19.1.6.2.1. If appropriate, continue the analysis. Discuss effect of the out of control situation with your group leader. The samples will be reanalyzed or the data will be qualified.

19.1.7. CCB

19.1.7.1. Is the problem with the solution?

19.1.7.1.1. Re-prepare

19.1.7.2. Is the problem with the calibration?

19.1.7.2.1. Re-calibrate and reanalyze.

19.2. **Digestion Related**

19.2.1. Preparation blank (BLK)

19.2.1.1. Is the problem with the instrument?

19.2.1.1.1. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.

19.2.1.2. Is the problem with the digestion?

19.2.1.2.1. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be re-digested or the data must be qualified on the final report.

19.2.2. **BS**

19.2.2.1. Is the problem with the instrument?

19.2.2.1.1. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.

19.2.2.2. Is the problem with the digestion?

19.2.2.2.1. If biased low, associated samples must be re-digested.

19.2.2.2.2. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

19.3. **Sample Matrix Related**

19.3.1. Replicate analysis RPD

19.3.1.1. The associated sample data must be qualified on the final report.

19.3.2. Spike analysis recovery

19.3.2.1. Is the analyte level in the sample greater than 4X the spiking level?

19.3.2.1.1. If yes, the spike recovery is not evaluated.

19.3.2.1.2. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.

19.3.3. Post digestion spike analysis recovery, when required

19.3.3.1. The associated sample data must be qualified on the final report.

19.3.3.2. Analysis by MSA may be requested by the client.

19.3.4. Serial dilution analysis

19.3.4.1. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?

19.3.4.1.1. If no, the serial dilution data cannot be evaluated.

19.3.4.1.2. If yes, a chemical or physical interference effect should be suspected. The analyst and or group leader must note this situation on the final analytical report.

20. Contingencies for Handling Out-of-Control or Unacceptable Data

20.1. Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

21. Waste Management

21.1. Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.2. All instrument waste is properly labeled as "Metals Analytical Effluent" and is stored in the appropriate area for neutralization and disposal by receiving personnel.

22. Equipment / Instrument Maintenance

22.1. Periodic cleaning of the introductory system, this includes torch, parascope cup, nebulizer and spray chamber.

22.2. New pump tubing on the peristaltic pump is replaced twice a week.

22.3. The water chiller fluid is replaced during the preventive maintenance scheduling.

22.4. The dust cover on the water chiller is vacuumed weekly.

23. Computer Hardware and Software

23.1. Computer hardware required 20Gb Hard drive, 512 MB Ram, dedicated Ethernet card and printer required.

23.2. Computer Software Thermo Iteva Version 2.8.0.96 copyright 2013, must have operating system of Windows XP.

24. Troubleshooting

24.1. Watch for nebulizer pressure to climb, it normally stays about 290 kPa but samples that contain high salts and turbidity or viscosity will clog the nebulizer and cause the pressure to climb to 340 kPa. When this occurs it results in QC failure. Clean the Nebulizer or replace it to remedy the problem.

24.2. Monitor the flow up through the sample lines and pump tubing to make sure that are not air bubbles and that the flow is smooth. New pump tubing can eliminate those problems.

25. References

25.1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update IV)*; Method 6010C.

25.2. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update V)*; Method 6010D.

25.3. USEPA *Code of Federal Requlations, 40, CH 1,PT 136;* Method 200.7 Revision 4.4; APX-B.

25.4. DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003. 10/25/2010

25.5. DOD Quality Systems Manual for Environmental Laboratories version 5.0, 7/2013. [Based on ISO/IEC 17025:2005(E) and the NELAC Institute (TNI) Standards, Volume 1, September 2009)]

25.6. Standard Methods – 22nd Edition.

25.7. ICAP 6000 Series ICP-OES operator manual.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Table 1 DL, LOD, LOQ and recovery limits

26.2. Table 1A, contains a list of the wavelengths used for each analyte.

26.3. Table 1B, Instrument Software Flags

26.4. Table 2, QA/QC summary table.

26.5. Table 3, Data Reviewers Checklist

26.6. Appendix I, Calibration Standard preparation

Table 1 - DL, LOD, LOQ and Recovery Limits

LL= Low Limit, UL = Upper Limit, LL5/UL5 are limits from QSM5

S/W	Method	Analyte	DL	LOD	LOQ	Units	LL	UL	LL5	UL5
Solid	6010C/D	Aluminum	10.0	20.0	40.0	mg/Kg	80	120	74	119
Solid	6010C/D	Antimony	1.00	1.60	2.00	mg/Kg	80	120	79	114
Solid	6010C/D	Arsenic	0.600	1.20	2.00	mg/Kg	80	120	82	111
Solid	6010C/D	Barium	1.00	4.00	8.00	mg/Kg	80	120	83	113
Solid	6010C/D	Beryllium	0.200	0.400	1.00	mg/Kg	80	120	83	113
Solid	6010C/D	Boron	2.00	4.00	8.00	mg/Kg	80	120	72	114
Solid	6010C/D	Cadmium	0.200	0.400	1.00	mg/Kg	80	120	82	113
Solid	6010C/D	Calcium	200	400	1000	mg/Kg	80	120	81	116
Solid	6010C/D	Chromium, total	0.400	0.800	2.00	mg/Kg	80	120	85	113
Solid	6010C/D	Cobalt	1.00	2.00	2.50	mg/Kg	80	120	85	112
Solid	6010C/D	Copper	0.800	1.60	2.00	mg/Kg	80	120	81	117
Solid	6010C/D	Iron	6.00	12.0	20.0	mg/Kg	80	120	81	118
Solid	6010C/D	Lead	0.300	0.600	1.00	mg/Kg	80	120	81	112
Solid	6010C/D	Magnesium	200	600	1000	mg/Kg	80	120	78	115
Solid	6010C/D	Manganese	0.600	1.20	3.00	mg/Kg	80	120	84	114
Solid	6010C/D	Molybdenum	1.00	2.00	4.00	mg/Kg	80	120	82	116
Solid	6010C/D	Nickel	0.600	1.20	2.00	mg/Kg	80	120	83	113
Solid	6010C/D	Potassium	200	600	1000	mg/Kg	80	120	81	116
Solid	6010C/D	Selenium	0.600	1.00	2.00	mg/Kg	80	120	78	111
Solid	6010C/D	Silver	0.200	0.400	2.00	mg/Kg	80	120	82	112
Solid	6010C/D	Sodium	200	600	1000	mg/Kg	80	120	83	118
Solid	6010C/D	Strontium	0.300	0.600	1.20	mg/Kg	80	120	83	114
Solid	6010C/D	Thallium	0.600	0.800	1.60	mg/Kg	80	120	83	111
Solid	6010C/D	Tin	2.00	10	40	mg/Kg	80	120	80	120
Solid	6010C/D	Titanium	1.00	2.00	4.00	mg/Kg	80	120	83	114
Solid	6010C/D	Vanadium	1.00	2.00	2.50	mg/Kg	80	120	82	114
Solid	6010C/D	Zinc	1.00	2.00	4.00	mg/Kg	80	120	82	113
Water	6010C/D	Aluminum	50.0	100	200	ug/L	80	120	86	115
Water	6010C/D	Antimony	5.00	8.00	10	ug/L	80	120	88	113
Water	6010C/D	Arsenic	3.00	6.00	10.0	ug/L	80	120	87	113
Water	6010C/D	Barium	5.00	10.0	40.0	ug/L	80	120	88	113
Water	6010C/D	Beryllium	1.00	2.00	5.00	ug/L	80	120	89	112
Water	6010C/D	Boron	10.0	20.0	40	ug/L	80	120	85	113
Water	6010C/D	Cadmium	1.00	2.00	5.00	ug/L	80	120	88	113
Water	6010C/D	Calcium	1000	2000	5000	ug/L	80	120	87	113
Water	6010C/D	Chromium, total	2.00	4.00	10.0	ug/L	80	120	90	113
Water	6010C/D	Cobalt	5.00	10.0	12.5	ug/L	80	120	89	114
Water	6010C/D	Copper	4.00	8.00	10.0	ug/L	80	120	86	114
Water	6010C/D	Iron	30.0	60.0	100	ug/L	80	120	87	115
Water	6010C/D	Lead	1.50	3.00	5.00	ug/L	80	120	86	113
Water	6010C/D	Magnesium	1000	3000	5000	ug/L	80	120	85	113
Water	6010C/D	Manganese	3.00	6.00	15.0	ug/L	80	120	90	114
Water	6010C/D	Molybdenum	5.00	10.0	20	ug/L	80	120	89	113
Water	6010C/D	Nickel	3.00	6.00	10.0	ug/L	80	120	88	113
Water	6010C/D	Potassium	1000	3000	5000	ug/L	80	120	86	114
Water	6010C/D	Selenium	3.00	5.00	10	ug/L	80	120	83	114
Water	6010C/D	Silver	1.00	2.00	10.0	ug/L	80	120	84	115
Water	6010C/D	Sodium	1000	3000	5000	ug/L	80	120	87	115
Water	6010C/D	Strontium	1.50	3.00	6.00	ug/L	80	120	90	113
Water	6010C/D	Thallium	3.00	4.00	8.00	ug/L	80	120	85	114

S/W	Method	Analyte	DL	LOD	LOQ	Units	LL	UL	LL5	UL5
Water	6010C/D	Tin	10.0	20.0	30.0	ug/L	80	120	88	115
Water	6010C/D	Titanium	5.00	10.0	20	ug/L	80	120	91	111
Water	6010C/D	Vanadium	5.00	10.0	12.5	ug/L	80	120	90	111
Water	6010C/D	Zinc	5.00	10.0	20.0	ug/L	80	120	87	115
Solid	6010C/D low	Aluminum	2.5	5	10	mg/Kg	80	120	74	119
Solid	6010C/D low	Antimony	0.25	0.4	0.50	mg/Kg	80	120	79	114
Solid	6010C/D low	Arsenic	0.15	0.3	0.5	mg/Kg	80	120	82	111
Solid	6010C/D low	Barium	0.25	0.5	2	mg/Kg	80	120	83	113
Solid	6010C/D low	Beryllium	0.05	0.1	0.25	mg/Kg	80	120	83	113
Solid	6010C/D low	Boron	0.5	1	2	mg/Kg	80	120	72	114
Solid	6010C/D low	Cadmium	0.05	0.1	0.25	mg/Kg	80	120	82	113
Solid	6010C/D low	Calcium	50	100	250	mg/Kg	80	120	81	116
Solid	6010C/D low	Chromium, total	0.1	0.2	0.5	mg/Kg	80	120	85	113
Solid	6010C/D low	Cobalt	0.25	0.5	0.625	mg/Kg	80	120	85	112
Solid	6010C/D low	Copper	0.2	0.4	0.5	mg/Kg	80	120	81	117
Solid	6010C/D low	Iron	1.5	3	7.5	mg/Kg	80	120	81	118
Solid	6010C/D low	Lead	0.075	0.15	0.25	mg/Kg	80	120	81	112
Solid	6010C/D low	Magnesium	50	150	250	mg/Kg	80	120	78	115
Solid	6010C/D low	Manganese	0.15	0.3	0.75	mg/Kg	80	120	84	114
Solid	6010C/D low	Molybdenum	0.25	0.5	1	mg/Kg	80	120	82	116
Solid	6010C/D low	Nickel	0.15	0.3	0.5	mg/Kg	80	120	83	113
Solid	6010C/D low	Potassium	50	150	250	mg/Kg	80	120	81	116
Solid	6010C/D low	Selenium	0.15	0.25	0.5	mg/Kg	80	120	78	111
Solid	6010C/D low	Silver	0.05	0.1	0.5	mg/Kg	80	120	82	112
Solid	6010C/D low	Sodium	50	150	250	mg/Kg	80	120	83	118
Solid	6010C/D low	Strontium	0.075	0.15	0.3	mg/Kg	80	120	83	114
Solid	6010C/D low	Thallium	0.15	0.2	0.4	mg/Kg	80	120	83	111
Solid	6010C/D low	Tin	0.5	2.5	10	mg/Kg	80	120	80	120
Solid	6010C/D low	Titanium	0.25	0.5	1	mg/Kg	80	120	83	114
Solid	6010C/D low	Vanadium	0.25	0.5	0.625	mg/Kg	80	120	82	114
Solid	6010C/D low	Zinc	0.25	0.5	1	mg/Kg	80	120	82	113
Water	6010C/D low	Aluminum	12.5	25	50	ug/L	80	120	86	115
Water	6010C/D low	Antimony	1.25	2	2.5	ug/L	80	120	88	113
Water	6010C/D low	Arsenic	0.75	1.5	2.5	ug/L	80	120	87	113
Water	6010C/D low	Barium	1.25	2.5	10	ug/L	80	120	88	113
Water	6010C/D low	Beryllium	0.25	0.5	1.25	ug/L	80	120	89	112
Water	6010C/D low	Boron	2.5	5	10	ug/L	80	120	85	113
Water	6010C/D low	Cadmium	0.25	0.5	1.25	ug/L	80	120	88	113
Water	6010C/D low	Calcium	250	500	1250	ug/L	80	120	87	113
Water	6010C/D low	Chromium, total	0.5	1	2.5	ug/L	80	120	90	113
Water	6010C/D low	Cobalt	1.25	2.5	3.125	ug/L	80	120	89	114
Water	6010C/D low	Copper	1	2	2.5	ug/L	80	120	86	114
Water	6010C/D low	Iron	7.5	15	25	ug/L	80	120	87	115
Water	6010C/D low	Lead	0.375	0.75	1.25	ug/L	80	120	86	113
Water	6010C/D low	Magnesium	250	750	1250	ug/L	80	120	85	113
Water	6010C/D low	Manganese	0.75	1.5	3.75	ug/L	80	120	90	114
Water	6010C/D low	Molybdenum	1.25	2.5	5.0	ug/L	80	120	89	113
Water	6010C/D low	Nickel	0.75	1.5	2.5	ug/L	80	120	88	113
Water	6010C/D low	Potassium	250	750	1250	ug/L	80	120	86	114
Water	6010C/D low	Selenium	0.75	1.25	2.5	ug/L	80	120	83	114
Water	6010C/D low	Silver	0.25	0.5	2.5	ug/L	80	120	84	115
Water	6010C/D low	Sodium	250	750	1250	ug/L	80	120	87	115

S/W	Method	Analyte	DL	LOD	LOQ	Units	LL	UL	LL5	UL5
Water	6010C/D low	Strontium	0.375	0.75	1.5	ug/L	80	120	90	113
Water	6010C/D low	Thallium	0.75	1	2.0	ug/L	80	120	85	114
Water	6010C/D low	Tin	2.5	5	7.5	ug/L	80	120	88	115
Water	6010C/D low	Titanium	1.25	2.5	5	ug/L	80	120	91	111
Water	6010C/D low	Vanadium	1.25	2.5	3.125	ug/L	80	120	90	111
Water	6010C/D low	Zinc	1.25	2.5	5.0	ug/L	80	120	87	115
TCLP	SW1311_6010C/D	Arsenic	0.03	0.06	0.1	mg/L	80	120	87	113
TCLP	SW1311_6010C/D	Barium	0.05	0.1	0.4	mg/L	80	120	88	113
TCLP	SW1311_6010C/D	Cadmium	0.01	0.02	0.05	mg/L	80	120	88	113
TCLP	SW1311_6010C/D	Chromium	0.02	0.04	0.1	mg/L	80	120	90	113
TCLP	SW1311_6010C/D	Lead	0.015	0.03	0.05	mg/L	80	120	86	113
TCLP	SW1311_6010C/D	Selenium	0.030	0.05	0.1	mg/L	80	120	83	114
TCLP	SW1311_6010C/D	Silver	0.01	0.02	0.1	mg/L	80	120	84	115

LL= Low Limit, UL = Upper Limit, LL5/UL5 are limits from QSM5

TABLE 1A

METAL	WAVELENGTH
Aluminum	396.1
Antimony	206.8
Arsenic	189.0
Barium	233.5
Beryllium	313.0
Boron	249.7
Cadmium	214.4
Calcium	317.9
Chromium	267.7
Cobalt	228.6
Copper	324.7
Iron	261.1
Lead	220.3
Magnesium	279.0
Manganese	257.6
Molybdenum	202.0
Nickel	231.6
Potassium	766.4
Selenium	196.0
Silver	328.0
Sodium	589.5
Strontium	421.5
Thallium	190.8
Tin	189.9
Titanium	334.9
Vanadium	292.4
Zinc	206.2

Table 1B – IEC listing indicating metals affected when a certain metal is present.

6500 Duo IECs
Metals Affected

Ag	_____
Al	_____ As, Pb, Sb, Se, Tl, Zn
As	_____ Cd
B	_____
Ba	_____
Be	_____ Zn
Ca	_____ Al, Pb, Ti, Zn, Ni, Se, Sr
Cd	_____ Cr
Co	_____ Cd, Pb, Tl
Cr	_____ As, Ba, Fe, Ni, Sb, Ti, Tl, V, Zn
Cu	_____ Pb, Zn
Fe	_____ Ag, Al, As, B, Ba, Cd, Co, Cr, Cu, Mg, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn
K	_____
Mg	_____ Cd, Cr, Pb, Ti, Zn
Mn	_____ Ag, Cr, Fe, Pb, Se, Tl, V
Mo	_____ Ag, Al, As, Be, Cd, Co, Cu, Mg, Ni, Pb, Sb, Se, Tl, V, Zn
Na	_____
Ni	_____ Cd, Co, Pb, Se
Pb	_____
Sb	_____ Ni, Zn
Se	_____ Zn
Sn	_____
Ti	_____ As, Be, Co, Cu, Pb, Sb, Se, Tl, V
Tl	_____ Ni
V	_____ Ag, Ba, Be, Cd, Cr, Cu, Mo, Pb, Sb, Se, Tl, Zn
Zn	_____ Ni, As

Table 1B – Instrument Software Flags

iTEVA Analysis Flags

The following errors may be encountered during analysis:

Error Flag	Reason
F	The check has failed the limits set in the Check Table.
W	The warning limit set in the Check Table has been reached/exceeded.
>	The result is above the print limits set for the method.
<	The result is below the print limits set for the method.
? value	There is a calculation error associated with the result (could be due to an invalid calibration, IEC etc.).
C	The concentration is beyond the calibrated range (past inflection point) .
c	The concentration yields negative value (below blank standard) .
z	The data does not have a valid calibration associated with it (this will happen if samples are run before a method is calibrated, unless the Output Mode is set to an option other than Concentration in Method/ Report Preferences/Output Mode).
k	There is an error on an interferent line. This error is also reported if the IEC threshold has been exceeded. See *Method/Element/General/IEC/Threshold.*
i	Interferent (overlapping Subarrays) - data can be used. This error is due to choosing two or more analytical lines that overlap with each other spectrally. Ideally, an alternate line should be chosen see *Method/New* and refer to the Interference Tables, or you can choose to apply an IEC correction to overcome the interference. See *Method/Elements/IEC.*
I	Interferent (overlapping subarrays) - data cannot be used as the peaks cannot be resolved.
D	The analyte peak is off the CID detector.
^	The peak is saturated (data not returned).
P	Plasma error, the plasma was extinguished during analysis.
*	Global failure - NO DATA ACQUIRED. May be due to a memory error due to too many data points.
s	Error on internal standard line.
Z	Internal standard error, normally due to an invalid calibration or incorrectly set up internal standard reference. This flag is also displayed if an analysis is run without a valid calibration.
N	The wavelength was de-selected before exposure, no data returned.
R	The wavelength failed intelligent rinse test (i.e. the maximum no. of attempts was exceeded).

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Usability**
Interference Check	once per calibration	IFA less than LOD if not verified contamination of standard. IFB must be within ±20%.	• Check IEC corrections for metals in the IFA.
Calibration Curve	• Prior to analyzing any samples • A minimum of a blank and 3-points for linear fits, if client specific requirement, or a blank and high standard. • **If blank and 3-points, lowest standard must be at or below the LOQ.**	• Linear calibration Corr. of 0.998 • DOD r-squared ≥ 0.990 • Must follow curve processing requirements from SOP QS08 • Low-Level standard calculated back to the curve should be within +/-20% • Mid-Level standard calculated back to the curve should be within +/-10%	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICB/CCB	• At the beginning of every sequence • For every 10-field samples • At the end of every sequence	Must meet the <LOD for DOD or < 2xMDL/DL	Re-run
ICV	Alternate source standard to be analyzed after every calibration curve	Must be in the range 90 to 110% for 6010C/D/DoD, or 95 to 105% for 200.7.	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
LLICV	Low standard at the LOQ/RL level run against the curve.	Within 20% DoD, 30% for 6010C/D to report at this LOQ/RL.	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources • Raise LOQ/RL to next passing level.
CCV	• At the beginning of every sequence • For every 10-field samples • At the end of every sequence	Must be in the range 90 to 110%	• Samples must be reanalyzed, if possible, if not samples are qualified with a "X" (high) or "Y" (low). • Two passing CCVs analyzed immediately following a failed CCV can be used to report unqualified data.
Closing LLCCV	At the end of every 6010C/D sequence	Should be in the range 70 to 130% for 6010C/D	NA

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Usability**
BLK	One per prep batch	≤±2xMDL for non-DoD ≤± ½ LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater, for DoD	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data qualification will be required
BS	One per prep batch	Must be in the range of 80 to 120% for 6010C/D/DOD QSM4.2; or 85 to 115% for 200.7; or specified limits for DoD QSM5.0.	• Rerun to confirm problem. • All samples associated with the LCS must be re-digested, reanalyzed if possible. • NCR will be required for data reported • If samples cannot be re-digested or re-analyzed Final Report data qualification will be required
MS	One per prep batch	Must be in the range of 80 to 120% for 6010C/D/DOD QSM4.2/200.7; or specified limits for DoD QSM5.0.	Final Report data qualification will be required
MSD	One per prep batch	Must be in the range of 80 to 120% for 6010C/D/DOD QSM4.2/200.7; or specified limits for DoD QSM5.0. RPD ≤ 20%	Final Report data qualification will be required
Sample Duplicate	One per prep batch	RPD ≤ 20%	Qualify samples
Dilution test (Only applicable for samples with concentrations > 50 x LOQ/MRL)	One per prep batch	Five-fold dilution must agree within ± 10% of the original measurement.	Perform post-digestion spike (PDS) addition;
Post Digestion Spike	One per prep batch (When dilution test fails or analyte concentration in all samples < 50 x LOD.)	±25% for DOD QSM 4.2, ±20% for DoD QSM 5.0/6010C/D	For the specific analyte(s) in the parent sample, apply J-qualify if acceptance criteria are not met.

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Usability**
IDOC/IDP Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from QS08 for the preparation and analysis of DOC samples	• Must meet the criteria of the BS for average accuracy	• Re-prep and / or • Re-analysis
MDL determination	Initial demonstration with quarterly verification from LOD, LOQ. May be required annually for specific certifications.	Refer to SOP QS09.	
LOD determination and verification	Prior to initial analysis with quarterly verification.	Refer to SOP QS08.	
LOQ establishment and verification	Prior to initial analysis with quarterly verification.	Refer to SOP QS08.	
Linear Dynamic Range Study (LDR) or verification	Study at initial setup with verification every six months	Within ± 10% of true value.	

Table 3

ANALYST DATA REVIEW CHECKLIST Sample Number(s):				
Batch Number(s):		**Sequence ID:**		
Method:	**6010C or 6010D (ICP)**			

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within holding times?				
2.a Were initial calibration curve QC criteria metwith low standard <=LOQ?				
2b Did 6010D low-std readback meet 80%-120%? Mid-std 90%-110%? (should) List and discuss with management prior to use.				
3. Were all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard?				
(If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did BS (blank spike) meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank (BLK) below the necessary limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor?				
10. Sample preparation information is correct and complete.				
11. Analytical results are correct and complete.				
12. The appropriate SOP's have been followed.				
14. "Raw data" including all manual integration's have been correctly interpreted.				
15. "Special" sample preparation and analytical requirements have been met and documented.				
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, non-conformance reports are complete.				
17. Data has been uploaded to the LIMS with correct analyst and analysis factors included.				
18. Reagents/Standards verified accurate on bench sheets and in LIMS.				
19. Calculation is shown on the raw data for sample _____ to validate final concentration in LIMS.				

Comments on any "No" response:

Analyst: _____ Date: _____

Second check: _____ Date: _____

APPENDIX I

Preparation Method for Calibration Standards

Analytical Standard Record

16K0095

Description:	SEQ-CAL2	Expires:	02/02/2017
Standard Type:	Calibration Standard	Prepared:	11/02/2016
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	11/04/2016 13:42 by RGB

Analyte	Parent	CAS Number	Concentration	Units
Aluminum	16F0711	7429-90-5	0.2	ug/mL
Antimony	16F0711	7440-36-0	0.01	ug/mL
Arsenic	16F0711	7440-38-2	0.005	ug/mL
Barium	16F0711	7440-39-3	0.01	ug/mL
Beryllium	16F0711	7440-41-7	0.002	ug/mL
Boron	16F0711	7440-42-8	0.03	ug/mL
Cadmium	16F0711	7440-43-9	0.002	ug/mL
Calcium	16F0711	7440-70-2	2	ug/mL
Chromium	16F0711	7440-47-3	0.004	ug/mL
Cobalt	16F0711	7440-48-4	0.01	ug/mL
Copper	16F0711	7440-50-8	0.01	ug/mL
Iron	16F0711	7439-89-6	0.1	ug/mL
Lead	16F0711	7439-92-1	0.003	ug/mL
Lithium (outside certification)	16F0711	7439-93-2	0.006	ug/mL
Magnesium	16F0711	7439-95-4	3	ug/mL
Manganese	16F0711	7439-96-5	0.006	ug/mL
Molybdenum	16F0711	7439-98-7	0.01	ug/mL
Nickel	16F0711	7440-02-0	0.006	ug/mL
Potassium	16F0711	7440-09-7	3	ug/mL
Selenium	16F0711	7782-49-2	0.005	ug/mL
Silver	16F0711	7440-22-4	0.003	ug/mL
Sodium	16F0711	7440-23-5	3	ug/mL
Strontium	16F0711	7440-24-6	0.006	ug/mL
Thallium	16F0711	7440-28-0	0.008	ug/mL
Tin	16F0711	7440-31-5	0.02	ug/mL
Titanium	16F0711	7440-32-6	0.01	ug/mL
Vanadium	16F0711	7440-62-2	0.01	ug/mL
Zinc	16F0711	7440-66-6	0.01	ug/mL

Parent Standards used:

Standard	Description	Prepared	Prepared By	Lot Nbr	Expires	Last Edit		(mls)
16F0711	METALS RL STOCK SOLN.#A	06/28/2016	Roger Burr		06/28/2017	06/28/2016 17:52	by RGB	1

Analytical Standard Record

Empirical Laboratories, LLC

12F0296

Description:	SEQ-CAL3	Expires:	09/19/2012
Standard Type:	Calibration Stan	Prepared:	06/19/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	06/19/2012 08:30 by RGB

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	1	ppm
Antimony	7440-36-0	1	ppm
Arsenic	7440-38-2	1	ppm
Barium	7440-39-3	1	ppm
Beryllium	7440-41-7	1	ppm
Boron	7440-42-8	1	ppm
Cadmium	7440-43-9	1	ppm
Calcium	7440-70-2	50	ppm
Chromium	7440-47-3	1	ppm
Cobalt	7440-48-4	1	ppm
Copper	7440-50-8	1	ppm
Iron	7439-89-6	10	ppm
Lead	7439-92-1	1	ppm
Aluminum	7429-90-5	10	ppm
Magnesium	7439-95-4	50	ppm
Zinc	7440-66-6	1	ppm
Molybdenum	7439-98-7	1	ppm
Nickel	7440-02-0	1	ppm
Potassium	7440-09-7	10	ppm
Selenium	7782-49-2	1	ppm
Silicon	7440-21-3	1	ppm
Silver	7440-22-4	0.5	ppm
Sodium	7440-23-5	50	ppm
Strontium	7440-24-6	1	ppm
Thallium	7440-28-0	1	ppm
Tin	7440-31-5	1	ppm
Titanium	7440-32-6	1	ppm
Vanadium	7440-62-2	1	ppm
Lithium	7439-93-2	1	ppm

Analytical Standard Record

Empirical Laboratories, LLC

12F0296

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
1110590	Sodium	09/22/2011	Roger Burr	03/15/2013	09/22/2011 17:03 by RGB	0.49
11J0586	QC2I	10/21/2011	Roger Burr	10/30/2012	10/21/2011 09:29 by RGB	1
11K0001	QCS7M	11/01/2011	Roger Burr	10/27/2012	11/01/2011 07:20 by RGB	1
11K0003	Calcium	11/01/2011	Roger Burr	04/27/2013	11/01/2011 07:24 by RGB	0.49
12B0334	Aluminum	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:12 by RGB	0.09
12B0336	Iron	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:13 by RGB	0.09
12B0592	Magnesium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:46 by RGB	0.49
12F0047	SN100	06/05/2012	Roger Burr	09/05/2012	06/05/2012 09:14 by RGB	1

Analytical Standard Record

Empirical Laboratories, LLC

12F0288

Description:	SEQ-CAL4	Expires:	09/19/2012
Standard Type:	Calibration Stan	Prepared:	06/19/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	200	Department:	METALS
Vials:	1	Last Edit:	06/19/2012 07:56 by RGB

Analyte	CAS Number	Concentration	Units
Silver	7440-22-4	2	ppm
Manganese	7439-96-5	10	ppm
Barium	7440-39-3	5	ppm

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11G0771	SILVER	07/29/2011	Roger Burr	01/22/2013	07/29/2011 14:33 by RGB	0.4
11K0002	BARIUM	11/01/2011	Roger Burr	04/27/2013	11/01/2011 07:22 by RGB	0.1
12B0589	MANGANESE	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:41 by RGB	2

Analytical Standard Record

Empirical Laboratories, LLC

12G0046

Description:	SEQ-CAL5	Expires:	10/03/2012
Standard Type:	Calibration Stan	Prepared:	07/03/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	07/03/2012 10:19 by RGB

Analyte	CAS Number	Concentration	Units
Magnesium	7439-95-4	500	ppm
Iron	7439-89-6	500	ppm
Aluminum	7429-90-5	500	ppm

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11G0766	ALUMINUM	07/29/2011	Roger Burr	01/22/2013	07/29/2011 14:29 by RGB	25
11L0203	IRON	12/07/2011	Roger Burr	05/29/2013	12/07/2011 10:07 by RGB	25
12B0592	Magnesium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:46 by RGB	25

Analytical Standard Record
Empirical Laboratories, LLC
12G0045

Description:	SEQ-CAL6	Expires:	10/03/2012
Standard Type:	Calibration Stan	Prepared:	07/03/2012
Solvent:	2% HN03 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	07/03/2012 10:16 by RGB

Analyte	CAS Number	Concentration	Units
Magnesium	7439-95-4	10	mg/L
Arsenic	7440-38-2	10	mg/L
Beryllium	7440-41-7	10	mg/L
Boron	7440-42-8	5	mg/L
Cadmium	7440-43-9	10	mg/L
Calcium	7440-70-2	10	mg/L
Chromium	7440-47-3	10	mg/L
Cobalt	7440-48-4	10	mg/L
Copper	7440-50-8	10	mg/L
Iron	7439-89-6	10	mg/L
Antimony	7440-36-0	10	mg/L
Lithium	7439-93-2	10	mg/L
Zinc	7440-66-6	10	mg/L
Manganese	7439-96-5	10	mg/L
Molybdenum	7439-98-7	10	mg/L
Nickel	7440-02-0	10	mg/L
Selenium	7782-49-2	10	mg/L
Strontium	7440-24-6	10	mg/L
Thallium	7440-28-0	10	mg/L
Tin	7440-31-5	5	mg/L
Titanium	7440-32-6	10	mg/L
Vanadium	7440-62-2	10	mg/L
Lead	7439-92-1	10	mg/L

Analytical Standard Record
Empirical Laboratories, LLC
12G0045

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11F0749	TIN	06/24/2011	Roger Burr	12/20/2012	06/24/2011 16:05 by RGB	0.25
11J0586	QC21	10/21/2011	Roger Burr	10/30/2012	10/21/2011 09:29 by RGB	50
12B0588	Boron	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:40 by RGB	2.5

Analytical Standard Record
Empirical Laboratories, LLC
12D0317

Description:	SEQ-CAL7	Expires:	07/31/2012
Standard Type:	Calibration Stan	Prepared:	04/12/2012
Solvent:	2% HN03 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	07/18/2012 08:46 by RGB

Analyte	CAS Number	Concentration	Units
Sodium	7440-23-5	100	ppm
Potassium	7440-09-7	100	ppm
Calcium	7440-70-2	100	ppm

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11B0105	Potassium	02/04/2011	Roger Burr	07/31/2012	02/04/2011 14:07 by RGB	5
11F0751	Sodium	06/24/2011	Roger Burr	12/20/2012	06/24/2011 16:10 by RGB	5
12B0591	Calcium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:45 by RGB	5

Analytical Standard Record

Empirical Laboratories, LLC

12E0004

Description:	SEQ-CAL8	Expires:	08/01/2012
Standard Type:	Calibration Stan	Prepared:	05/01/2012
Solvent:	2% HNO3 5% HCL	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	05/01/2012 08:30 by RGB

Analyte	CAS Number	Concentration	Units
Sodium	7440-23-5	500	ppm
Magnesium	7439-95-4	100	ppm
Calcium	7440-70-2	500	ppm

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11K0003	Calcium	11/01/2011	Roger Burr	04/27/2013	11/01/2011 07:24 by RGB	25
12B0590	Sodium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:43 by RGB	25
12B0592	Magnesium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:46 by RGB	5

Analytical Standard Record

Empirical Laboratories, LLC

12G0359

Description:	ICV METALS	Expires:	07/19/2012
Standard Type:	Calibration Stan	Prepared:	07/18/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	07/18/2012 08:46 by RGB

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	1	ppm
Antimony	7440-36-0	1	ppm
Arsenic	7440-38-2	1	ppm
Barium	7440-39-3	1	ppm
Beryllium	7440-41-7	1	ppm
Boron	7440-42-8	1	ppm
Cadmium	7440-43-9	1	ppm
Calcium	7440-70-2	50	ppm
Chromium	7440-47-3	1	ppm
Cobalt	7440-48-4	1	ppm
Copper	7440-50-8	1	ppm
Iron	7439-89-6	10	ppm
Lead	7439-92-1	1	ppm
Aluminum	7429-90-5	10	ppm
Magnesium	7439-95-4	50	ppm
Zinc	7440-66-6	1	ppm
Molybdenum	7439-98-7	1	ppm
Nickel	7440-02-0	1	ppm
Phosphorus, Total (as P)	7723-14-0	1	ppm
Potassium	7440-09-7	10	ppm
Selenium	7782-49-2	1	ppm
Silicon	7440-21-3	5	ppm
Silver	7440-22-4	0.5	ppm
Sodium	7440-23-5	50	ppm
Strontium	7440-24-6	1	ppm
Thallium	7440-28-0	1	ppm
Tin	7440-31-5	1	ppm
Titanium	7440-32-6	1	ppm
Vanadium	7440-62-2	1	ppm
Lithium	7439-93-2	1	ppm

Analytical Standard Record
Empirical Laboratories, LLC
12G0359

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11D0726	QC23	04/29/2011	Roger Burr	07/31/2012	04/29/2011 15:34 by RGB	1
1180587	Magnesium	09/22/2011	Roger Burr	03/15/2013	09/22/2011 16:59 by RGB	0.49
1180590	Sodium	09/22/2011	Roger Burr	03/15/2013	09/22/2011 17:03 by RGB	0.49
11K0003	Calcium	11/01/2011	Roger Burr	04/27/2013	11/01/2011 07:24 by RGB	0.49
12B0334	Aluminum	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:12 by RGB	0.09
12B0336	Iron	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:13 by RGB	0.09
12B0687	QCS7A	02/23/2012	Roger Burr	02/28/2013	02/23/2012 16:00 by RGB	1

Analytical Standard Record
Empirical Laboratories, LLC
12G0360

Description:	CCV METALS	Expires:	07/19/2012
Standard Type:	Calibration Stan	Prepared:	07/18/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	07/18/2012 08:47 by RGB

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	1	ppm
Antimony	7440-36-0	1	ppm
Arsenic	7440-38-2	1	ppm
Barium	7440-39-3	1	ppm
Beryllium	7440-41-7	1	ppm
Boron	7440-42-8	1	ppm
Cadmium	7440-43-9	1	ppm
Calcium	7440-70-2	50	ppm
Chromium	7440-47-3	1	ppm
Cobalt	7440-48-4	1	ppm
Copper	7440-50-8	1	ppm
Iron	7439-89-6	10	ppm
Lead	7439-92-1	1	ppm
Aluminum	7429-90-5	10	ppm
Magnesium	7439-95-4	50	ppm
Zinc	7440-66-6	1	ppm
Molybdenum	7439-98-7	1	ppm
Nickel	7440-02-0	1	ppm
Potassium	7440-09-7	10	ppm
Selenium	7782-49-2	1	ppm
Silicon	7440-21-3	1	ppm
Silver	7440-22-4	0.5	ppm
Sodium	7440-23-5	50	ppm
Strontium	7440-24-6	1	ppm
Thallium	7440-28-0	1	ppm
Tin	7440-31-5	1	ppm
Titanium	7440-32-6	1	ppm
Vanadium	7440-62-2	1	ppm
Lithium	7439-93-2	1	ppm

Analytical Standard Record

Empirical Laboratories, LLC

12G0360

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11B0590	Sodium	09/22/2011	Roger Burr	03/15/2013	09/22/2011 17:03 by RGB	0.49
11J0586	QC21	10/21/2011	Roger Burr	10/30/2012	10/21/2011 09:29 by RGB	1
11K0001	QCS7M	11/01/2011	Roger Burr	10/27/2012	11/01/2011 07:20 by RGB	1
11K0003	Calcium	11/01/2011	Roger Burr	04/27/2013	11/01/2011 07:24 by RGB	0.49
12B0334	Aluminum	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:12 by RGB	0.09
12B0336	Iron	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:13 by RGB	0.09
12B0592	Magnesium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:46 by RGB	0.49
12F0047	SN100	06/05/2012	Roger Burr	09/05/2012	06/05/2012 09:14 by RGB	1

Analytical Standard Record

Empirical Laboratories, LLC

14G0174

Description:	RL STD A	Expires:	10/08/2014
Standard Type:	Calibration Stand:	Prepared:	07/08/2014
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	07/11/2014 12:36 by RGB

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	0.006	ug/mL
Antimony	7440-36-0	0.008	ug/mL
Arsenic	7440-38-2	0.006	ug/mL
Barium	7440-39-3	0.01	ug/mL
Beryllium	7440-41-7	0.002	ug/mL
Cadmium	7440-43-9	0.002	ug/mL
Calcium	7440-70-2	2	ug/mL
Chromium	7440-47-3	0.004	ug/mL
Cobalt	7440-48-4	0.01	ug/mL
Copper	7440-50-8	0.008	ug/mL
Iron	7439-89-6	0.06	ug/mL
Lead	7439-92-1	0.003	ug/mL
Aluminum	7429-90-5	0.1	ug/mL
Magnesium	7439-95-4	3	ug/mL
Zinc	7440-66-6	0.01	ug/mL
Molybdenum	7439-98-7	0.01	ug/mL
Nickel	7440-02-0	0.006	ug/mL
Potassium	7440-09-7	3	ug/mL
Selenium	7782-49-2	0.006	ug/mL
Silver	7440-22-4	0.002	ug/mL
Sodium	7440-23-5	3	ug/mL
Strontium	7440-24-6	0.006	ug/mL
Thallium	7440-28-0	0.004	ug/mL
Tin	7440-31-5	0.02	ug/mL
Titanium	7440-32-6	0.01	ug/mL
Vanadium	7440-62-2	0.01	ug/mL
Lithium (outside certification)	7439-93-2	0.006	ug/mL

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

14G0174

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
14A0389	METALS RL STOCK SOLN.#A	01/21/2014	Roger Burr	01/21/2015	07/11/2014 12:36 by RGB	1

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

14G0175

Description:	RL STD #1	Expires:	10/08/2014
Standard Type:	Calibration Stand	Prepared:	07/08/2014
Solvent:	2% HN03 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	07/11/2014 12:37 by RGB

Analyte	CAS Number	Concentration	Units
Selenium	7782-49-2	0.005	ug/mL
Lead	7439-92-1	0.003	ug/mL
Boron	7440-42-8	0.03	ug/mL
Arsenic	7440-38-2	0.005	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
14E0324	METALS RL STOCK SOLN. 1	05/14/2014	Roger Burr	05/14/2015	07/11/2014 12:37 by RGB	1

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

14E0324

Description:	METALS RL STOCK SOLN. 1	Expires:	05/14/2015
Standard Type:	Analyte Spike	Prepared:	05/14/2014
Solvent:	2% HN03 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	1000	Department:	METALS
Vials:	1	Last Edit:	07/11/2014 12:37 by RGB

Analyte	CAS Number	Concentration	Units
Selenium	7782-49-2	0.5	ug/mL
Lead	7439-92-1	0.3	ug/mL
Boron	7440-42-8	3	ug/mL
Arsenic	7440-38-2	0.5	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
13C0381	SELENIUM	03/19/2013	Roger Burr	09/14/2014	03/19/2013 16:31 by RGB	0.5
13L0594	Boron	12/27/2013	Roger Burr	04/30/2015	12/27/2013 13:52 by RGB	3
14B0262	ARSENIC	02/14/2014	Roger Burr	07/28/2015	02/14/2014 16:17 by RGB	0.5
14B0267	Lead	02/14/2014	Roger Burr	07/28/2015	02/14/2014 16:26 by RGB	0.3

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

14A0389

Description:	METALS RL STOCK SOLN.#A	Expires:	01/21/2015
Standard Type:	Analyte Spike	Prepared:	01/21/2014
Solvent:	2% HN03 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	1000	Department:	METALS
Vials:	1	Last Edit:	07/11/2014 12:36 by RGB

X100 FOR ANALYSIS. USED FOR RLSTD-A

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	0.6	ug/mL
Antimony	7440-36-0	0.8	ug/mL
Arsenic	7440-38-2	0.6	ug/mL
Barium	7440-39-3	1	ug/mL
Beryllium	7440-41-7	0.2	ug/mL
Cadmium	7440-43-9	0.2	ug/mL
Calcium	7440-70-2	200	ug/mL
Chromium	7440-47-3	0.4	ug/mL
Cobalt	7440-48-4	1	ug/mL
Copper	7440-50-8	0.8	ug/mL
Iron	7439-89-6	6	ug/mL
Lead	7439-92-1	0.3	ug/mL
Aluminum	7429-90-5	10	ug/mL
Magnesium	7439-95-4	300	ug/mL
Zinc	7440-66-6	1	ug/mL
Molybdenum	7439-98-7	1	ug/mL
Nickel	7440-02-0	0.6	ug/mL
Potassium	7440-09-7	300	ug/mL
Selenium	7782-49-2	0.6	ug/mL
Silver	7440-22-4	0.2	ug/mL
Sodium	7440-23-5	300	ug/mL
Strontium	7440-24-6	0.6	ug/mL
Thallium	7440-28-0	0.4	ug/mL
Tin	7440-31-5	2	ug/mL
Titanium	7440-32-6	1	ug/mL
Vanadium	7440-62-2	1	ug/mL
Lithium (outside certification)	7439-93-2	0.6	ug/mL

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

14A0389

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
12F0166	COBALT	06/12/2012	Roger Burr	05/11/2013	06/12/2012 14:23 by RGB	1
12G0723	ARSENIC	07/30/2012	Roger Burr	01/25/2014	07/30/2012 16:15 by RGB	0.6
12G0724	Lead	07/30/2012	Roger Burr	01/25/2014	07/30/2012 16:16 by RGB	0.3
12L0061	SILVER	12/04/2012	Roger Burr	05/09/2014	12/04/2012 12:48 by RGB	0.2
12L0062	BARIUM	12/04/2012	Roger Burr	05/09/2014	12/04/2012 12:49 by RGB	0.1
12L0065	Antimony	12/04/2012	Roger Burr	05/09/2014	12/04/2012 12:55 by RGB	0.8
13A0259	CADMIUM	01/15/2013	Roger Burr	07/10/2014	01/15/2013 14:45 by RGB	0.2
13A0260	COPPER	01/15/2013	Roger Burr	07/10/2014	01/15/2013 14:46 by RGB	0.8
13A0261	LITHIUM	01/15/2013	Roger Burr	07/10/2014	01/15/2013 14:47 by RGB	0.6
13A0266	BERYLLIUM	01/15/2013	Roger Burr	07/10/2014	01/15/2013 14:52 by RGB	0.2
13B0291	THALLIUM	02/12/2013	Roger Burr	07/31/2014	02/17/2014 16:00 by RGB	0.4
13C0377	ZINC	03/19/2013	Roger Burr	09/14/2014	03/19/2013 16:27 by RGB	1
13C0378	CHROMIUM	03/19/2013	Roger Burr	09/14/2014	03/19/2013 16:28 by RGB	0.4
13C0379	Magnesium	03/19/2013	Roger Burr	09/14/2014	03/19/2013 16:29 by RGB	30
13C0380	NICKEL	03/19/2013	Roger Burr	09/14/2014	03/19/2013 16:30 by RGB	0.6
13C0381	SELENIUM	03/19/2013	Roger Burr	09/14/2014	03/19/2013 16:31 by RGB	0.6
13C0382	VANADIUM	03/19/2013	Roger Burr	09/14/2014	03/19/2013 16:32 by RGB	1
13F0307	IRON	06/11/2013	Roger Burr	11/20/2014	06/11/2013 09:30 by RGB	0.6
13F0727	Calcium	06/27/2013	Roger Burr	11/20/2014	06/27/2013 17:11 by RGB	20
13H0392	Molybdenum	08/19/2013	Roger Burr	01/18/2015	08/19/2013 16:34 by RGB	1
13H0393	MANGANESE	08/19/2013	Roger Burr	01/18/2015	08/19/2013 16:36 by RGB	0.6
13H0394	TIN	08/19/2013	Roger Burr	01/18/2015	08/19/2013 16:37 by RGB	2
13H0395	STRONTIUM	08/19/2013	Roger Burr	01/18/2015	08/19/2013 16:38 by RGB	0.6
13H0396	Titanium	08/19/2013	Roger Burr	01/18/2015	08/19/2013 16:39 by RGB	1
13K0524	Potassium	11/22/2013	Roger Burr	04/30/2015	11/22/2013 08:39 by RGB	30
13L0591	ALUMINUM	12/27/2013	Roger Burr	04/30/2015	12/27/2013 13:33 by RGB	1
13L0593	Sodium	12/27/2013	Roger Burr	04/30/2015	12/27/2013 13:50 by RGB	30

Reviewed By _____ Date _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP 145 REVISION #: 15 EFFECTIVE DATE: 20170328

DETERMINATION OF INORGANIC ANIONS IN WATER BY ION CHROMATOGRAPH USING THE DIONEX ION CHROMATOGRAPH WITH HYDROXIDE ELUENT AND DIONEX AS18 COLUMN

References:
USEPA METHOD 300.0 Revision 2.1/ SW846 Method 9056A

APPROVALS:

Lab Director: _____ Date: 20170328

Data Quality Manager: _____ Date: 20170328

Group Leader: _____ Date: 20170328
Jade Holliman

Changes Summary:

SOP145_R15_20170328_Anions

- Removed information relative to IC1 – instrument has been removed from service, and removed references to 9056 leaving only 9056A references.
- Updated routine bromide limits.

SOP145_R14_20151221_Anions

- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS. Removed "Was the appropriate SOP followed?".

SOP145_R13_20151111_Anions

- Initial EPA method references updated to include the revision number.

SOP145_R12_20150921_Anions

- References to section supervisors revised to reflect group leaders.
- References to SOP145A removed and section 14.3.2 added for preparation of soils.
- Removed specifics from section 5 summary.
- Section 6 updated to remove method definitions and simply reference QS08.
- Section 7.1.7 updated to reflect general statement of 4.5 minutes following NO2.
- Section 11 updated to include solid holding time.
- Section 12 updated to remove QC specifics and reference Table 2.
- Section 13 updated to reflect IC2 updates and reference Table 2.
- Section 14 updated to add IC2 operating instructions.
- Analyst Review Checklist updated.

Revision 11 20140729

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark update to include proprietary reference.
- MSDS sheet reference updated to reflect vendor website access
- Section 9, 13 and 14 updated to reference new instrumentation
- Sections 21- 25 populated/updated.
- QSM references added to section 26.
- Table 1 updated to include QSM recovery limits.
- Table 2 updated to include QSM5.0 criteria
- Table 3 Data review checklist updated.

Revision 10, 20130806

- Updated Table 3, Analyst Data Review Checklist to include manual integrations checked by a second reviewer.
- Defined standard expiration dates for vendor stocks, intermediate standards and dilute working standards in section VII and moved Traceability to this section.

Revision 09, 08/20/2012

- QS SOP references added as appropriate.
- Anion F-Table from DoD QSM added to appendix.

- Checklist updated to require LCR recovery calculation on the raw data and add space to record sample reflecting full recalculation.
- Reference to SOP145A added for soils/sediments.

Revision 08 Date: 05/16/2011

- This is an update from SOP revision 07 dated 03//25/10
- Frequency requirements for MDL study, calibration curve and linear calibration range verification added.
- References updated to include method 9056A
- Linear calibration range verification criteria added.
- Addressed method 9056 request for dilution with eluent.

Revision Date: 03/25/2010

- The SOP has been reviewed for accuracy and completeness.
- All references to analysis of ortho-phosphorus by this method have been removed.

Table of Contents

DETERMINATION OF INORGANIC ANIONS IN WATER BY ION CHROMATOGRAPH USING THE DIONEX ION CHROMATOGRAPH WITH HYDROXIDE ELUENT AND DIONEX AS18 COLUMN

References:
USEPA METHOD 300.0 Revision 2.1/ SW846 Method 9056A

1. Identification of the Test Method

USEPA METHOD 300.0 Revision 2.1/ SW846 Method 9056A

2. Applicable Matrix or matrices

This method covers the determination inorganic common anions in reagent water, surface water, ground water, and other aqueous matrixes. This method may be applied to soils/sediments after creation of a leachate.

3. Limits of Detection and Quantitation

See table 1.

4. Scope and Application, Including Parameters to be Analyzed

4.1. This method covers the determination of the following inorganic common anions in reagent water, surface water, ground water, and other aqueous matrixes. This method may be applied to soils/sediments after creating a leachate.

PART A.--Common Anions

Chloride	Nitrate	Fluoride
Nitrite	Bromide	Sulfate

4.2. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

4.3. When the method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a laboratory fortified matrix sample covering the anions of interest. The fortification procedure is described in the Quality Control section.

4.4. Users of the method data should state the data-quality objectives prior to analysis. Analyst using this method must demonstrate the ability to generate acceptable results with the method, using the procedures described in the Quality Control section.

5. Summary of the Test Method

A small volume of sample is introduced into an ion chromatograph (IC). The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

6. Definitions

6.1. Laboratory Quality System QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7. Interference

7.1. Interferences can be divided into three different categories: **direct chromatographic coelution**, where an analyte response is observed at very nearly the same retention time as the target anion; **concentration dependant coelution**, which is observed when the response of higher than typical concentrations of the neighboring peak overlap into the retention window of the target anion; and, **ionic character displacement**, where retention times may significantly shift due to the influence of high ionic strength matrices (high mineral content or hardness) overloading the exchange sites in the column and significantly shortening target analytes' retention times.

7.1.1. A direct chromatographic coelution may be solved by changing columns, eluent strength, modifying the eluent with organic solvents (if compatible with IC columns), changing the detection systems, or selective removal of the interference with pretreatment. Sample dilution will have little to no effect. The analyst must verify that these changes do not negatively affect performance by repeating and passing all the criteria in the Quality Control Section.

7.1.2. Sample dilution may resolve some of the difficulties if the interference is the result of either concentration dependant coelution or ionic character displacement, but it must be clarified that sample dilution will alter your Minimum Reporting Limit (MRL or LOQ) by a proportion equivalent to that of the dilution. Therefore, careful consideration of project objectives should be given prior to performing such a dilution. An alternative to sample dilution may be dilution of the eluent.

7.1.3. Pretreatment cartridges can be effective as a means to eliminate certain matrix interferences. Prior to using any pretreatment, the analyst should be aware that all **instrument calibration standards must be pretreated in exactly the same manner** as the pretreated unknown field samples. The need for these cartridges has been greatly reduced with recent advances in high capacity anion exchange columns.

7.1.3.1. Extreme caution should be exercised in using these pretreatment cartridges. Artifacts are known to leach from certain cartridges, which can foul the guard, and analytical columns causing loss of column capacity indicated by shortened retention times and irreproducible results. Frequently compare your calibration standard chromatograms to those of the column test chromatogram (received when the column was purchased) to insure proper separation and similar response rations between the target analytes is observed.

7.1.4. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in an ion chromatogram. These interferences can lead to false positive results for target analytes as well as reduced detection limits as a consequence of elevated baseline noise.

7.1.5. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

7.1.6. Any anion that is only weakly retained by the column may elute in the retention time window of fluoride and potentially interfere. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the analyst to generate precision and accuracy information in each sample matrix.

7.1.7. Close attention should be given to the potential for carry over peaks from one analysis which will effect the proper detection of analytes of interest in a second, subsequent analysis. The elution of nitrate indicates the end of a chromatographic run. A run time including an additional 4-5 minutes following nitrate is recommended to allow for the proper elution of any potentially interfering late peaks. It is the responsibility of the analyst to confirm that no late eluting peaks have carried over into a subsequent analysis thereby compromising the integrity of the analytical results.

8. Safety

8.1. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be follow lab wide.

8.2. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.

8.3. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.4. MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your Group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9. Equipment and Supplies

9.1. Ion Chromatograph (IC) – Analytical system complete with eluent generator, an ion chromatographic pump, injection valves, both guard and analytical separator columns, suppressor, conductivity detector, and computer based data acquisition system. Dionex DX-500, Dionex ICS-2100, or equivalent.

9.1.1. Anion guard column--Dionex Ion Pac AG18 4mm (P/N 060551), or equivalent. This column functions as a protector of the separator column. If omitted from the system, the retention times will be shorter.

9.1.2. Anion separator column--Dionex Ion Pac AS18, 4mm (P/N 060549), or equivalent. An optional column (2mm or 4 mm) may be used if comparable resolution of peaks is obtained, and the quality control requirements can be met. Comparable results can be attained using the Dionex, AS17, 4 mm column.

9.2. Gradient Pump – Dionex GP50, or Dionex AS-DV, or equivalent.

9.3. Anion suppressor device--The data presented in this method were generated using an Ultra 4 mm Dionex Anion Self Regenerating Suppressor (ASRS, P/N 082540). An equivalent suppressor device may be utilized provided comparable conductivity detection limits are achieved and adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background conductivity. Proper suppressor performance is essential to analytical data reproducibility and sensitivity of the conductivity detector.

9.3.1. The ASRS was set to perform electrolytic suppression at a current setting of 300 ma using the external water mode. External water was delivered to the suppressor directly from a pressurized source at a flow rate of 5 mL/min. It should be noted that while Empirical Laboratories has the suppressor currently set at 300 mA, no external water is being used at this time.

9.4. Detector--Conductivity cell (Dionex CD20, Dionex ICS Series VWD 1CH, or equivalent) capable of providing data as required in the Quality Control section of this SOP.

9.5. Data Acquisition System--The Dionex Peaknet Data Chromatography Software version 5.2, Chromeleon 7, or equivalent.
9.6. Top-Loading balance-- Used to accurately weigh soil samples.
9.7. Micro beakers -- Plastic, disposable - used during sample preparation.
9.8. Syringes--Plastic, disposable, 5 mL - used during sample preparation.
9.9. Eppendorfs with variable settings- 1mL and 5 mL. Must be calibrated quarterly (verified **daily** for QSM5.0).
9.10. Bottles -- High density polyethylene (HDPE) or glass, amber or clear, 30 mL, 125 mL, 250 mL. 500mL. for sampling and storage of calibration solutions.
9.11. Particulate filters-- 0.45 micron syringe filters, specifically designed for IC applications (Thermo Scientific Target 2, PN 033911C, or equivalent). These cartridges are used to remove particulates from the sample matrix while loading the sample manually or if the autosampler employed does not filter the sample during loading.

NOTE: See method for several types of pretreatment cartridges that are available and may be useful depending on the matrices of the samples normally processed.

9.12. Autosampler PolyVials 5-mL size, with filter caps, 250 each --Dionex cat log # 38141.
9.13. Shaker for use when preparing soil samples.
9.14. Centrifuge to aid in separation after soil preparation.
9.15. Centrifuge tubes--50 mL capacity

10. Reagents and Standards

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

10.1. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method. **All reagents shall be made from ACS reagent grade chemicals. All reagents used for distillation and analysis are entered into Element. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the samples they were used with.**
10.2. Reagent water-- Distilled or deionized water 17.8 Mohm or better, free of anions of interest. Water should contain particles no larger than 0.20 microns.
10.3. A system or apparatus which automatically generates the hydroxide eluent (Dionex EG40, or equivalent) is an acceptable alternative to physically preparing the hydroxide eluent.
10.4. Stock standard solutions, 1000 mg/L (1mg/mL): Stock standard solutions are purchased as certified solutions from selected vendors. Expiration dates are assigned as identified by the vendor.
10.5. Intermediate standard solutions: Prepared by dilution of stock standard solutions and assigned expiration date of 3 months from preparation.
10.6. Dilute working standards: Prepared by dilution of intermediate standard solutions and assigned expiration date of 2 days from preparation.

Traceability

A record shall be maintained on all reference materials within Element. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in Element as well as on the container's label.

All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date, date opened. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded within Element. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

11. Sample Collection, Preservation, Shipment, and Storage

11.1. Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2. Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

11.3. Sample preservation and holding times for the anions that can be determined by this method are as follows:

PART A: Common Anions

Analyte	Preservation	Holding Time
Bromide	None required	28 days
Chloride	None required	28 days
Fluoride	None required	28 days
Nitrate-N	**Cool to 4 °C**	**48 hours**
Nitrite-N	**Cool to 4 °C**	**48 hours**
Sulfate	Cool to 4 °C	28 days

11.4. When collecting a sample from a treatment plant employing chlorine dioxide, the sample must be sparged with an inert gas (helium, argon, nitrogen) prior to addition of the EDA preservative at time of sample collection.

11.5. There is no specified holding time for soil samples. The laboratory will consider six months as the maximum holding time.

12. Quality Control

12.1. Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2. The laboratory is required to operate a formal quality control (QC) program. The requirements of this program consist of an initial demonstration of laboratory performance, and subsequent analysis in each analysis batch of a Laboratory Reagent Blank, Laboratory Fortified Blank, Instrument Performance Check Standard, calibration check standards, Laboratory Fortified Sample Matrices (LFM) and either Field, Laboratory or LFM duplicate sample analyses. This section

details the specific requirements for each of these QC parameters. The laboratory is required to maintain performance records that define the quality of the data that are generated.

12.3. INITIAL DEMONSTRATION OF PERFORMANCE

12.3.1. The initial demonstration of performance is used to characterize instrument performance (determination of accuracy through the analysis of the QCS) and laboratory performance (determination of MDLs) prior to performing analysis by this method.

12.3.2. Quality Control Sample (QCS) – When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within ±10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

12.3.3. Method Detection Limit (MDL)—MDLs are established for all analytes, using reagent water (blank) fortified at a concentration of three to five times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

MDL = (t) x (S)

Where,

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates].

S = standard deviation of the replicate analyses.

12.3.4. MDLs should be generated when a new operator begins work or whenever there is a significant change in the background, or instrument response. Method recommends MDLs every 6 months but Empirical performs annually with quarterly verifications.

12.4. Assessing Laboratory Performance

12.4.1. Laboratory Reagent Blank (BLK) – The laboratory must analyze at least one LRB with each analysis batch. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis. See table 2 for criteria.

12.4.2. Laboratory Fortified Blank (BS)—The BS should be prepared at concentrations similar to those expected in the field samples and ideally at the same concentration used to prepare the MS/MSD. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required concentration dependant control limits that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses. See table 2 for criteria.

12.4.3. Instrument Performance Check Solution (ICV) – The Initial Calibration Check Standard is to be evaluated as the instrument performance check solution in order to confirm proper instrument performance. The acceptable limits for this standard are 90 to 110%. Small variations in retention time can be anticipated when a new solution of eluent (or when the KOH cartridge is changed) is prepared but if shifts of more that 2% are observed in the IPC retention time, some type of instrument problem is present. Potential problems improperly prepared eluent, erroneous method parameters programmed such as flow rate or some other system problem. The chromatographic profile (elution order) of the target anions following an ion chromatographic analysis should closely replicate the profile displayed in the test chromatogram that was shipped when the column was purchased. As a column ages, it is normal to see a gradual shift and shortening of retention times, but if after several years of use, extensive use over less than a year, or use with harsh samples, this retention time has noticeably shifted to any less than 80% of the original recorded value, the column may require cleaning or replacement. Particularly if resolution problems are beginning to become common between previously resolved peaks. A laboratory must retain a historic record of retention times for all the target anions in the ICV to provide evidence of an analytical column's vitality.

12.5. Assessing Analyte Recovery and Data Quality

12.5.1. Laboratory Fortified Sample Matrix (MS) – The laboratory adds a known amount of analyte to a minimum of 10% of the field samples within an analysis batch. The MS sample is prepared from a sample matrix which has been analyzed prior to fortification. The analyte concentration must be high enough to be detected above the original sample and should adhere to the QC requirements. It is recommended that the solutions used to fortify the MS be prepared from the same stocks used to prepare the calibration standards and not from external source stocks. This will remove the bias contributed by an externally prepared stock and focus on any potential bias introduced by the field sample matrix.

12.5.1.1. If the fortified concentration is less than the observed background concentration of the unfortified matrix, the recovery should not be calculated. This is due to the difficulty in calculating accurate recoveries of the fortified concentration when the native sample concentration is so high.

12.5.1.2. Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample. Percent recovery should be calculated using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

where, R = percent recovery.
C_s = fortified sample concentration
C = sample background concentration
S = concentration equivalent of analyte added to sample.

12.5.1.3. If the recovery of any analyte falls outside the designated LFM recovery range and the performance for that analyte is shown to be in control, the recovery problem

encountered with the LFM is judged to be matrix induced and the results for that sample and the LFM are reported with a "matrix induced bias" qualifier. See Table 2 for criteria.

12.5.2. FIELD OR LABORATORY DUPLICATES –Analyze either a field, matrix spike duplicate or a laboratory duplicate for a minimum of 10% of the collected field samples or at least one with every analysis batch, whichever is greater. The sample matrix selected for this duplicate analysis must contain measurable concentrations of the target anions in order to establish the precision of the analysis set and insure the quality of the data. If none of the samples within an analysis batch have measurable concentrations, the LFM should be employed as a laboratory duplicate.

12.5.2.1. Calculate the relative percent difference (RPD) of the initial quantitated concentration (I_c) and duplicate quantitated concentration (D_c) using the following formula,

$$RPD = \frac{(I_c - D_c)}{([I_c + D_c]/2)} \times 100$$

12.5.2.2. Duplicate analysis acceptance criteria are found in Table 2.

12.5.2.3. If the RPD fails to meet these criteria, the samples must be reported with a qualifier identifying the sample analysis result as yielding a poor duplicate analysis RPD. This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses) it indicates a problem with the instrument or individual technique.

12.5.3. Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

12.5.4. In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of different columns, injection volumes, and/or eluents, to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in the QC section and adhere to the condition of baseline stability.

12.5.5. The laboratory adopts additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the client and the nature of the samples. Whenever possible, the laboratory performs analysis of quality control check samples and participates in relevant performance evaluation sample studies.

13. Calibration and Standardization

13.1. Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2. Establish ion chromatographic operating parameters.

13.2.1. Estimate the Linear Calibration Range (LCR) – The LCR should cover the expected concentration range of the field samples.

13.2.2. For an individual anion calibration curve, a minimum of five calibration standard concentrations is required. Generally, a total of eight calibration concentrations are analyzed to provide five concentrations for each anion.

13.3. Prepare the calibration standards by carefully adding measured volumes of one or more stock standards to a volumetric flask and diluting to volume with reagent water. Chloride and sulfate are generally calibrated from 0.5-250 mg/L; nitrate from 0.05-25 mg/L. Fluoride and nitrite from 0.05-5.0mg/L and Br from 0.126-63.0 mg/L.

13.4. Using a 4mm column, allow the instrument to inject the programmed amount of each calibration standard. Tabulate peak area responses against the concentration. The results are used to prepare calibration curves using a linear least squares fit for each analyte. Acceptable calibration curves are confirmed after reviewing the curves for linearity and passing the criteria for the initial calibration check standard. See Table 2 for criteria

13.4.1. Use of peak areas is strongly recommended since they have been found to be more consistent, in terms of quantitation, than peak heights. Peak height can tend to be suppressed as a result of high levels of common anions in a given matrix which can compete for exchange sites. Using peak areas, it is the analyst responsibility to review all chromatograms to insure accurate baseline integration of target analyte peaks since poorly drawn baselines will more significantly influence peak areas than peak heights.

13.5. Once the calibration curves have been established they must be verified prior to conducting any sample analysis using an initial calibration check standard. This verification must be performed on each analysis day or whenever fresh eluent has been prepared. A continuing calibration check standard must be analyzed after every tenth field sample and at the end of the analysis set as an end calibration check standard. The response for the initial, continuing and end calibration check must satisfy the QC criteria found in table 2. If during the analysis set, the response differs by more than the calibration verification criteria, or the retention times shift more than ±5% from the expected values for any analyte, the test must be repeated, using fresh calibration standards. If the results are still outside these criteria, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable calibration check standard must be reanalyzed.

13.5.1. Control limits for calibration verification are found in table 2.

13.5.2. Linear Calibration Range must be verified every 6 months by recalculating from a standard at the highest concentration of the analytes' calibration curve. Control limits are found in table 2.

13.6. After satisfying the requirements, the levels selected for the other calibration check standards should be varied between a middle calibration level and the highest calibration level.

13.7. Software Entry of new curve (IC2)

13.7.1. Enter Chromeleon Software from Desktop and setup calibration as a stand-alone run.

13.7.2. Loading a Run: Set up sequence using a previous run as the Template.

13.7.2.1. Make levels 1-8 and name each sample under the SAMPLE heading column. (CAL1-CAL8 plus ICV)

13.7.2.2. SAMPLE TYPE is Calibration Standard.

13.7.2.3. Enter method name under METHOD heading. In most cases, AS18 Method file will be used.

13.7.2.4. Set status to Idle.

13.7.2.5. All other column headings are defaulted to enter "1".

13.7.2.6. To include a command to Shut Down the pump at the end of the run: Name the row following the last sample, Pump Off under the sample heading. Use Method Shutdown

13.7.3. When finished, copy to CALS folder in Chromeleon.

13.7.4. Integrate or assign peaks, as necessary.

13.7.5. Upload data to the LIMS

13.7.6. To change the processing method in Chromeleon:

13.7.6.1. Pull up the CAL run and go into the data.

13.7.6.2. Under data processing home, click processing method.

13.7.6.3. A new pane will pop up – select the calibration tab.

13.7.6.4. For "Origin of standards for fixed calibration", select path that leads to the CAL.

13.7.6.5. Drop points that are not being used by double clicking in the box next to the check mark then selecting the points to drop.

13.7.6.6. Click update.

14. Procedure

14.1. Other columns, chromatographic conditions, or detectors may be used if the requirements of the QC section are met.

14.2. Check system calibration daily and, if required, recalibrate as necessary – minimum every 6 months.

14.3. Sample Preparation

14.3.1. For refrigerated or samples arriving to the laboratory cold, ensure the samples have come to room temperature prior to conducting sample analysis by allowing the samples to warm on the bench for at least 1 hour.

14.3.2. For soils:

14.3.2.1. Weigh a five gram sample into a tared centrifuge tube.

14.3.2.2. Add deionized water up to the 50 mL mark on the tube, place the cap on the tube and secure it well.

14.3.2.3. Place the tube in a shaker for one hour.

14.3.2.4. Place the centrifuge tubes in the centrifuge for 5 minutes at ~ 2000 rpm.

14.4. Using a Luer lock, plastic 5 to 10 mL syringe, withdraw the sample from the micro beaker and attach a 0.45 um particulate filter (demonstrated to be free of ionic contaminants) directly to the syringe. Filter the sample into an autosampler vial.

14.5. Using a 4 mm column, allow the system to add the programmed amount of sample. Tabulate peak area responses against the concentration. During this procedure, retention times must be recorded. Use the same size loop for standards and samples. Record the resulting peak size in area units. An automated constant volume injection system may also be used.

14.6. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

14.7. If the response of a sample analyte exceeds the calibration range, the sample may be diluted with an appropriate amount of reagent water and reanalyzed. If this is not possible then three new calibration concentrations must be employed to create a separate high concentration curve, one standard near the estimated concentration and the other two bracketing around an interval

equivalent to ±25% the estimated concentration. The latter procedure involves significantly more time than a simple sample dilution therefore; it is advisable to collect sufficient sample to allow for sample dilution or sample reanalysis, if required. (Note: Method 9056A samples should be diluted with eluent. As the instrument is configured with an eluent generator, this is not applied.)

14.8. Shifts in retention time are inversely proportional to concentration. Nitrate, phosphate and sulfate will exhibit the greatest degree of change, although all anions can be affected. In some cases this peak migration may produce poor resolution or make peak identification difficult. High sulfate results often cause coelution with Bromide and require a dilution to report Bromide results.

14.9. High Chloride concentrations may result in carryover. Samples following a sample with high chloride concentrations should be carefully reviewed.

14.10. For samples with high intensities, blow-ups are required to see low-level peaks. Provide blow-ups of intensity below 250 for any chromatogram with intensities greater than 250.

14.11. Should more complete resolution be needed between any two coeluting peaks, the eluent can be diluted. This will spread out the run, however, and will cause late eluting anions to be retained even longer. The analysts must verify that this dilution does not negatively affect performance by repeating and passing all the QC criteria.

14.11.1. Eluent dilution will reduce the overall response of an anion due to chromatographic band broadening which will be evident by shortened and broadened peaks. This will adversely affect the MDLs for each analyte.

14.12. **Instrument Information and Startup Routine** (IC2)

14.12.1. Fill Eluent water container with Modulab water from Wet Chemistry Lab.

14.12.2. Reconnect container to instrument.

14.12.3. At analyst discretion, change guard column frit. If necessary, check primary column frits as well.

14.12.4. Open the eluent supply valve. Make sure and hit okay for the warning. Prime for approximately 10 minutes.

14.12.5. Turn pump off. Close valve. Turn pump, eluent generator, Cr-TC and Suppressor on.

14.12.6. Wait for conductivity to reach approximately 0 (total approximately 0.300).

14.12.7. Load the auto sampler cartridges and put autosampler into RUN state.

14.12.8. Allow system to come to equilibrium then start run.

14.13. **Shut down routine for is as follows:**

14.13.1. If the instrument is not going to be operated for a period of time, run deionized water through the eluent lines for ~ 30 minutes to an hour to rinse the lines.

14.13.2. Stop the OFF/ON pump and then select SRS-OFF.

14.13.3. Close gas supply valves and eluent valves on the eluent bottles. Turn off the supply. Is not necessary to vent the eluent bottles.

14.13.4. Power down the modules in any order.

14.14. **General Sample Loading and Run Set-up (IC2)**

14.14.1. Enter Chromeleon Software from Desktop.

14.14.2. Loading a Run: Set up sequence using a previous run as the Template.

14.14.2.1. Name each sample under the SAMPLE heading column. (CCV, CCB, CRL, BS, BLK, sample #'s, etc.)

14.14.2.2. SAMPLE TYPE is sample unknown unless loading a calibration curve.

14.14.2.3. Enter method name under METHOD heading. Processing method is IC2 Anion Processing Method is used

14.14.2.4. Set status to Idle.

14.14.2.5. All other column headings are defaulted to enter "1". Samples requiring dilution should be left at "1" and manual calculation is required.

14.14.2.6. To include a command to Shut Down the pump at the end of the run: Name the row following the last sample, Pump Off under the sample heading. Use Method Shutdown.

Typical run-log:

1	Blank
2	SEQ - CCV
3	SEQ - CCB
4	SEQ - CRL1
5	SEQ - CRL2
6	Batch # - BS1
7	Batch # - BLK1
8	Sample
9	Sample @ 10X
10	Sample @ 50X
11	Sample
12	Sample
13	Sample
14	SEQ - CCV
15	SEQ - CCB
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	Sample
22	Batch #-MS1
23	Batch #-MSD1
24	Batch #- DUP1
25	Sample @ 10X
26	Sample
27	Sample
28	Sample
29	SEQ - CCV
30	SEQ - CCB
31	Sample
32	Sample
33	Sample
34	Sample
35	Sample
36	Batch #-MS2
37	Batch #-MSD2
38	Batch #- DUP2
39	Sample
40	Sample
41	Sample

42 Sample
43 SEQ - CCV
44 SEQ - CCB
45 Sample
46 Sample
47 Sample
48 CCV
49 CCB
50 PumpOff

14.14.3. Save a schedule under File/Save as, using the date as the title of the Schedule.
14.14.4. Load autosampler vials in the same order as the scheduled run.
14.14.5. Click run. The autosampler will inject into the first sample and the run should continue until completion.

15. Data Analysis and Calculations

15.1. Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.
15.2. Prepare a calibration curve for each analyte by plotting instrument response, as peak area, against standard concentration. Compute sample concentration by comparing sample response with the standard curve. If a sample has been diluted, multiply the response by the appropriate dilution factor.
15.3. Report ONLY those values that fall between the lowest and the highest calibration standards. Samples with target analyte responses exceeding the highest standard should be diluted and reanalyzed. Samples with target analytes identified but quantitated below the concentration established by the lowest calibration standard should be reported as below the minimum reporting limit (MRL).
15.4. Report results for Part A anions in mg/L.
15.5. Report NO_3^- as N

16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. See section 12.3. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed in Quality Control SOP QS03 is completed by each analyst and then provided to the group leader for further processing and approval. See SOP QS08 and Table 2 for criteria and corrective actions associated to the method performance items.

17. Pollution Prevention

17.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

17.2. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

17.3. For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria.

19. Corrective Actions and Out-of-Control Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20. Contingencies for Handling Out-of-Control or Unacceptable Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

21. Waste Management

21.1. Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.2. The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.3 from method 300.1.

22. Equipment / Instrument Maintenance

22.1. Routinely check the bed support assembly (frit), Dionex PN 42955, on the inlet side of the guard column. If discolored at all, replace with a new one.

22.2. Prime the pump if any air bubbles are noticed in the lines.

22.3. Preventative maintenance needs to be done yearly which includes the following:

22.3.1 Sample line from auto sampler to the injection part needs to be replaced

22.3.2 Pump head maintenance with maintenance Kit, Dionex PN 55639

22.3.3 Injection Valve maintenance with kit, IC: Dionex PN 55649, IC2: Dionex PN 075000, Rebuild kit for IC2: Dionex PN 075973

22.4 If routine maintenance did not bring the instrument back, more involved maintenance may be required. Notify Group Leader.

23. Computer Hardware and Software

23.1. IC2

23.1.1. Computer: Dell Optiplex 9010 running Windows 7.

23.1.2. Acquisition/Data Processing Software: Chromeleon 7.

24. Troubleshooting

If maintenance in section 22 does not bring the instrument back within requirements, discuss further actions with your Group leader.

25. References

25.1. Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Anions by Ion Chromatography", 18th Edition of Standard Methods (1992).

25.2. Dionex, System DX500 Operation and Maintenance Manual, Dionex Corporation, Sunnyvale, California 94086, 1996.

25.3. Method Detection Limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, page 1426, December, 1981.

25.4. American Society for Testing and Materials. Test Method for Anions in Water by Chemically – Suppressed Ion Chromatography D4327-91. Annual Book of Standards, Vo. 11.01 (1993).

25.5. Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B; MDL determination.

25.6. Hautman, D.P. & Bolyard, M. Analysis of Oxyhalide Disinfection By-products and other Anions of Interest in Drinking Water by Ion Chromatography. Jour. Of Chromatog., 602, (1992), 65-74.

25.7. USEPA Methods 300.0 Revision .1 August 1993; *Method for Determination of Inorganic Substances* (EPA/600/R-93/100) / *Method for the Determination of Organic and Inorganic Compounds in Drinking Water* (Vol. 1, EPA 815-R-00-014).

25.8. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update IV); Method 9056A.

25.10 DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003.) Dated 10/25/2010

25.11 DoD Quality Systems Manual for Environmental Laboratories version 5.0, 7/2013 [Based on ISO/IEC 17025:2005(E) and the NELAC Institute (TNI) Standards, Volume 1, (September 2009)].

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Table 1, DL/LOD/LOQ and recovery limits table.

26.2. Table 2, QA/QC summary table

26.3. Table 3, Analyst Data Review Checklist

Table 1 DL/LOD/LOQ and Recovery Limits

S/W	Method	Analyte	LOQ	LOD	DL	Units	LL Method	UL Method	RPD Method	LL QSM5	UL QSM5	RPD QSM5
Water	300_	Bromide	2.5	1.25	0.50	mg/L	90	110	15	90	110	15
Water	300_	Chloride	0.50	0.33	0.17	mg/L	90	110	15	90	110	15
Water	300_	Fluoride	0.25	0.10	0.033	mg/L	90	110	15	90	110	15
Water	300_	Nitrate as N	0.25	0.10	0.033	mg/L	90	110	15	90	110	15
Water	300_	Nitrite as N	0.25	0.10	0.033	mg/L	90	110	15	90	110	15
Water	300_	Sulfate as SO4	2.5	1.0	0.33	mg/L	90	110	15	90	110	15
Solid	9056A	Bromide	25	12.5	5.0	mg/Kg	80	120	15	86	116	15
Solid	9056A	Chloride	5.0	3.3	1.7	mg/Kg	80	120	15	87	115	15
Solid	9056A	Fluoride	2.50	1	0.33	mg/Kg	80	120	15	73	128	15
Solid	9056A	Nitrate as N	2.50	1	0.33	mg/Kg	80	120	15	87	111	15
Solid	9056A	Nitrite as N	2.50	1	0.33	mg/Kg	80	120	15	86	115	15
Solid	9056A	Sulfate as SO4	25.0	10	3.3	mg/Kg	80	120	15	87	115	15
Water	9056A	Bromide	2.5	1.25	0.50	mg/L	80	120	15	91	110	15
Water	9056A	Chloride	0.500	0.33	0.170	mg/L	80	120	15	87	111	15
Water	9056A	Fluoride	0.25	0.10	0.0330	mg/L	80	120	15	88	112	15
Water	9056A	Nitrate as N	0.25	0.10	0.0330	mg/L	80	120	15	88	111	15
Water	9056A	Nitrite as N	0.25	0.10	0.0330	mg/L	80	120	15	87	111	15
Water	9056A	Sulfate as SO4	2.5	1.0	0.330	mg/L	80	120	15	87	112	15

Table 2. Common Anions Analysis (Method 300.0 or 9056A)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Quarterly	Detection at established LOD			
LOQ establishment and verification	Quarterly	Recovery within LCS limits at 1x-4x established LOQ.			
Retention time (RT) window width calculated for each analyte	After method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT over a 24-hour period.	NA.	NA.	
Initial calibration (ICAL) for all analytes (minimum three standards and one calibration blank)	ICAL prior to sample analysis repeated every 6 months or as required when ICV exceeds limits.	$r \geq 0.995$. or $r2 \geq 0.990$	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Linear Calibration Range (LCR)	Every 6 months (with calibration)	All analytes within ± 10% of true value and retention times within appropriate windows at highest range.	Validate next lowest standard. Samples exceeding the LCR must be diluted to within range.		

Table 2. Common Anions Analysis (Method 300.0 or 9056/A) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration verification (ICV) (second source)	Once after each ICAL, prior to beginning a sample run.	All analytes within ± 10% of true value and retention times within appropriate windows.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte	Once per multipoint calibration.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Midrange continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All project analytes within established retention time windows. Within ± 10% of true value.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.

Table 2. Common Anions Analysis (Method 300.0 or 9056/A) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported	One per preparatory batch.	Recovery ± 20% 9056/A Recovery ± 10% EPA 300.0 For DoD QSM limits, see table 1 above.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix.	Recovery ± 20% For DoD QSM limits, see table 1 above. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table 2. Common Anions Analysis (Method 300.0 or 9056/A) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD)	One per preparatory batch per matrix (see Box D-7).	Recovery ± 20% (see above) RPD ≤ 15% (between MS and MSD). For DoD QSM limits, see table 1 above.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample duplicate (replicate)	One per every 10 samples (QSM4.2). One per every 20 samples (QSM5.0/Methods) unless MSD performed.	%D (between sample and sample duplicate) ≤ 10% QSM4.2. ≤ 15% methods and QSM5.0	Correct problem and reanalyze sample and duplicate.	Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	The data shall be evaluated to determine the source of difference.
Results reported < LOQ	NA.	NA.	NA.	Apply J-flag to all results < LOQ.	

Table 3, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):		
Sequence Number:	**Run Date:**	**Instrument ID:**
Method: EPA 300.0/SW846 9056/A	**Calibration:**	**NCR:**

QA/QC Item	Yes	No	NA	2nd Review
1. Is the calibration low standard less than or equal to the MRL/LOQ?				
2. Are the calibration fit criteria within QC limits for each anion?				
3. LCR verification: Does the highest point of the curve for each anion meet RT limits and have calculated LCR recovery within 90%-110%?				
4. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
5. Are the Continuing Calibration Verification (CCV) standards within criteria, analyzed every 10 field samples and at the end of the sequence?				
6. Are Continuing Calibration Blanks (CCB) within criteria and analyzed every 10 field samples? Do Method Blanks meet the Control Limits?				
7. Are all samples exceeding calibration range diluted and reanalyzed?				
8. Did CRL meet control limits?				
9. Did BS, Laboratory Fortified Blank or blank spike meet control limits?				
10. Did MS/MSD meet control limits? Did Duplicate meet control limits?				
11. Did you return samples back to cold storage immediately after use and fill in the logbook?				NA
12. Were samples analyzed for Nitrate (as N) and Nitrite (as N) completed within the 48-hr holding time? Other anions within 28 days?				
13. Is sample preparation information correct and complete?				
14. Were all samples filtered through a 0.45*µ*m filter?				NA
15. Are analytical results correct and complete?				
16. Is documentation complete (e.g., all anomalies in the analytical sequence have been documented, nonconformance forms are complete.)?				
17. Did you initial/date the appropriate printouts and report sheets?				
18. Are all manual integrations initialed/dated and checked by a second reviewer?				
19. Is data uploaded correctly with primary analyst/instrument correct?				
20. Are reagents/standards verified accurate on bench sheets and in LIMS?				
21. Sample(s)_____ showing full re-calculation from raw data to LIMS final concentration.				

Comments on any "No" response:

_____

_____

_____

_____

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP153 **REVISION #: 09** **EFFECTIVE DATE: 20151221**

SULFIDE by STANDARD METHODS SM 4500-S^{2-} C+F-2011 (TITRIMETRIC, IODINE) WITH SAMPLE PRETREATMENT TO REMOVE INTERFERING SUBSTANCES OR TO CONCENTRATE THE SULFIDE

APPROVALS:

Lab Director: _____ Date: **20151221**

Data Quality Manager: _____ Date: **20151221**

Group Leader: _____ Date: **20151221**
Betty Quillen

Changes Summary

SOP153_R09_20151221_SULF

- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.

SOP153_R08_20150924_SULF

- All references to supervisor updated to reflect group leader.
- Section 10 updated to reference QS04.
- Section 14 updated – review thoroughly.
- Table 3 removed.
- Data reviewers checklist updated and assigned as Table 3.

Revision 07, 20140721

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark update to include proprietary reference.
- 10.0 Directions included for making reduced volumes of reagents.
- 12.2.1 Included option to purchase BS commercially.
- 12.3 Clarified as to matrix spike preparation.
- 14.0 Review as various steps include clarifications or updated wording.

Revision 06, 20130701

- References to sample duplicates replaced with matrix spike/matrix spike duplicate indicating analysis of blank spike duplicate if insufficient sample volume received for MS/MSD.
- Sodium Thiosulfate titrant information updated to indicate quarterly standardization.
- Section 14.1.2 added to require additional Zinc Acetate to assure complete precipitation.
- Section 14.2.1 updated to clarify sample volume determination.
- Section 14.2.6 to remove reference to stir bar and include agitation of samples after each titrant addition.
- Section 14.2.7 updated to clarify determination of end point.

Revision 05, 12/21/2012

- Method reference on cover page and throughout document updated to remove EPA 376.1 and reflect standard methods as SM 4500-$S2^-$ C+F-2011.
- Section 7.2 added to clarify action if sample received with obvious sedimentation.
- Section 12.2 updated to reflect true value determination of spike standard.
- Section 12.3 updated to reflect analysis of BSD if MS/MSD volume is not available.
- Supplies updated to include 300mL glass bottle and glass fiber filter.
- Table 4 updated to reflect data reviewer checklist.

Reviewed 09/16/2011 by E. Crosby and found to need no update. Approved by Betty Quillen.

Revision 04, 09/07/10

- The SOP is an update from Revision 03 dated 05/27/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

SULFIDE by STANDARD METHODS SM 4500-S2^{-} C+F-2011 (TITRIMETRIC, IODINE) WITH SAMPLE PRETREATMENT TO REMOVE INTERFERING SUBSTANCES OR TO CONCENTRATE THE SULFIDE

1. Identification of the Test Method

This SOP is compliant with SM 4500-S2^{-} C+F-2011.

2. Applicable Matrix or matrices

This method is applicable to the measurement of total and dissolved sulfides in drinking, surface, and saline waters, domestic and industrial wastes.

3. Limits of Detection and Quantitation

SM 4500-S2^{-} C+F-2011	Units	Detection Limit	Limit of Detection	Limit of Quantitation
Sulfide using 250 mL	mg/L	0.8	2.0	4.0
Sulfide using 1 L	mg/L	0.20	0.5	1.0

4. Scope and Application, Including Parameters to be Analyzed

4.1. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

4.2. Acid insoluble sulfides are not measured by the use of this test. (Copper sulfide is the only common sulfide in this class.).

4.3. This method is suitable for the measurement of sulfide in concentrations above 1 mg/L.

5. Summary of the Test Method

Excess iodine is added to a sample which has been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back-titrated with sodium thiosulfate.

6. Definitions

6.1. Laboratory Quality System QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2. **Preparation Blank (PB)-** Laboratory reagent water that is treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the apparatus.

6.3. **Blank Spike (BS)-** An aliquot of reagent water or other blank matrices to which known quantities of the method analyte is added in the laboratory. The LCS is analyzed exactly like a

sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The LCS is given a unique identifier so that it is traceable to its source and concentration and expiration date.

6.4. **Analysis Batch-** An analysis batch is a group of twenty field samples, a preparation blank, a laboratory control sample, a matrix spike, and either a matrix spike duplicate or a sample duplicate.

6.5. **Sample Duplicate-** Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analysis of sample one and sample two indicate precision associated specifically with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.

6.6. **Matrix Spike-** An aliquot of an environmental sample to which known quantities of the method analyte is added in the laboratory. The matrix spike is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analyte in the sample matrix must be determined in a separate aliquot and the measured values in the matrix spike corrected for background concentrations.

6.7. **Method Detection Limit (MDL)--** The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

6.8. **Performance Evaluation Sample (PE)--** A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst.

7. Interference

7.1. The iodometric method suffers interferences from reducing substances that react with iodine, including thiosulfate, sulfite and various organic compounds, both solid and dissolved. Interferences due to sulfite, thiosulfate, iodide and many other soluble substances are eliminated by first precipitating ZnS in the samples, removing the supernatant, and replacing it with distilled water.

7.2. When preserved samples are received with a significant amount of solids, please discuss with the Group Leader. The PM must be informed that the sample cannot be analyzed as received. If there is sufficient unpreserved volume there is the option to filter the sample and then preserve for sulfide and proceed, reporting the result as "Soluble" Sulfide and flagged as received without preservation. This would need to be approved by the client before proceeding.

8. Safety

8.1. Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be follow lab wide.

8.2. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential heath hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

8.3. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be warn when handling samples and chemicals.

8.4. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.

8.5. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.

8.6. MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

9. Equipment and Supplies

9.1. 25 mL burette

9.2. 250 mL Bel-Arte plastic bottle or 1000 mL Bel-Arte plastic bottle

9.3. Vacuum pump

9.4. Magnetic stirrer with Teflon coated stirring bars

9.5. Buchner funnel

9.6. 500 mL Erlenmeyer flask

9.7. 300 mL Glass Bottle

9.8. Glass Fiber Filters (Whatman GF/F), or equivalent. Glass fiber filters are fragile and should be handled with care.

10. Reagents and Standards

Quality Systems SOP QS04 " TRACEABILITY AND EXPIRATION DATES OF TEST - RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

10.1. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method. All reagents shall be made from ACS reagent grade chemicals. All reagents used for distillation and analysis are entered into Element. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the samples they were used with.

10.2. A LIMS record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label.

10.3. All working standards made from reference materials shall be labeled with unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the LIMS where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in LIMS. Measurements made during standards

preparation (e.g., from weighting operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

10.4. Use DI water to prepare reagents.

10.5. Zinc acetate, 2N: dissolve 220 grams Zn $(C2H3O2)_2 \cdot 2$ H2O in 870 mL D.I. water; this makes 1 liter solution. Use same ratio of reagent to Deionized water to make a smaller volume of reagent.

10.6. Sodium Hydroxide, 6N: dissolve 240 grams of NaOH in about 600 mL of D.I. water. Dilute to 1 liter. Use same ratio of reagent to Deionized water to make a smaller volume of reagent.

10.7. Hydrochloric acid, 6N: add 250 mL concentrated HCl to 250 mL D.I. water, mix well. Use same ratio of reagent to Deionized water to make a smaller volume of reagent.

10.8. Standard iodine solution, 0.025N: dissolve 20 to 25 grams KI in a little water and add 3.2 grams iodine. After iodine has dissolved, dilute to 1000 mL in a volumetric flask. Standardize against 0.0250N Na2S03, using thyodene as indicator. **(Purchased commercially must have a COA on file for this reagent- request this COA when ordering the reagent)**

10.9. Standard sodium thiosulfate titrant solution, 0.025N: dissolve 6.205 grams Na2S203•5H20 in distilled water. Add 1.5 mL 6N NaOH or 0.4 grams solid NaOH and dilute to 1000 mL. Standardize quarterly with bi-iodate solution. **(Purchased commercially must have a COA on file for this reagent- request this COA when ordering the reagent.)**

10.10. Standard potassium bi-iodate solution, 0.025ON: dissolve 812.4 mg $KH(IO3)_2$ in distilled water and dilute to 1000 mL. Standardization of thiosulfate--dissolve approximately 2 grams KI, free from iodate, in an Erlenmeyer flask with 100 to 150 mL distilled water. Add 1 mL 6N H2SO4 or a few drops of concentrated H2SO4 and 20.00 mL standard bi-iodate solution. Dilute to 200 mL and titrate liberated iodine with thiosulfate titrant, adding 1 scoop thyodene toward end of titration, when a pale straw color is reached. When the solutions are of equal strength, 20.00 mL 0.0250N Na2S202 should be required. If not, adjust the Na2S203 solution to 0.025ON using N1V1 = N2V2 equation.

10.11. Thyodene

11. Sample Collection, Preservation, Shipment, and Storage

11.1. Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2. Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form.

11.3. Samples are taken in 250 mL Bel-Art bottles or 1000 mL Bel-Art bottles. Preserve with zinc acetate 2N and 6N NaOH. There should be no air-space in the container.

11.4. The holding time for sulfides preserved in this manner is 7 days.

12. Quality Control

12.1. Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2. An initial demonstration must be performed by each analyst performing this method. Four Blank Spikes (BS) are analyzed. See **Table 2** for acceptance criteria.

12.2.1. <u>The BS is made by diluting 33.5 mL of 5% sodium Sulfide solution to 200 mL with de-ionized water and preserving with NaOH. The true value is determined by titration using 3 mLs of this solution and diluting to 250mLs with DI water. The true value must be determined each day the standard is used. When the standard drops below half of it's first determined concentration, a new BS must be prepared. The BS can be purchased commercially.</u>

12.3. Analyze a Matrix Spike (MS), Matrix Spike Duplicate (MSD) and Blank Spike with each analytical batch. If there is insufficient sample volume to do a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) of a sample, a Blank Spike Duplicate is analyzed with the batch. **<u>Matrix spikes are prepared by adding 3 mLs of solution prepared in 12.2.1 (33.5 mL of 59. NAS diluted to 200mLs preserved with NaOH) to the sample and preserving with more zinc acetate solution.</u>**

12.4. **<u>Note: Prepare the blank, BS and BSD or MS/MSD (when there is sample volume) as early in the morning as possible on the day you are analyzing sulfides due to the amount of time it takes for the precipitate to settle out so that filtering is possible. Normally if you have a whole batch of samples you can filter the samples while the QC is settling out.</u>**
<u>MS/MSD's require extra up to 5 mLs of Zinc acetate when preparing. Zinc acetate tends to lower the pH so after adding extra Zinc acetate be sure to check the pH and make sure that the pH is > 9.0</u>

13. Calibration and Standardization

13.1. Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2. Traceability: All reference materials must be assigned an Element # and labeled accordingly. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded in the batch sheet in Element. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

14. Procedure

<u>Important</u>: Prior to analysis, check pH of sample and record pH on bench sheet to confirm correct sample aliquot has been retrieved from refrigerator.

14.1. Pretreatment

14.1.1. Mark meniscus of all Samples before adding anything else. If sample or QC has not been preserved with zinc acetate and NaOH, place 3 to 5 pasteur pipettes for 250 mL

samples or 15 mLs for 1L samples of 2N zinc acetate solution into a 250 mL/1L polyurethane bottle, fill with sample, and add 3mLs for 250 mL samples and 10 to 12 mLs for 1L samples 6N NaOH solution (check pH again after adding Zn acetate and adjust pH to >9.0. Stopper with no air bubbles under stopper and mix by rotating back and forth vigorously about a transverse axis. Vary volume of reagents added according to sample so that the resulting precipitate is not excessively bulky and settles readily. Add enough NaOH to produce a pH above 9.

14.1.2. If the samples are already preserved, add another two Pasteur pipettes of 2N zinc acetate to each sample to ensure the proper amount of precipitate is achieved if the samples are being prepared for MS/MSD or if there is no precipitate in the sample or more NaOH must be added to achieve a pH of >9.0.

14.1.3. Let precipitate settle for 1 hour for 250 mL samples and 3 hours for 1Liter samples. The treated sample is relatively stable and can be held for several hours; however, if much iron is present, oxidation may be fairly rapid. Holding time for this sample is 7 days.

14.2. Preparation and Titration of Sample:

14.2.1. **Mark meniscus of sample volume on side of bottle so you can measure the volume by adding water to the bottle to the meniscus and then measuring in a graduated cylinder.** Filter precipitate through glass fiber filter paper GF/F 9.0 cm in a Buchner funnel. Save the filter and all precipitate and discard filtered sample.

14.2.2. Measure exactly, amount of standard iodine solution (estimated to be an excess over the amount of sulfide present in the sample--usually 2 to 10 mL) into a 500 mL Erlenmeyer flask or original sample bottle. Add distilled water, if necessary, to bring volume and iodine solution to 20 mL. Note: Blank and samples normally only require 2 mLs of Iodine whereas the Iodine BS, BS, MS/MSD will require 8 mLs of Iodine.

14.2.3. Add 2 mL 6N HCl.

14.2.4. Place filter with precipitate, making sure you wipe sides of Buchner funnel to get any precipitate clinging to the sides, into bottle with iodine solution and acid. Add 200 mL D.I. water. If the sample does not stay yellow, more Iodine must be added. Record all volumes of Iodine added on the Bench Sheet.

14.2.5. Fill a 25 mL Burette with 0.0250 N sodium thiosulfate solution.

14.2.6. While titrating, agitate sample after each sodium thiosulfate addition to ensure the sample is thoroughly mixed.

14.2.7. After the sample color becomes a light yellow, add one scoop of thyodene to sample and titrate slowly with sodium thiosulfate titrant. The sample will turn a dark blue when thyodene is added. Slowly add enough titrant to turn the sample from dark blue to colorless. Record mL of titrant used. The sample will turn blue again but, the first change from blue to colorless is the endpoint.

14.2.8. Do a blank, blank spike, matrix spike and matrix spike duplicate (see note in 12.3 above) a blank is done with each batch of samples taking D.I. water through the entire procedure the same as the samples including pretreatment. The detection limit is 1.0 mg/L sulfide when using 250 mL of sample and 0.20 mg/L when using 1 Liter.

14.2.9. Discard titrated sample.

15. Data Analysis and Calculations

15.1. Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2. One milliliter 0.0250N iodine solution reacts with 0.4 mg S2-:

$$\text{Mg S2-/L} = \frac{(AXB) - (CXD)}{\text{Sample}} \text{ x } 16000 \text{ x (Ratio of final to mL initial volume)}$$

where:

A	=	mL standard iodine solution
B	=	normality of iodine solution
C	=	mL Na2S2O3 solution
D	=	normality of Na2S2O3 solution
Final volume	=	200 mL
Initial volume	=	measured volume of original sample

16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality Manual is completed by each analyst and then provided to the Group Leader for further processing and approval. Four LCS's are reported with a precision and accuracy sheet for the DOC. See section 12.2 for LCS preparation.

See Table 2 for criteria and corrective actions associated to the following method performance items:

16.1 Method Detection Limit Study or Detection Limit Determination
16.2 Limit of Detection Verification
16.3 Limit of Quantitation or Reporting Limit Verification
16.4 Demonstration of Capability (DOC)
16.5 PT Studies

17. Pollution Prevention

17.1. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

17.2. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option. Quantity of chemicals purchased should

be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

17.3. For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

18.1. Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria.

18.2. If the LCS exceeds table 2 criteria, make fresh sulfide standard and try again. If it still does not work, then contact Group Leader.

19. Corrective Actions and Out-of-Control Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20. Contingencies for Handling Out-of-Control or Unacceptable Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

21. Waste Management

21.1. Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

21.2 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society.

22. Equipment / Instrument Maintenance

25 mL burette, clean and rinse with DI water after finished, store upside down.

23. Computer Hardware and Software

Not applicable.

24. Troubleshooting

24.1. There's only enough sample for one attempt.

24.2. Try remaking the sulfide working standard and then remaking the BS if the BS fails.

24.3. If there are significant solids in the sample, contact supervisior before attempting analysis.

25. References

25.1. Standard Methods 22nd Edition SM 4500-$S2^-$ C+F-2011.

25.2. DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003.) Dated 10/25/2010

25.3. DoD Quality Systems Manual for Environmental Laboratories version 5.0, 07/2013 [(Based on ISO/IEC 17025:2005(E) and the NELAC Institute (TNI) Standards, Volume 1, (September 2009)]. July 2013

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Table 1, DL/LOD/LOQ table included in section 3.0

26.2. Table 2, Method Quality Control Requirements Summary.

26.3. Table 3, Data Reviewers Checklist

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
BLK	One per prep batch	• Must be less than the detection limit.	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
BS	One per prep batch	80 to 120% recovery	• Follow guidelines from SOP QS05
BSD	One per prep batch, when MS/MSD not included.	80 to 120% recovery	• Follow guidelines from SOP QS05
MS	One per prep batch, if sample volume available.	75 to 125% recovery	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	75 to 125% recovery RPD ≤ 20%	• Follow guidelines from SOP QS05
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between 80-120%, with a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	Once per year to meet specific state requirements.	o See QS09 for specifications	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

Table 3, Data Reviewers Checklist (Prior to approving data)

Sulfide Data 2nd check

- ❑ Analyst authorization
- ❑ All general information complete
- ❑ Base MDL
- ❑ Correct Units
- ❑ Corrections crossed out & initialed
- ❑ Titrant age ok
- ❑ BLK, BS per day & per 20 samples & in control
- ❑ 10% of calculations checked
- ❑ BSD done when no MS/MSD volume provided
- ❑ Holding time met
- ❑ Problems discussed with manager
- ❑ Necessary NCR attached
- ❑ Data in LIMS and verified back to bench sheet
- ❑ All Reagents used have been assigned Element #'s that have been entered in the bench sheet
- ❑ Reagents/Standards verified accurate on bench sheets and in LIMS
- ❑ All initial volumes have been updated in the bench sheet including for the QC
- ❑ All dates are correct in the bench sheet
- ❑ Additional information needed for reports:

2nd checked by/date:

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP 154 **REVISION #: 13** **EFFECTIVE DATE: 20161121**

TOTAL ALKALINITY, CARBONATE, BICARBONATE and CARBON DIOXIDE (calculation)

METHOD: SM 2320 B-2011 and SM 4500-CO2 D-2011

APPROVALS:

Lab Director: _____ Date: **20161121**

Data Quality Manager: _____ Date: **20161121**

Group Leader: _____ interim Date: **20161121**

Changes Summary

SOP154_R13_20161121_ALK

- Standardization of titrants updated in sections 10.3 and 10.4 to indicate documentation in alkalinity titrant standardization log.

SOP154_R12_20151221_ALK

- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.

SOP154_R11_20150921_ALK

- All references to supervisor updated to reflect group leader.
- Section 10 updated to reference QS04
- Section 14.1 updated to record time of each sample.
- Section 12 updated for worksheet location
- Table 3 removed and Data Reviewer Checklist made to reflect Table 3 with updates.

Revision 10, 07/22/14

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark update to include proprietary reference.
- Section 8.0 updated to include standardized references including MSDS sheet reference updated to reflect vendor website access.
- Sections 10.3 and 10.4 updated to indicate standardization monthly.
- Section 10.4 updated to include option to purchase reagent from a vendor.
- Section 13.2 added to direct user to reagent section for standardization procedure.
- Section 14.2 added air pocket warning.
- Sections 14.5.1.7, 14.1.5.1.8, 14.14.1, 14.14.2 procedure updated.
- Section 11 updated to reference SOP QS10.
- Sections 22.0, 23.0 and 24.0 added/populated.
- Table 4 Data Reviewer checklist updated.
- CO2 calculation from alkalinity by SM 4500-CO2 D-2011 added (section 15 and 25)

Revision 09, 04/10/13

- Removed references to EPA method 310.1 and updated standard methods reference format
- Section 7.3 updated to reflect all known interference possibilities.
- Section 9.5 updated to add Temperature probe.
- Sections 10.3 and 10.4 updated to add mg CaCO3 equivalents.
- Reference to duplicate removed and replaced with MS/MSD references in section 12.3.
- Spreadsheet directory updated in section 12.4.
- Calibration section 14.5 updated.
- Wording of section 14.6 updated.

Revision 08, 09/16/11

- Revised pH probe to pH and temperature probe in Equipment and Supplies section.

Revision 07, 09/21/10

- Incorrect reference to GC/FID in section 1.1 was updated to titration.
- References to preparation blanks as PB were updated to BLK and references to laboratory control samples were updated to blank spikes/blank spike duplicates as BS/BSD.

- Magnetic stirrer and stir bars were added to equipment list.
- Units were updated from mg/L to mg CaCO3/L.
- Table 2 criteria were updated for method specifics.

Revision 06, 09/07/10

- The SOP is an update from Revision 05 dated 05/27/2009
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

TOTAL ALKALINITY, CARBONATE, BICARBONATE and CARBON DIOXIDE (Calculation) METHOD: SM 2320 B-2011 and SM4 4500-CO2 D-2011

1. **Identification of the Test Method**
   1.1. The titration method is used to analyze TOTAL ALKALINITY, CARBONATE, BICARBONATE, utilizing Standard Methods 2320B and CARBON DIOXIDE (calculation) using SM 4500-CO2 D-2011.

2. **Applicable Matrix or Matrices**
   2.1. This method is applicable to drinking, surface, and saline waters, and domestic and industrial wastes. Soils are leached 10 grams to 100 mLs and the analysis performed on the leachate.
   2.2. The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 10 mL.

3. **Limits of Detection and Quantitation**
   3.1. The detection limit is 1.0 mg/L $CaCO_3$. The limit of quantitation is the same.

4. **Scope of Application, Including Components to Be Analyzed**
   4.1. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

5. **Summary of the Test Method**
   5.1. When the sample is being analyzed for phenolthalein alkalinity, carbonate, bicarbonate and total alkalinity, an unaltered sample is titrated to an electrometrically-determined endpoint of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way. The sample is then titrated to a pH exactly 0.3 pH units lower and the calculation for 2320B (in calculation section of this SOP) is used to calculate the samples for phenolthalein alkalinity, total alkalinity, carbonate and bicarbonate results. See 14.14.3 for samples with pH greater than 8.3.

6. **Definitions**
   6.1. Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7. **Interferences**
   7.1. Substances, such as salts or weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.
   7.2. For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in *Annual Book of ASTM Standards*, Part 31, "Water," p. 115, D-1067, Method D (1976).
   7.3. Soaps, oily matter, suspended solids, or precipitates may coat the pH electrode, and cause a sluggish response. Allow additional time between titrant additions to let electrode come to equilibrium or clean the electrode occasionally. Do not filter, dilute, concentrate, or alter sample.

8. **Safety**
   8.1. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab wide.
   8.2. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.3. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential heath hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

8.4. MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your section group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9. **Equipment & Supplies**

9.1. pH meter that uses a glass electrode and can be read to 0.01 pH units. The analyst will note on the data which pH meter (Orion 420A) is used.

9.2. 50 mL disposable beakers with wide enough mouths to allow room for burette tip and pH and temperature probe.

9.3. 10 mL Class A microburette.

9.4. Magnetic Stirrer and tiny stir bars.

9.5. Temperature probe.

10. **Reagents and Standards**

Quality Systems SOP QS04 " TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

10.1. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

- ACS reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- Certified stock standards and reagents are purchased from Ultra Scientific, NSI, Ricca and other vendors depending on their availability. The date they are received is noted on the label or container they are received in, on the certificate of analysis (COA) and in the LIMS system. They are given a sequential number in the LIMS system which is also noted on the container and the COA which is then scanned and stored in the LIMS system. The date the standards are opened is recorded in the LIMS system. All stock standards are stored at 4°C.

10.2. Sodium carbonate solution, approximately 0.05N: Place 2.5 $\pm$0.2 g (to nearest mg) Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1-liter, Class A, volumetric flask and dilute to the mark. The Na_2CO_3 solution must be disposed of after one week.

10.3. Standard Acid (sulfuric or hydrochloric), 0.1N (high titrant): Dilute 3.0 mL concentrated H_2SO_4 or 8.3 mL concentrated HCl to 1 liter with distilled water.

- Standardize upon makeup and then monthly by potentiometric titration. Dilute 40 mL of 0.05N Na2CO3 solution to 100 mL with deionized water and titrate potentiometrically with the Standard Acid to a pH of about 5. Lift electrode and rinse into beaker. Boil solution gently for 3 to 5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH Inflection point (3 units lower). 1 mL 0.1000N solution is equal to 5.00 mg CaCO3. Calculate normality using the following equation and document information in the titrant standardization log.

$$N = \frac{A \times B}{53.00 \times C}$$

where: A = g Na_2CO_3 weighed into 1 liter
B = mL Na_2CO_3 solution
C = mL acid used to inflection point

10.4. Standard Acid (sulfuric or hydrochloric), 0.02N (low titrant): Dilute 200.0 mL of 0.1000 N Standard Acid to 1 liter with distilled water. This reagent may also be purchased from an approved vendor such as Fisher or Tennessee Reagents. When the titrant is received, an Element number is assigned to it. A Certificate of Analysis (COA) should have been requested when placing the order for the titrant but may also be retrieved off the vendor website. The LIMS ID assigned to that titrant is written on the COA and the COA is submitted for scanning in the copier room.

- Standardize upon makeup for prepared solution and re-standardize prepared or purchased standards monthly by potentiometric titration of 15.0 mL 0.05N Na_2CO_3 solution with the Standard Acid to a pH of about 5. Lift electrode and rinse into beaker. Boil solution gently for 3 to 5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH Inflection point (3 units lower). 1 mL 0.020N solution is equal to 1.00 mg CaCO3 - calculate normality as above. Record standardization information in the titrant standardization log and enter the new date, standard ID, normality in the Excel worksheet.

11. **Sample Collection, Preservation, Shipment, and Storage**

11.1. No preservation necessary except to keep chilled to 4°C until sample is analyzed. Do not open sample bottle until analysis.

11.2. The holding time for these samples is 14 days.

12. **Quality Control**

12.1. Run a blank spike (BS) for each batch of samples (maximum of 20 samples per day). If the BS does not fall in the range of 80 to 120%, corrective action must be taken to find the problem and correct it.

12.2. Run a preparation blank (BLK) for each batch of samples (maximum of 20 samples per day). The BLK should be less than the reporting limit.

12.3. Analyze a matrix spike (MS) and matrix spike duplicate (MSD) every 20 samples. The percent recoveries on a MS and MSD should be within 75 to 125% and the relative percent difference (RPD) on duplicates should be less than 20%.

12.4. Analyze a duplicate (DUP) every 20 samples. The relative percent difference (RPD) on duplicates should be less than 20%.

12.5. The Excel file for calculations is located at: "V:\Standard Operating Procedures\Current SOP File Directory\Worksheets" or can be accessed through TOC_SOPs_Controlled_Documents spreadsheet at V:\Standard Operating Procedures.

12.6. Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\%\ \text{Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

13. **Calibration and Standardization**
    13.1. Quality Systems **SOP QS08** "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.
    13.2. See reagents to find procedure on standardizing the titrant.

14. **Procedure**
    14.1. Write down time each sample is measured.
    14.2. Fill 10 mL microburette with Standardized Acid. Be sure to look for air pockets in the tip below the stopcock and make sure that you get the air pockets out before starting the titration.
    14.3. Pick titrant according to estimated total alkalinity. For example, a drinking water or groundwater sample would probably use the 0.020 N titrant and a wastewater sample would probably use the 0.10 N titrant. Historical data is very useful for this.
    - A sample size of 25 mL is usually appropriate. If you use less than 1 mL of your high titrant, then you must titrate a new sample using low titrant. Using less than 1 mL of your low titrant is valid. When the samples are soils a 12 gram portion diluted to 120 mLs is used.

    14.4. Sample size should be such that a sufficiently large volume of titrant is used (1 to 10 mL titrant).
    14.5. Standardize and calibrate the pH meter according to laboratory procedures below. Using the Orion 420A pH meter. The calibration buffer readings must be within ±0.05 pH units of the true value. If not, recalibration is necessary. Record this information in the pH log book. If automatic temperature compensation is not provided, make titration at 25 ±2°C. Check the buffer every 3 hours after calibration. The reading should be within ±0.20 pH units.
    - Initial calibration (2 point) The Orion 420A meter is capable of both auto-calibration and manual calibration. We calibrate manually.

    14.5..1. Turn the power on. It will display a quick check mode and then the reading will stabilize. The meter should be in the pH mode, if not, press the "mode" key until the pH mode indicator is displayed.
    14.5..2. Uncover the hole on the upper part of the electrode (If electrode is equipped with one). This allows a uniform flow of the filling solution. (It should be kept covered when not in use.)
    14.5..3. Rinse the electrode with deionized water and blot dry with a kimwipe (do not wipe the electrode).
    14.5..4. The meter must be calibrated prior to use with two pH buffer standards (4 and 7). Place both the ATC and the pH electrode in pH 7 buffer first, with stirring bar turning. Make sure the electrodes are not touching the bottom or sides of the beaker. Buffers must be freshly poured up daily from their 2 to 4 Liter storage containers into disposable beakers and be at room temperature.
    14.5..5. Press the "2nd" key, followed by the "cal" key. It will display the time and date of the last calibration and then "P1" will be displayed in the lower field -- this means it is ready to read the first calibration standard.
    14.5..6. Wait for a stable pH display and the meter will say "ready". Press the "no" key. The first digit will start flashing. Scroll up or down using the arrow-head keys until the correct value appears in the first digit (it will be 7 for the 7.00 buffer) then press "yes". Continue in the same manner until all the digits have been correctly entered, then press "yes" to enter the new value. The display will remain frozen for a few seconds, then "P2" will be displayed which means that the meter is ready to read the second calibration standard.
    14.5..7. Rinse the electrode and place in the pH 4 buffer with stirring bar turning. Wait for a stable pH reading and then enter the correct value as you did for the 7 buffer. Once the P3 comes up, then press the measure print key and the slope will be displayed in the main field. lt will come up quickly and then disappear, so you have to be watching the display to make sure you don't miss it.
    14.5..8. The slope should be between 92 and 102. (Record the slope in the calibration logbook and on the data sheet.) If it is not within this boundary you should inspect the electrode

and meter and recalibrate with fresh buffers. If the slope is still out of range, check the manual for troubleshooting. Also record the temperature in the log book.

14.5..9. The meter will automatically advance to the "measure" mode.

14.6. Carefully pour 25 mLs into disposable beaker by gently pouring down the side of the vessel so as to have the least aeration to sample as possible. Place a small magnetic stirring bar in vessel and start magnetic stirrer at medium to slow stirring.

- Note 1: When soils are being analyzed 12 grams is weighed into a 120 mL bottle and diluted to 120 mLs. Place in the shaker for one hour. Mix the sample well and use 25 mLs to analyze. Make sure you get a representative sample for analysis.
- Note 2: Where sample volume is adequate and low titrant is used (alkalinity concentration is less than 20 mg CaCO3/L), a sample volume of 100 to 200 mL should be used and titration should be performed using a 10 mL microburette.

14.7. Place pH probe (which has been rinsed with DI water and patted dry with a Kimwipe) in the sample such that the probe tip is not touching the sides or bottom of the flask or beaker. If the probe has a protective cover, this is not a consideration.

14.8. Make sure there are no air bubbles at the bottom of filled burette. Wipe tip of burette so that no extra drops are clinging to it. Place tip of burette into mouth of vessel so that it is above the surface of the sample but is not touching the sides of the flask and drops can go nowhere but into the sample (e.g., drops from burette are not going onto pH probe or walls of flask but directly into sample).

14.9. Titrate a blank and an LCS first. This will let you know that the normalities of titrant are correct. If the result is out of the acceptable range of the LCS, run a duplicate LCS. If still out of range, find another second source and if still incorrect, restandardize titrant. First double-check titrant normality.

14.10. Record sample pH after reading is stable for 5 to 10 seconds.

14.11. Titrate sample to pH 4.5. This must be done slowly so as not to miss the exact pH. Record titrant volume.

14.12. The minimum titrant volume to be employed using high titrant is 1 mL. If high titrant is being used, go to low titrant; if low titrant doesn't work, use more sample. Be aware of sample volume that may be needed for other analyses. Do not dilute.

14.13. When titrating the sample, be sure to allow time for the pH to equilibrate so that the inflection point will not be passed.

14.14. Potentiometric titration of low alkalinity

- For alkalinity of < 20 mg CaCO3/L titrate 100 – 200 mL as above using a 10 mL microburet and 0.02 N acid solution. Be sure that sample volume is adequate for all tests being run on the unpreserved aliquot before using 100 to 200 mLs.
- Stop titration at pH in range of 4.3-4.7 (generally 4.5), record volume and exact pH. Very carefully add titrant to lower pH exactly 0.3 pH units and record volume.
- If the pH of the sample is above 8.3, the sample needs to be titrated for phenolthalein alkalinity, first check original pH. If it is not above 8.3, the phenolthalein result will be below the minimum detection limit. If the original pH is above 8.3, titrate sample as in above procedure but down to 8.3 instead of 4.5, and record titrant volume in box A on the alkalinity bench sheet. Then proceed with the regular procedure titrating to pH 4.5, and recording this result in box B on the alkalinity bench sheet, then carefully titrate exactly 0.3 pH units lower to pH 4.2 and record titrant volume at this level, in box C on the alkalinity bench sheet.

14.15. Potentiometric titration of high alkalinity: Use a sufficiently large volume of titrant (>20 mL in a 50 mL buret) to obtain good precision while keeping volume low enough to permit sharp endpoint.

- For >1000 mg $CaCO_3$/L use 0.1 N titrant

14.15..1. For alkalinity of > 1000 mg $CaCO_3$/L, titrate 25 – 50 mL as above using a 50 mL burette and 0.10 N acid solution.

14.15..2. Stop titration at pH in range of 4.5, record volume and exact pH. See note above.

15. **Data Analysis and Calculations**

15.1. Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2. The detection limit is 1.0 mg/L $CaCO_3$.

15.3. Potentiometric titration to pH 4.5 (high alkalinity)

Total Alkalinity mg/ L $CaCO_3$ = $\frac{A \times N \times 50{,}000}{mL \text{ of Sample}}$

Where: A = mL Standard Acid to pH 4.5
N = Normality Standard Acid

Potentiometric titration of low alkalinity = T below

15.4. **Method 2320B calculations**

Use the following notation in below calculations:

P = Phenolthalein alkalinity
T = Total alkalinity

$P = \frac{A \times N \times 50{,}000}{mL \text{ of Sample}}$

$T = \frac{(2B-C) \times N \times 50{,}000}{mL \text{ of Sample}}$

If	P	= 0	Carbonate	= 0	Bicarbonate	= T
If	P	< 1/2T	Carbonate	= 2P	Bicarbonate	= T-2P
If	P	= 1/2T	Carbonate	= 2P	Bicarbonate	= 0
If	P	> 1/2T	Carbonate	= 2(T-P)	Bicarbonate	= 0
If	P	= T	Carbonate	= 0	Bicarbonate	= 0

Where: A = mL titrant to pH 8.3
B = mL titrant to pH 4.5
C = mL titrant to pH 4.2
N = normality Standard Acid

15.5 **Method 4500 calculations – CO2 D** (Free carbon dioxide)

Mg CO2/L = 2.0 x B x 10(6-pH)

Where: B = bicarbonate alkalinity, from A.

E. Total carbon dioxide:

Mg total CO2/L = A + 0.44 (2B + C)

Where: A = mg free CO2/L
B = bicarbonate alkalinity from A, and
C = carbonate alkalinity from B

16. **Method Performance**

16.1. Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples. The data is calculated for accuracy and precision requirements (see group leader for the form to be used). The DOC form, as listed in SOP QS03 is completed by each analyst and then provided to

the group leader for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM, DOD limits will be used.**

16.2. Forty analysts in seventeen laboratories analyzed synthetic water samples containing increments of bicarbonate, with the following results:

Increment as Alkalinity mg/L, CaCO3	Precision as Standard Deviation mg/L, CaCO3	Accuracy as Bias, %	Accuracy as Bias, mg/L, CaCO3
8	1.27	+10.61	+0.85
9	1.14	+22.29	+2.0
113	5.28	-8.19	-9.3
119	5.36	-7.42	-8.8

17. **Pollution Prevention**

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability. Please see Waste Disposal SOP QS14, for instruction of proper disposal of waste generated from this area.

18. **Data Assessment and Acceptance Criteria for Quality Control Measures**

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. **Corrective Actions and Out-of-Control Data**

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20. **Contingencies for Handling Out-of-Control or Unacceptable Data**

20.1. Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.2. If the preparation blank is higher than the reporting limit, all samples less than ten times the concentration of the blank must be reanalyzed.

20.3. If the blank spike (BS) is out of the range of 80 to 120%, and the % recovery is high (higher than 120%), only sample concentrations less than the reporting limit are acceptable data. Otherwise, all data with concentrations above the method detection limit must be reanalyzed with an BS in the range of 80 to 120%. If the BS is low (less than 80%), all samples must be reanalyzed.

21. **Waste Management**

Please see Waste Disposal SOP QS14, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

22. **Equipment / Instrument Maintenance**

22.1. The burette must be kept clean and without chips to the tip.

22.2. pH probes that have a hole at the top of the barrel must not be allowed to go dry. Keep the hole covered when the probe is not in use. When the filling solution is low refill with the appropriate filling solution for that type of probe.

22.3. pH probes must be kept clean. Oils and greases will coat the probe and cause problems with accurate readings.

23. **Computer Hardware and Software**
    23.1. Not applicable

24. **Troubleshooting**
    24.1. When the burette starts to deliver dropwise when the stopcock is completely open, the burette is clogged and must be cleaned before continuing with the titration .
    24.2. If the pH meter is acting sluggish and not producing a steady reading for several minutes, look at the probe closely. Try cleaning the probe with methanol to remove oils and greases that may have adhered to the bulb. Then recalibrate the probe.

25. **References**

    25.1. Standard Methods for the Examination of Water and Wastewater 22nd Edition, method 2320B, editorial revisions -2011 (SM 2320 B-2011, SM 4500-CO2 D-2011).
    25.2 DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003.) Dated 10/25/2010
    25.3 Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories. Based on ISO/IEC 17025:2005(E) and The NELAC Institute (TNI) Standards, Volume 1, (September 2009) Dated July 2013
    25.2. Dye, J.F. 1958. Correlation of the two principle methods of calculating the three kinds of alkalinity. Journal AWWA, 50:812.

26. **Tables, Diagrams, Flowcharts and Validation Data**
    26.1. Table 1, DL/LOD/LOQ
    26.2. Table 2, for all technical methods, should always be the QA/QC summary table.
    26.3. Table 3, Technical Completeness / Accuracy Checklist
    26.4. Table 4, Data Reviewers Checklist

TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard (Units mg CaCO3/L)		
Parameter	**RL**	**LowCal**
TOTAL ALKALINITY , CARBONATE, BICARBONATE	1.0	NA

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
BLK	One per prep batch	• Must be less than the LOD	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
BS	One per prep batch	80 to 120%	• Follow guidelines from SOP QS05
BSD	One per prep batch, when MS/MSD not included.	80 to 120% and 20% RPD	• Follow guidelines from SOP QS05
MS	One per prep batch, if sample volume available.	75 to 125%	• Follow guidelines from SOP QS05
MSD/DUP	One each per prep batch, if sample volume available.	75 to 125% and 20% RPD/20% RPD	• Follow guidelines from SOP QS05
DOC Study	• Initially per analyst prior to reporting data • Annually	• Average percent recovery should be between 80-120%, with a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	Not required for this analyte	• NA	• NA

Table 3, Data Reviewers Checklist (Prior to approving data)

Alkalinity, Carbonate, Bicarbonate, CO2 **Data 2nd check** ❑ Analyst authorization ❑ All general information complete ❑ Correct Units ❑ Corrections crossed out & initialed ❑ BLK/BS per day & per 20 samples & in control ❑ TV's & sources indicated ❑ Titrant standardized monthly ❑ Titrant normality recorded ❑ 10% check of calculation ❑ MS/MSD per 20 samples ❑ DUP per 20 samples ❑ Buffer checked every 3 hours and within criteria ❑ Samples noted when <DL ❑ BS prep. indicated &/or calculation shown ❑ Holding time met ❑ Data entered in LIMs correctly ❑ Batch sheet completed accurately ❑ Reagents/Standards verified on bench sheets and in LIMS ❑ Problems discussed with manager ❑ Necessary NCR's attached ❑ Additional information needed for reports:
2nd checked by/date:

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP 186 **REVISION #: 11** **EFFECTIVE DATE: 20151221**

FILTERABLE RESIDUE, TOTAL DISSOLVED SOLIDS & TOTAL DISSOLVED VOLATILE SOLIDS VIA SM 2540 C-2011 (GRAVIMETRIC DRIED AT 180^{O}C)

APPROVALS:

Lab Director: _____ Date: **20151221**

Data Quality Manager: _____ Date: **20151221**

Group Leader: _____ Date: **20151221**
Betty Quillen

Reviewed 20170311 by Rick Davis and found to need no revisions.

Changes Summary

SOP186_R11_20151221_code

- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.

SOP186_R10_20150921_code

- All references to supervisor updated to reflect group leader.
- Section 10 updated to reference QS04.
- Table 1 added.
- Table 3 removed and Data Reviewers Checklist renamed Table 3 with updates.

Revision 09, 20140730

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark update to include proprietary reference.
- MSDS sheet reference updated to reflect vendor website access.
- Sections 3.0, 6.6, 10.2, 14.1.2, 14.1.3, 14.4.1, 22-26 updated.

Revision 08, 20130701

- Updated standard method reference to reflect "-2011" and verified SOP includes appropriate QC references.
- Updated method blank abbreviation to BLK in section 5 and throughout SOP. Changed references to laboratory control sample (LCS) to Blank Spike (BS).
- Removed reference to MDL study in QC summary table 2 and throughout the SOP.

Revision 07, 20120820

- Standard methods reference format updated.
- Constant weight criteria added.
- Data Review Checklist added.

Revision 06, 20110516

- The SOP is an update from Revision 05 dated 09/07/10
- Added requirement to record oven temperature and Date/Time when placing samples in and taking samples out of oven.
- Updated Table 2 to reflect duplicate every 10 samples with 5% RPD criteria (as was indicated in body of SOP)

Revision 05, 09/07/10

- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

FILTERABLE RESIDUE, TOTAL DISSOLVED SOLIDS & TOTAL DISSOLVED VOLATILE SOLIDS VIA SM 2540 C-2011 (GRAVIMETRIC DRIED AT 180^{O}C)

1. Identification of the Test Method

This SOP is compliant with Standard Methods 2540 C-2011 Gravimetric, Dried at 180°C.

2. Applicable Matrix or matrices

This SOP is applicable to – drinking, surface, and saline waters, domestic and industrial wastes.

3. Limits of Detection and Quantitation

TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard (Units mg/L)		
Parameter	**LOQ**	**LowCal**
Total Dissolved Solids (TDS)	20	NA
Total Dissolved Volatile Solids (TDVS)	20	NA

4. Scope and Application, Including Parameters to be Analyzed

Each parameter that is analyzed and reported under the scope of this SOP is listed in Table 1 above.

5. Summary of the Test Method

5.1. A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated at 104°C and dried to constant weight at 180°C.

5.2. If residue, non-filterable (TSS) is being determined, the filtrate from that method may be used for residue, filterable.

6. Definitions

6.1. Laboratory Quality System QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2. **Filterable residue-** is defined as those solids capable of passing through a glass fiber filter and dried to a constant weight at 180°C.

6.3. **Preparation Blank (BLK) -** Laboratory reagent water that is treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the apparatus.

6.4. **Sample Duplicate-** Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analysis of sample one and sample two indicate precision associated specifically with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.

6.5. **Performance Evaluation Sample (PE) --** A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are

used to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst.

6.6. Blank Spike – An aliquot of reagent water or other blank matrices to which known quantities of the method analyte is added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The LCS is given a unique identifier so that it is traceable to its source, concentration and expiration date.

7. Interference

7.1. Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

7.2. Samples containing high concentrations of bicarbonate will require careful and possible prolonged drying at 180°C to ensure that all the bicarbonate is converted to carbonate.

7.3. Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

8. Safety

8.1. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be follow lab wide.

8.2. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of nitrile gloves and lab coats is highly recommended.

8.3. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.4. MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9. Equipment and Supplies

9.1. Glass fiber filter discs 90 cm, without organic binder Whatman 934-AH or equivalent.

9.2. Filtering flask, 500 mL or more.

9.3. Buchner funnels 90 cm, porcelain made by Coors or 1.5 micron filter disks and syringes.

9.4. 100 to 150 mL porcelain crucibles, low wide-form or 150 mL beakers.

9.5. Drying oven, 104°C ± 2°C.

9.6. Drying oven, 180°C ± 2°C.

9.7. Desiccator

9.8. Analytical Balance

9.9. Vacuum pump

9.9.1. **NOTE:** Regular maintenance must be done on the vacuum pump periodically. Always check oil holders to make sure oil is in it up to the red fill line before using. Make sure that reservoir is hooked up in line in case water filters over. This reservoir should be hooked up such that any water coming over will be collected in a flask so it will not go directly into the pump itself. This reservoir should be dumped and cleaned out periodically.

10. Reagents and Standards

Quality Systems SOP QS04 " TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

10.1. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method. All reagents shall be made from ACS reagent grade chemicals. All reagents used for distillation and analysis are entered into Element. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the samples they were used with.

10.2. Blank Spike (BS): The BS is Purchased commercially. A COA is scanned and located at the Element number assigned under Standards in Element.

11. Sample Collection, Preservation, Shipment, and Storage

11.1. Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2. Use resistant glass or plastic bottles provided that the material in suspension does not adhere to container walls. Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate sample at 4°C up to time of analysis to minimize microbiological decomposition of solids.

11.3. In no case hold samples more than 7 days. Bring samples to room temperature before analysis.

12. Quality Control

12.1. Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2. Do a duplicate every tenth sample. Duplicates should agree within 5% of each other.

12.3. Do a total dissolved solids BS with each batch of samples to check accuracy of analytical balance and drying ovens. If the first weighing result is within specification limits, more weighings are not necessary.

13. Calibration and Standardization

13.1. Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization. The detection limit for TDS is 20 mg/L.

13.2. Calibrate analytical balance daily or as used by weighing a 50 gram and an 80 gram weight to check calibration. Record weight results.

13.3. Calibrate analytical balance monthly by checking the whole range of weights in reference standards set.

14. Procedure

14.1. Crucible / Beaker Preparation

14.1.1. Wash 100 to 150 mL crucibles or beakers in hot soapy water, rinse with tap water and then thoroughly inside and out with D. I. water, place in 180°C oven until needed. Make sure that desiccant in desiccator is placed in 105°C oven overnight to dry when the desiccant under-goes a color change. **Step down from 180°C to 104°C before put in desiccator.**

14.1.2. On the day TDSs are to be analyzed, place beakers or crucibles in desiccator for 30 minutes then get a tare weight and record. After weighing, handle crucible with tongs only.

14.1.3. Place a 9.0 CM, 11.0 CM or 12.5 CM 935-AH glass fiber filter, wrinkled surface up, in a clean buchner-funnel (depending on size buchner funnel available). Place buchner funnel in a clean, dry filtering flask and apply vacuum, wash the filter with three successive 50 mL volumes of de-ionized water. Remove all traces of water by continuing to apply vacuum after water has passed through, discard washings. This rinses the filter and also allows the filter to be well seated in the Buchner funnel before the sample is filtered. **Alternately, use a purchased filter disk with a 25 mL syringe to filter samples into the cleaned and tared crucible and/or beaker.After the correct volume of sample, get 10 mLs of DI water and rinse through filter.**

14.2. Determination of Sample volume

14.2.1. The following criteria are used to determine the volume of sample to be used for analysis: According to New Jersey DEP the difference in the weight of the crucible versus the weight of sample in mg cannot be greater than 300 mg. When the difference is 200 mg or more a smaller volume of sample must be used for analysis. Therefore, on all samples which require TDS, when no prior knowledge of the sample is available the conductivity of the sample must be checked and the volume used for analysis adjusted as follows:

CONDUCTIVITY (µmho/cm)	**TDS VOLUME (mL)**
0 to 2750	100
2750 to 5500	50
5500 to 11000	25
11000 to 27000	10
27000 to 55000	5
55000 to 270000	1

If the conductivity is over 270000 please inform your group leader.

14.3. Total Filterable Solids

14.3.1. Place buchner funnel in a clean, dry filtering flask, begin suction. Shake sample vigorously so that a representative sample may be poured off. Rapidly pour 100 mL of sample by means of a 100 mL graduated cylinder onto prepared filter.

14.3.2. Filter the sample through the glass fiber filter, rinse three times with de-ionized water, continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

14.3.3. Transfer entire volume of filtrate in filtering flask to a weighed crucible and evaporate to dryness in a 104°C ±2 oven. This normally requires 24 hours, or overnight.

14.3.4. Dry the evaporated sample for at least one hour at 180°C ±2°C. **Step down to 104°C 1 hour before cooling.** Cool in a desiccator and weigh. Repeat heating, drying and cooling cycle until a constant weight is obtained (<4% difference from last weight) or until weight loss is less than 0.50 mg. Oven temperature and Date/Time must be recorded when placing samples in and taking samples out of oven.

14.4. Total Residual Filterable Volatile Solids (TDVS)

14.4.1. After total filterable solids are run, place crucible used for this purpose in 550 ±50°C furnace for 15 minutes. Step down to 104°C before putting in desicator. Allow crucible to cool in desiccator for 1 hour and weigh. Repeat heating, drying and cooling cycle until a constant weight is obtained (<4% difference from last weight) or until weight loss is less than 0.50 mg. Oven temperature and Date/Time must be recorded when placing samples in and taking samples out of oven.

15. Data Analysis and Calculations

15.1. Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2. Calculate filterable reside as follows:

$$\text{TDS mg/L} = \frac{(A-B)1000}{\left(\frac{C}{1000}\right)}$$

Where: A = weight of dried residue + dish in grams
B = weight of dish in grams
C = volume of sample used in mL.

15.3. Calculate filterable volatile residue as follows:

$$\text{TDVS mg/L} = \frac{(A-B)1000}{\frac{C}{1000}}$$

Where: A = weight of dried residue + dish in grams.
B = weight of dried residue at 550°C ±50°C + Dish I
C = volume of sample used in mL.

16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within in Quality Control SOP QS03 is completed by each analyst and then provided to the group leader for further processing and approval.

See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

16.1 Demonstration of Capability (DOC)
16.2 PT Studies

17. Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

18.1. Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria.

18.2. Table 2 of this Sop provides information on QC samples, frequency, and the associated criteria specific to the performance of this SOP.

19. Corrective Actions and Out-of-Control Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20. Contingencies for Handling Out-of-Control or Unacceptable Data

20.1. Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20.2. If a blank is not less than the reporting limit or BS recovery is not within 80 to 120%, corrective action is necessary. Consult your group leader for specific action to take.

21. Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

22. Equipment / Instrument Maintenance

22.1. If oven is not working, notify Group leader and email maintenance.

22.2. If pump is not working, notify Group leader and email maintenance.

22.3. If balance is not working, notify Group leader and email maintenance.

23. Computer Hardware and Software

23.1. Not Applicable.

24. Troubleshooting

24.1. Regular maintenance must be done on the vacuum pump periodically. Always check oil holders to make sure oil is in it up to the red fill line before using. Make sure that reservoir is hooked up in line in case water filters over. This reservoir should be hooked up such that any water coming over will be collected in a flask so it will not go directly into the pump itself. This reservoir should be dumped and cleaned out periodically.

24.2. If weight seems off, insure you are weighing the correct sample.

24.3. Inspect glassware or crucibles before using to insure they are not cracked as this will give you false results.

24.4. Make sure ovens are reading correct temperature. If not, adjust accordingly.

24.5. Properly clean funnel and accessories before use to insure that there are no contaminants getting into the sample.

24.6. Insure glassware was properly cleaned to avoid inaccurate data.

24.7. Insure balance is reading accurately.

24.8. Insure desiccant is the correct color. If not, place in oven. Remove after one hour. If this is the case, use the other desiccator.

25. References

25.1. Standard Methods for the Examination of Water and Wastewater – 22nd Edition.

25.2. SOKOLOFF, V.P. 1933. Water of crystallization in total solids of water analysis. *Ind. Eng. Chem.*, Anal. Ed. 5:336

25.3. DoD Quality Systems Manual for Environmental Laboratories version 5.0, 7/2013 [Based on ISO/IEC 17025:2005(E) and the NELAC Institute (TNI) Standards, Volume 1, (September 2009)].

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Table 2, Method Quality Control Requirements Summary.

26.2. Table 3, Technical Completeness / Accuracy Checklist

26.3. Table 4, Data Reviewers Checklist

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
MB/BLK	One per prep batch	• Must be less than the LOD	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
BS	One per prep batch	Must be in the range of 80 to 120%	• Follow guidelines from SOP QS05
BSD	One per prep batch when insufficient sample volume to duplicate a batch sample.	RPD must be 5% or less	• Follow guidelines from SOP QS05
Sample Duplicate	One for every 10 samples	RPD must be 5% or less	If the data can be reanalyzed it should be. If not the data is flagged and an NCR is generated.
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Must meet the criteria of the BS for average accuracy • Precision criteria is 5% or less	• Re-prep and / or • Re-analysis

Table 3. TDS/TDVS Data Reviewers Checklist

- ❑ Analyst authorization
- ❑ All general information complete
- ❑ Correct Units
- ❑ Corrections crossed out, initialed and dated with reason (if needed)
- ❑ Analytical Balance Calibrated
- ❑ BLK per day & per 20 samples & in control
- ❑ BS per day & in control
- ❑ Duplicate per 10 & in control
- ❑ Handwritten data sheets match excel data sheet
- ❑ True Values & sources indicated
- ❑ Samples noted when <DL
- ❑ 10% of calculations checked
- ❑ BS prep. indicated &/or calculation shown
- ❑ Holding time met
- ❑ On and off Dates/Times recorded
- ❑ On and off Temperatures recorded
- ❑ Data in LIMS and reviewed
- ❑ Reagents/Standards verified accurate on bench sheets and in LIMS.
- ❑ Problems discussed with group leader
- ❑ Necessary NCR's attached
- ❑ NCR written when Duplicate was outside the ±5% range!
- ❑ Additional information needed for reports:

2nd checked by/date:

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP 198 REVISION #: 16 EFFECTIVE DATE: 20161222

TOXICITY CHARACTERISTIC LEACHING PROCEDURE METHOD 1311 AND SYNTHETIC PRECIPITATION LEACHING PROCEDURE METHOD 1312

APPROVALS:

Lab Director: _____ Date: **20161222**

Data Quality Manager: _____ Date: **20161222**

Group Leader: _____ interim Date: **20161222**

Changes Summary

SOP198_R16_20161222_TCLP_SPLP
- SPLP 1312 procedure added to sections throughout this SOP
- Section 9.5 updated to indicate pH meter should be accurate to + 0.05 units at 25°C.
- Section 12.2.2 updated to reflect spike may not be less than five times the method detection limit.
- Section 13.2.2 updated to indicate "until 2 successive weighings yield the same value within ±1%".
- Section 16 updated IDOC procedure for TCLP/SPLP leachate generation.

SOP198_R15_20161108_TCLP
- Documentation procedures updated to remove logbooks and record necessary information on BCH_TCLP_SPLP_ZHE.rpt bench sheet.
- Section 9.1 - Tumbler rotation verification updated to indicate documentation within LIMS maintenance log by tumbler.
- Section 12.1 indicates TCLP/ZHE Vessels to be logged as reagents for tracking 20 uses then blank for each vessel.

SOP198_R14_20160815_TCLP
- Method and SOP references updated in section 5.
- References to reagent log removed and updated to reflect LIMS entry/ID.
- TCLP/SPLP logbook and ZHE logbook attachments updated in section 26.
- Updated section 16 items.

SOP198_R13_20151123_TCLP
- TCLP/SPLP logbook attachment updated in section 26.

SOP198_R12_20150921_TCLP
- All references to supervisor updated to reflect group leader.
- Section 10 updated to reference QS04.
- Section 16 updated to reference QS03.

Revision 11, 07/18/2014
- Watermark update to include proprietary reference.
- Section 8.2 updated to reflect use of nitrile gloves.
- Section 10.7 updated to reflect option to purchase commercial concentrates of the extraction fluids.
- Section 13.2 updated to clarify process of proportioning extraction fluid when 100g of sample is not available.
- Section 13.2.10 updated to reflect option to use Teflon Tape or Teflon Liner to improve vessel seal.
- Sections 22,23 and 24 added.
- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.

Revision 10, 11/01/12
- Section 11.5 updated to require "**Record the matrix spike sample and spike ID on the TCLP/SPLP bench sheet."**
- Section 12.1 appended with "Tracking is required in the TCLP/SPLP vessel log (see appendix). Record "X" when the vessel is used and populate the "blank" field with date when it is used for a blank. Blank is required after every 20th time a vessel is used."
- Appendix added reflecting table from logbook used to track use of vessel and blanks prepared after every 20th sample in each vessel.
- Sections13.2.10 and 13.3.12.3 updated to include: "Add note of temperature range and affected samples in associated workorder comments section."

Revision 09, 10/31/11
- The SOP is an update from Revision 08 dated 09/16/11
- Added requirement to monitor temperature over 18 hours with Min/Max thermometer.
- Added updated logbook to record all parameters involved in the process.
- Updated format to include 22 components and all quality system SOP references.

Revision 08, 09/16/11
- The SOP is an update from Revision 07 dated 08/31/10

- Removed “soapy” from ZHE water wash and replaced extraction fluid with water in ZHE extraction procedure.

Revision 07, 08/31/10

- The SOP is an update from Revision 06 dated 12/22/08
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

TOXICITY CHARACTERISTIC LEACHING PROCEDURE METHOD 1311
AND
SYNTHETIC PRECIPITATION LEACHING PROCEDURE METHOD 1312

1. ID of the method

This SOP is compliant with – SW846 Methods 1311 and 1312

2. Applicable matrix and matrices

This SOP is applicable to – liquid, solid, and multiphasic wastes.

3. Limits of detection and quantitation:

See analytical method SOPs.

4. Scope and application, including parameters to be analyzed:

The test method is applicable to liquids, solids and multi-phasic wastes.

5. Summary of the method

5.1. For liquid samples, (i.e., those containing less than 0.5% dry solid material), the sample, after filtration, is defined as the extract.

5.2. For samples containing greater than 0.5% solids, a 5 g and 100 g aliquot of wet solids are generated. The liquid phase for the 100 g aliquot, if any, is separated from the solid phase and stored at 4°C for later analysis; the particle size of the solid phase is reduced, if necessary. The 5 g aliquot is used to determine the appropriate extraction fluid for TCLP. The 100 g aliquot is added to the appropriate extraction fluid and tumbled for 18+/-2 hours. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration.

5.3. If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

6. Definitions

Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7. Interferences

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods and related SOPs.

8. Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab wide.

8.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex or nitrile gloves and lab coats is highly recommended.

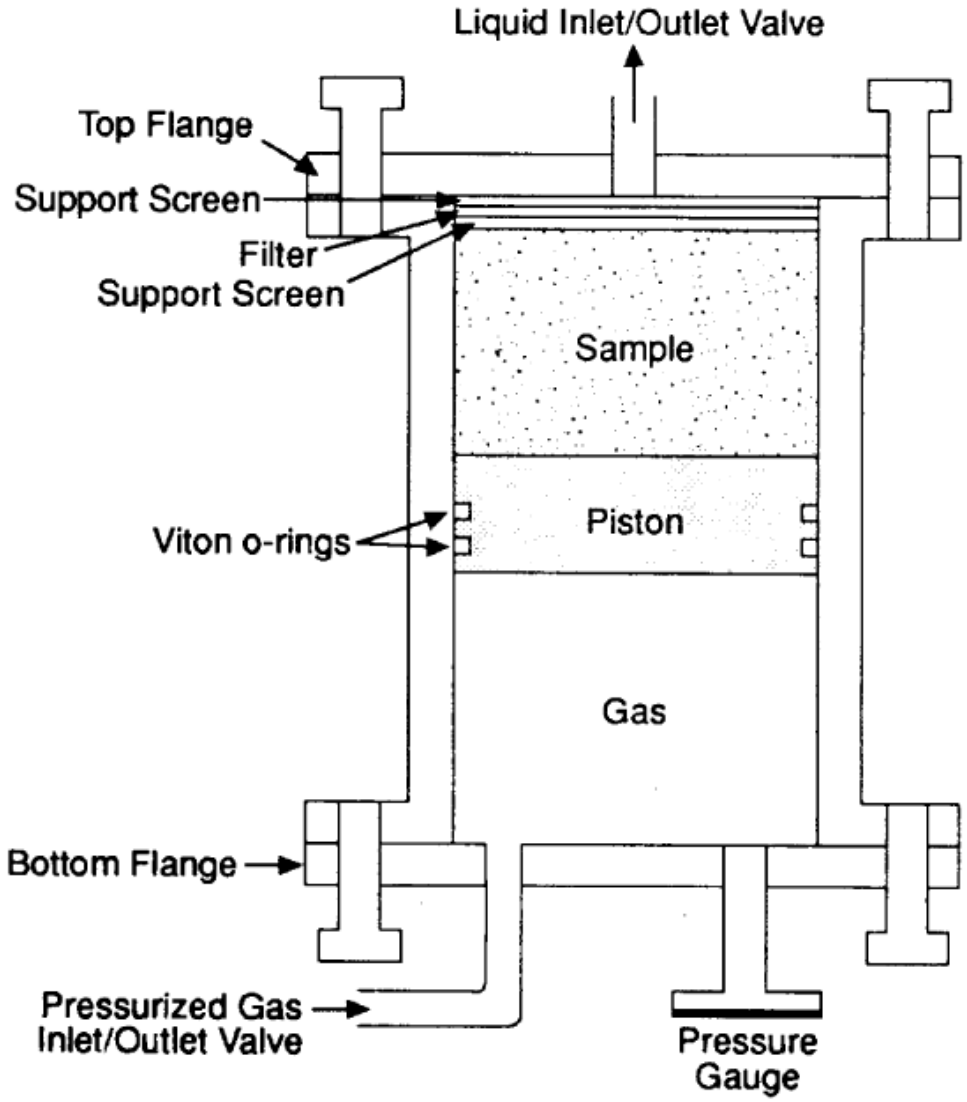
8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.4 MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9. Equip. and supplies

9.1. Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 $\pm$ 2 rpm. This rate is measured and recorded in the LIMS maintenance log for each tumbler (with labels indicating "initials/date/rate" placed on the tumbler) at least once per quarter to ensure accuracy of the

equipment. The LIMS is setup to track due dates for maintenance items and send reminders to the section when maintenance is due.



9.2. Extraction Vessels

9.2.1. Zero-Headspace Extraction Vessel (ZHE). The ZHE (depicted above) allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel. The vessels shall have an internal volume of 500-600 mL, and be equipped to accommodate a 90-110 mm filter. The devices contain VITON®[1] O-rings which should be replaced frequently.

9.2.1.1. For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 30 to 40 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.

9.2.1.2. The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 10 minutes, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

[1] VITON® is a trademark of Du Pont.

9.2.1.3. Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure. Whereas the volatiles procedure refers to pounds per square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds.

9.2.2. Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel. The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste. It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated.

9.3. Filtration Devices: It is recommended that all filtrations be performed in a hood or well-ventilated area.

9.3.1. Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

9.3.2. Filter Holder: When the waste is evaluated for other than volatile analytes, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. Vacuum filtration can only be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration.

9.3.3. Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components.

9.4. Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 µm (Whatman GF/F), or equivalent. **Pre-filters must not be used. When evaluating the mobility of metals**, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

9.5. pH Meters: The meter should be accurate to + 0.05 units at 25°C.

9.6. ZHE Extract Collection Devices: When both liquid and solid phases exist, TEDLAR®[2] bags are used to collect the initial liquid phase and the final extract of the waste from the ZHE device. When only a solid phase exists, the final extract is collected in a 40 mL VOA vial.

9.7. ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit, or other ZHE device)

9.8. Laboratory Balance: Accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

9.9. Beaker or Erlenmeyer flask, glass, various sizes

9.10. Watch glass

9.11. Magnetic stirrer.

9.12. Tubing used in transfer of sample from ZHE to VOA vial- supplier is Dow Corning, material is Silastic. Fisher Cat No. 515-014 size 0.188 in (4.78 mm) I.D. x 0.375 in (9.53 mm) O.D. (or equivalent).

10. Reagents and standard

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method. All reagents shall be made from ACS reagent grade chemicals. All reagents used for distillation and analysis are entered into Element. **These reagents must be added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the associated samples.**

10.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where

[2] TEDLAR® is a registered trademark of Du Pont.

such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.2. Reagent Water. Reagent water is defined as water in which an interferent is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods.

10.2.1. A water purification system may also be used to generate reagent water for volatile extractions.

10.2.2. Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at 90 ± 5 degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

10.3. Hydrochloric acid (1N), HCl, made from ACS reagent grade.

10.4. Nitric acid (1N), HNO_3, made from ACS reagent grade.

10.5. Sodium hydroxide (15N), NaOH, made from ACS reagent grade.

10.6. Glacial acetic acid, CH_3CH_2COOH, ACS reagent grade.

10.7. Sulfuric acid/nitric acid (60/40 percent mixture) H_2SO_4/HNO_3. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid. If preferred, a more dilute H_2SO_4/ HNO_3 acid mixture may be prepared and used in steps 10.4.1 and 10.4.2 making it easier to adjust the pH of the extraction fluids. i.e. 6mL/4mL dilute to 100mL with DI.

10.8. TCLP Extraction fluid (Premade concentrates may be purchased commercially from vendors such as Environmental Express or Miller Analytical).

10.8.1. Extraction fluid #1: Add 11.4 mL glacial acetic acid to approximately 1800 mL DI water. Add 8.6 mL 15N sodium hydroxide and dilute to 2 L. Seal and invert four times. Determine the pH which should be 4.93 +/- 0.05. Document preparation and pH in the LIMS and add LIMS ID to the bench sheet.

10.8.2. Extraction fluid #2: Add 11.4 mL glacial acetic acid to approximately 1800 mL DI water and dilute to 2 L. Seal and invert four times. Determine the pH which should be 2.88 +/- 0.05. Document preparation and pH in the LIMS and add LIMS ID to the bench sheet.

10.9. **SPLP Extraction Fluids (Premade concentrates may be purchased commercially from vendors such as Environmental Express or Miller Analytical)**

10.9.1. Extraction fluid #1: This fluid is made by adding the 60/40 percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water until the pH is 4.20 ± 0.05. The fluid is used to determine the leachability of soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters. Determine the pH and record on the bench sheet. Record Fluid in LIMS and record LIMS ID on bench sheet. NOTE: Solutions are unbuffered and exact pH may not be attained.

10.9.2. Extraction fluid #2: This fluid is made by adding the 60/40 percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water until the pH is 5.00 ±0.05. The fluid is used to determine the leachability of soil from a site that is west of the Mississippi River. Record Fluid in LIMS and record LIMS ID in on bench sheet.

10.9.3. Extraction fluid #3: This fluid is reagent water and is used to determine cyanide and volatiles leachability.

NOTE: All extraction fluids are purchased "certified" or prepared fresh and in volume quantity that matches the number of extractions to be performed on a given day. The bench sheet is used to record the fluid type of choice with LIMS ID for traceability to the lot numbers of reagents used.

10.9. Analytical standards are prepared according to the appropriate analytical method.

11. Sample collection, preservation, shipment and storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.1. Extreme care must be taken when taking samples. Samples should be collected using an appropriate sampling plan.

11.2. There may be requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for the preliminary evaluations of the percent solids and the particle size. An aliquot may be needed to conduct the nonvolatile analyte extraction procedure. If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

11.3. Preservatives will not be added to samples before extraction.

11.4. When the sample is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon -lined septum capped vials and stored at 4°C. Samples should be opened only immediately prior to extraction. Volatiles may be preserved prior to ZHE extraction, if client requested.

11.5. Extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts must be spiked according to their prospective analytes before preservation. Record the matrix spike sample and spike ID on the preparation bench sheet. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2 after spiking, unless precipitation occurs (see Section 13.3.13 if precipitation occurs). Extracts or portions of extracts for volatile analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. Preserve according to the analytical methods.

12. Quality control

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.1. A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. No bias correction is to be taken into consideration (Fr 57, 227). Tracking is required in the LIMS. Add reagent ID for each vessel used in a batch so that use may be tracked through the LIMS. Blank is required after every 20th time a vessel is used.

12.2. A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.). One matrix spike must be analyzed for each analytical batch. Preparation personnel will follow the matrix spike addition guidance provided in each analytical method.

12.2.1. Matrix spikes are to be added after filtration of the extract and before preservation.

12.2.2. The matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same volume of extract as that which was analyzed for the unspiked sample.

12.2.3. The matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. If interferences exist, an alternate approach may be required.

12.2.4. Matrix spike recoveries are calculated by the following formula:

$$\%R\ (\%Recovery) = 100\ (X_s - X_u)/K$$

where:

X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample.

12.3. All quality control measures described in each analytical methods must be followed.

12.4. The use of method of standard addition (MSA) shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the extract is not at least 50% and the concentration does not exceed the regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

12.4.1. The MSA requires preparing calibration standards in the sample matrix rather than reagent water. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The forth aliquot is the unknown. The first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water. Analyze all four aliquots.

12.4.2. Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependent variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

12.5. Extraction holding times.

SAMPLE MAXIMUM HOLDING TIMES [DAYS]				
	From: Field collection To: TCLP/SPLP extraction	From: TCLP/SPLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total elapsed time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable

If holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Of course, exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

13. Procedure

13.1. Documenting TCLP, SPLP and ZHE:

13.1.1. Batch samples and necessary QC prior to initiating process. All reagents/standards/equipment to be used in the process must be added to the LIMS bench sheet including (but not limited to) filters, acid, fluids, **vessels**, etc.

13.1.2. Print the completed bench sheet using print format "bch_TCLP_SPLP_ZHE.rpt"

13.1.3. The bench sheet has columns for all the required information needed for leaching the sample. Add an extra sheet for details/comments if samples are particularly complicated. Make sure all fields are completed or marked "NA" if not applicable.

13.1.4. Once the leachate is tumbled, filtered, labeled, and all information written in the required columns on the bench sheet (including the weight of sample used or any comments required), enter the data in the LIMS bench sheet and set the status to "prepared".

13.1.5. Scan the handwritten bench sheet with barcode for upload to the LIMS and place in the archive bin.

13.2. Preliminary Evaluations

Perform preliminary evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo extraction. These preliminary evaluations include: (1) determination of the percent solids; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration; (3) determination of whether the solid portion of the waste requires particle size reduction; and (4) determination of which of the extraction fluids are to be used for the nonvolatile extraction of the waste.

13.2.1. Percent wet solids: Percent wet solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

13.2.1.1. If the sample will obviously yield no liquid when subjected to pressure filtration proceed to Section 13.1.3.

13.2.1.2. If the sample is liquid or has more than one phase, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Section 9.3.2 and is outlined in Sections 13.2.1.3 through 13.2.1.9.

13.2.1.3. Pre-weigh the filter making sure the lot number is recorded on the bench sheet.

13.2.1.4. Assemble the filter holder and filter following the manufacturer's instructions. Place the weighed filter in the Buchner funnel.

13.2.1.5. Mix the sample and weigh out a representative subsample of (100 gram minimum) and record exact weight.

13.2.1.6. Allow sludges to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

13.2.1.7. Quantitatively transfer the sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 13.2.1.5 to determine the weight of the waste sample that will be filtered.

13.2.1.8. Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

13.2.1.9. The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances.

Remove filter carefully from the Buchner funnel so as to get as much of the solid as possible.

Record the weight of the liquid and solid phases. Determine the weight of the solid phase of the sample by subtracting the weight of the liquid phase from the weight of the total sample, as determined in Section 14.2.1.5 or 14.2.1.7

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid}}{\text{Total weight of waste}} \times 100$$

13.2.2. If the percent solids is equal to or greater than 0.5%, then proceed either to Section 13.2.3 to determine whether the solid material requires particle size reduction or to Section 13.2.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids is less than 0.5%, then proceed to Section 13.3.9 if the nonvolatile TCLP/SPLP is to be performed and to Section 13.4 with a fresh portion of the waste if the volatile TCLP/SPLP is to be performed.

13.2.2.1. Remove the solid material and filter from the filtration apparatus.

13.2.2.2. Dry the filter and solid material at 100 ± 20°C until 2 successive weighings yield the same value within ±1%. Record the final weight.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

13.2.2.3. Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{\text{(Wt. of dry waste + filter) - tared wt. of filter}}{\text{Initial wt. of waste}} \times 100$$

13.2.2.4. If the percent dry solids is less than 0.5%, then proceed to Section 13.2.9 if the nonvolatile TCLP/SPLP is to be performed, and to Section 13.3 if the volatile TCLP/SPLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP/SPLP is to be performed, return to the beginning of this Section (13.2) and, with a fresh portion of waste, determine whether particle size

reduction is necessary (Section 13.2.3) and determine the appropriate extraction fluid (Section 13.2.4). If only the volatile TCLP/SPLP is to be performed, see the note in Section 13.2.4.

13.2.3. Determination of whether the waste requires particle size reduction: Using the solid portion of the sample, evaluate the solid for particle size. Particle size reduction is required, unless the solid passes through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken.

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample-specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

13.2.4. Determination of appropriate non-volatiles extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents, determine the appropriate fluid for the non-volatiles extraction as follows:

NOTE: If TCLP/SPLP extraction is only required for volatiles, proceed to Section 13.4.

13.2.4.1. Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 250 mL beaker or Erlenmeyer flask.

13.2.4.2. Add 96.5 mL of reagent water to the beaker, cover with a watch-glass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use TCLP extraction fluid #1. Proceed to Section 13.3.

13.2.4.3. If the pH is >5.0, add 3.5 mL 1N HCl, **slurry briefly**, cover with a watch-glass, heat to 50°C, and hold at 50°C for 10 minutes. (Note: Do not mix the sample during heating). The hot block may be used for the purpose of heating and holding at 50°C for 10 minutes.

13.2.4.4. Let the solution cool to room temperature (use Hobart to aid in cooling, 30 to 60 minutes) and record the pH. If the pH is <5.0, use TCLP extraction fluid #1. If the pH is >5.0, use TCLP extraction fluid #2. Note: The pH must be measured as quickly as possible in the phase of the procedure. It is critical to measure the pH as soon as the sample has reached room temperature. Proceed to Section 13.3.

13.2.4.5. For SPLP soils, if the sample is from a site that is east of the Mississippi River, SPLP extraction fluid #1 should be used. If the sample is from a site that is west of the Mississippi River, SPLP extraction fluid #2 should be used.

13.2.4.6. For SPLP wastes and wastewater, SPLP extraction fluid #1 should be used.

13.2.4.7. For SPLP cyanide containing wastes and/or soils, SPLP extraction fluid #3 (reagent water) must be used because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

13.2.4.8. If the aliquot of the waste used for the preliminary evaluation was determined to be 100% solid, then it can be used for the Section 13.3 extraction (assuming at least 100 grams remain), and the Section 13.4 extraction (assuming at least 25 grams remain).

13.3. Procedure for Metals and Semi-Volatiles

A minimum sample size of 100 grams (solid and liquid phases) is recommended. If less than the specified amount is used, proportion extraction fluid accordingly and notify manager. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semi-volatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP/SPLP extract will be sufficient to support all of the analyses required.

All glassware and equipment must be thoroughly cleaned before use. A hot soapy water wash, rinse with tap water, then repeat following with a DI water rinse. Rinse with 1:1 HNO_3 , Methanol and acetone, and a final rinse with DI water.

13.3.1. If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid), weigh out a subsample of the waste (100 gram minimum) and proceed to Section 13.3.9.

13.3.2. If the sample is liquid or multi-phasic, liquid/solid separation is required. This involves the filtration device described in Section 9.3.2 and is outlined in Sections 13.3.4 to 13.3.8.

13.3.3. Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Section 9.4).

13.3.4. Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids, the liquid portion, after filtration, is defined as the TCLP/SPLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required. For wastes containing >0.5% dry solids use the percent solids information to determine the optimum sample size (100 gram minimum) for filtration.

13.3.5. Allow sludge to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered, followed by filtration of the solid portion of the waste through the same filtration system.

13.3.6. Refer to Section 13.2.1.7.

13.3.7. Refer to Section 13.2.1.8.

13.3.8. If the waste contains <0.5% dry solids, proceed to Section 13.3.13. If the waste contains >0.5% dry solids, and if particle size reduction of the solid was needed in Section 13.2.3, proceed to Section 13.3.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 13.3.11.

13.3.9. Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 13.2.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

13.3.10. Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent wet solids} \times \text{weight of waste fltered}}{100}$$

Slowly add this amount of appropriate extraction fluid to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape or Teflon liner be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature shall be maintained at 23 ± 2ºC during the extraction period. Reset the Min/Max thermometer at the beginning of the tumbling cycle and record Min/Max temperatures at the end of the tumbling cycle. Report exceedences using NCR form. Add note of temperature range and affected samples in associated work-order comments section. See SOP QS05.

NOTE: As rotation continues, pressure may build up within the extractor bottle for some types of wastes. To relieve excess pressure, the extractor bottle should be periodically opened (e.g., after 15 minutes, and 30 minutes) and vented into a hood.

13.3.11. Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Section 13.2.1.8. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed.

13.3.12. Prepare the extract as follows:

13.3.12.1. If the sample contained no initial liquid phase, the filtered liquid material obtained is defined as the extract. Proceed to Section 13.3.13.

13.3.12.2. If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 13.3.12.1 with the initial liquid phase of the waste obtained in Section 13.2.1.8. This combined liquid is defined as the extract. Proceed to Section 13.3.13.

13.3.12.3. If the initial liquid phase of the waste, as obtained from Section 13.2.1.8, is not or may not be compatible with the filtered liquid resulting from Section 13.3.12.1, do not combine these liquids. Analyze these liquids, collectively defined as the extract, and combine the results mathematically, as described in Section 13.3.14.

13.3.13. Following collection of the extract, the pH of the extract must be recorded. Immediately split aliquots, give to appropriate analyst for spiking and preservation of the extract for analysis. Metal aliquots must be acidified with nitric acid to pH <2 after spiking. (If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible.) All other aliquots must be stored under refrigeration (4°C) until analyzed. Extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes.

If the individual phases are to be analyzed separately, determine the volume of the individual phases (to $\pm$ 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = On next page
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

13.4. Volatiles Extraction Procedure

Use ZHE device to obtain TCLP/SPLP extract for analysis of volatile compounds only. TCLP extraction for volatile constituents uses only TCLP extraction fluid #1 while SPLP extraction for volatile constituents uses only SPLP extraction fluid #3.

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

All glassware and equipment must be thoroughly cleaned before use. A hot water wash, rinse with tap water, then repeat following with a DI water rinse. Rinse with methanol and a final rinse with DI water.

13.4.1. Pre-weigh the filtrate collection container and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis.

13.4.2. Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with DI water). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 13.4, Section 13.2.1 and/or 13.2.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

13.4.3. If the waste is 100% solid, weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Section 13.4.5.

13.4.4. If the waste contains < 0.5% dry solids, the liquid portion of waste, after filtration, is defined as the extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing $\geq$ 0.5% dry solids, use the percent solids information determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

13.4.4.1. For wastes containing < 5% solids, weigh out a 500 gram subsample of waste and record the weight.

13.4.4.2. For wastes containing $\geq$ 5% solids, determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{percent wet solids}} \times 100$$

13.4.5. If particle size reduction of the solid portion of the waste was required in Section 13.2.3, proceed to Section 13.4.6. If particle size reduction was not required in Section 13.2.3, proceed to Section 13.3.7.

13.4.6. Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Section 13.2.3. The means used to effect particle size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriately altered, proceed to Section 13.4.7.

13.4.7. Waste sludge need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

13.4.8. Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 13.4.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Section 13.2.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Section 13.4.12.

13.4.9. Attach the pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

13.4.10. The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase. (Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid.) If the original waste contained <0.5% dry solids, this filtrate is defined as the extract. Proceed to Section 13.4.15.

13.4.11. The liquid phase may now be either analyzed immediately or preserved and stored at 4°C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \text{ x percent wet solids x weight of waste filtered}}{100}$$

13.4.12. The following details how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. TCLP extraction fluid #1 is used in all TCLP cases while SPLP extraction fluid #3 is used in all SPLP cases. With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be pre-flushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

13.4.12.1. After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

13.4.12.2. Place the ZHE in the rotary agitation apparatus and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction occurs) shall be maintained at 23 ± 2°C during agitation. Reset the Min/Max thermometer at the beginning of the tumbling cycle and record Min/Max temperatures at the end of the tumbling cycle. Report exceedences using NCR form. Add note of temperature range and affected samples in associated work-order comments section. See SOP QS05.

13.4.13. Following the 18 ± 2 hour period, check the pressure behind the ZHE piston by checking gauge and quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Section 9.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag or VOC vials) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Section 13.4.9. All extract shall be filtered and collected if the TEDLAR® bag is used, if the extract is multi-phasic, or if the waste contained an initial liquid phase.

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

13.4.14. If the original waste contained no initial liquid phase, the filtered liquid material obtained from above is defined as the extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from above and the initial liquid phase (Section 13.3.9) are collectively defined as the extract.

13.4.15. Following collection of the extract, immediately prepare the extract for analysis (one of the vials will be spiked at this point) and store with minimal headspace at 4°C. Analyze the extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).

C_2 = The concentration of the analyte of concern in the second phase (mg/L).

14. Calibration and standardization

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

15. **Data analysis and calculations**

Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

16. **Method performance**

Initial Demonstration of Capability (DOC)/Performance (IDP): Each analyst must perform an IDOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-L samples with experienced supervision. For the TCLP/SPLP procedure IDOC, an SVOC TCLP QC sample is purchased and prepared in 4 vessels using 1/4th the blank matrix and 1/4th the spike solution specified then extracted/analyzed via the normal TCLP SVOC procedure and verified within the QC sample limits. The DOC form, as listed in Quality Control SOP QS03 is completed by each preparation/analysis employett and then provided to the Group Leader for further processing and approval per SOP QS03.

17. Pollution prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data assessment and acceptance criteria for QC measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria.

19. Corrective actions and out-of-control data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20. Contingencies for handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

21. Waste management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

22. Equip./instrument maintenance

22.1.1. **Tumblers**

22.1.1 TCLP/SPLP

– Call maintenance for motor check/service if it does not meet 30+/-2 RPM

- Call maintenance if closing screws get stripped for repair/replacement

22.2.1 ZHE

- Call maintenance for motor check/service if it does not meet 30+/-2 RPM
- If screw posts get bent/stripped, replace with new posts (Assoc. Design)

22.2ZHE's

22.2.1 Clean and check units before each use

22.2.2 Replace o-rings if necessary due to age, wear or loss of pressure

22.2.3 If top or bottom screws break, replace with new ones. (Pliers may be used)

22.3TCLP/SPLP Bottles

22.3.1 Clean before each use. Rinse with 1:1 HN03, methanol and acetone rinse with DI water.

22.3.2 Use Teflon lid liners when organics are involved.

22.4Vac Pump

22.4.1 Refill oil container when low
22.4.2 Call maintenance for any mechanical errors
22.4.3 Replace vac tubing as needed due to age or wear

22.5Funnel and Flask

22.5.1 Clean appropriately before use and in between each sample

22.6pH Meter

22.6.1 Calibrate before use
22.6.2 Record calibration in logbook
22.6.3 If meter breaks, tell group leader, and use a different meter, with documentation.

23. Computer hardware and software

23.1. **Not applicable**

24. Troubleshooting

24.1. If TCLP/SPLP container leaks due to pressure build-up, re-tumble.
24.2. When a sample is leached that has a concentration above the regulatory limit, the vessel it is leached in, must be cleaned thoroughly before it is used again. In extreme cases a blank should be run in the vessel before it is used for a new sample to ensure cleanliness.

25. References

25.1. Test Methods for Evaluating Solid Waste, SW-846, Third Edition and updates
25.2. DoD Quality Systems Manual for Environmental Laboratories version 5.0, 7/2013 [Based on ISO/IEC 17025:2005(E) and the NELAC Institute (TNI) Standards, Volume 1, (September 2009)].

26. Tables, diagrams, flowcharts and validation data

NA

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 201 REVISION #: 34 EFFECTIVE DATE: 20170411

GC/MS SEMIVOLATILES, Regular and Low-Level, BY EPA METHOD 625 AND SW846 METHOD 8270D

APPROVALS:

Lab Director: _____ Date: **20170411**

Data Quality Manager: _____ Date: **20170411**

Group Leader: _____ Date: **20170411**
Randy Ward

Changes Summary

SOP201_R34_20170411_BNA

- All references to 3-Methylphenol and 4-Methylphenol removed leaving only 3-Methylphenol/4-Methylphenol.

SOP201_R33_20160831_BNA

- Table 10 updated to include problematic compounds from SW846 8270D Section 1.4.

SOP201_R32_20160812_BNA

- Table 1A and table 1B updated for in-house limits with headers defined
- References to Target and specific instruments removed.
- LLOQ and IDP terminology added from SW-846 Update V.
- MDL/LOD/LOQ references simplified in table 2.
- Updated checklist for low-standard read-back to include list/discuss.

SOP201_R31_20160414_BNA

- Target list updated in table 1A and table 6 to include Dibenz(a,c)anthracene and Perylene for 8270D and 625 waters.

SOP201_R30_20151221_BNA

- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.
- Calibration Standard references in appendix 26.12 reduced to include only one example calibration in order to reduce size of SOP (63 pages removed!).

SOP201_R29_20150921_BNA

- Section 10 updated to reflect QS04, to reference Standards Appendix 26.12 in 10.5 and to reference digital thermometer use in 10.4.
- Section 12.5 updated to clarify exceptions.
- Sections 13 updated to indicate LOQ-level standard back-calculation on linear curve fit and to reference Standards Appendix 26.12 where appropriate.
- Section 14 updated to reference Standards Appendix 26.12 where appropriate.
- Section 14.1 updated to clarify low-level full-scan
- Table 1 separated into 1a and 1b for regular and low-level full-scan with updated in-house limits.
- Table 2 updated for MDL, LOD, LOQ, linear curve fit verification and to add immediate analysis of 2 passing CCVs for QSM.
- Analyst Review Checklist updated.
- Table 7 removed.
- Standards Appendix added for routine standard makeup information.

SOP201-R28_20150120_BNAPAH

- References to section supervisors updated to group leaders.
- References to ChroEval updated to ChromEval and BNA6 added to instrumentation references.
- Restriction on ICAL forced through origin removed.

- Checklist updated to reflect greater than instead of less than for correlation coefficients and add instrument ID to line 10.

SOP201-R27_20141020_BNAPAH

- Table 2 tuning section updated to correctly reference table number 8.

SOP201_R26_20140929_BNAPAH

- Section 12.6 inserted and data reviewers' checklist updated to address structural isomers.
- Section 14.2 updated to address spectral selection for DFTPP tune.

SOP201_R25_20140825_BNAPAH

- Checklist updated to correct calibration criteria.

Revision 24, 20140722

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark has been updated to include proprietary reference.
- Section 8.0 MSDS sheet reference updated to reflect vendor website access.
- Sections 9g and 14.5 updated to reflect the use of Chemstation/Enviroquant for data processing on BNA3, BNA4 and BNA5.
- Section 11 updated to reference SOP QS10.
- Section 13.2 updated for regular SVOC concentration range and to reflect low-concentration SVOCs instead of just PAHs.
- Sections 22.0, 23.0 and 24.0 added/populated.
- Table 1 updated to reflect DL/LOD/LOQ and control limits for QSM 4.2 and QSM 5.0
- Table 2 updated to include QSM 5.0 requirements
- Table 7 updated to reflect additional low-level SVOC analytes

Revision 23, 09/09/2013

- Added additional GC injection port liner type in section 9.0.
- Added DOD concentration requirement for IDOC's in section 12.6

Revision 22, 08/20/2012

- MSDS location updated.
- Internal standard spike volume corrected from 20ul to 10ul.
- Common contaminant reference in table 2 updated to reflect phthalates.
- Table 3 removed and all tables renumbered accordingly.
- Data Review Checklist updated.
- Internal Standard Association tables updated.
- List of 8270D SPCCs added and referenced in table 2.
- List of poor performing compounds added and referenced in table 2.

Revision 21, 05/16/2011

- A requirement to record the amount and ID of the internal standard used was added to section 14.3.

Revision 20, 4/13/10

- The SOP is formatted to simplify the text and place all method/program specifications in the SOP tables.

Revision 19, 4/11/10

- The SOP is an update from Revision 18 dated 9/16/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DOD samples are analyzed.

Table of Contents

GC/MS SEMIVOLATILES, Regular and Low-Level, BY EPA METHOD 625 AND SW846 METHOD 8270D

1.0 Identification of the Test Method

This SOP is based primarily on SW-846 Methods 8000B/8000C/8000D/8270C/8270D. *Federal Register* Method 625 and CLP Methods for Semi-volatiles have also been used in the development of this SOP.

2.0 Applicable Matrix or Matrices

This SOP is used for the analysis of semi-volatile organic compounds (including low-level full-scan) in a variety of matrices (soils, sediments, waters, etc.).

3.0 Limits of Detection and Quantitation

See Table 1

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is routinely analyzed and reported under the scope of this SOP is listed in the Appendix of this SOP. This table also lists the associated Detection Limit, Limit of Detection and Reporting Limit (also defined as the Limit of Quantitation or Lower Limit of Quantitation.).

4.2 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC/MS using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program, the pressure program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra from the sample. Analytes are quantitated relative to known standards using the internal standard method.

6.0 Definitions –

Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

7.1 All raw data (samples & QC) must be evaluated for interferences. If contamination occurs, determine whether the source of interference is in the preparation or clean-up of the samples and take corrective action to eliminate the problem.

7.2 Contamination by carryover can occur when samples of high-concentration and low-concentration are analyzed sequentially. To reduce carryover, the sample syringe must be rinsed with solvent between injections. If an unusually high sample is

detected, a solvent blank should be analyzed for cross contamination or the subsequent sample should be evaluated for cross-contamination.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab-wide.

8.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.

8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.

8.4 MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9.0 Equipment & Supplies

a HP 5890/6890/7890GC complete with electronic pressure control and temperature programmable gas chromatograph suitable for split-less injection.

b Column: RTX-5 (or equivalent) 30 m x 0.25 mm I.D. x 0.25 µm film thickness fused silica capillary column.

c HP 5971/5973/5975 mass spectrometer capable of scanning from 35 to 500 amu every second or less, using 70 volts electron energy in electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all the tuning criteria of the EPA methods.

d HP 7673/7683 autosampler capable of reproducibility from one injection to another proven by meeting QC and calibration criteria.

e HP GC/MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

f Acquisition Software: HP Chemstation system is interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

g Data Processing Software: Chemstation/Enviroquant is used for data processing. Each system accepts and stores acquired data. Each system plots by extracted ion current profile (EICP). Each system is also capable of integrating the abundances in any EICP between specified times or scan-number limits.

h Micro syringes – gas tight 5uL and larger.

i Liners – 2mm or 4mm: straight, single gooseneck, "direct connect", or with glass wool (for split injection).

j Septa 11mm.

k Seals- dual vespel stainless steel or gold plated 0.8mm.

l Vials- 2ml and larger (clear or amber).

m Volumetric flasks- 10ml and larger class A with glass stopper.

10.0 Reagents and Standards –

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST-RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. All certificates received with standards must be marked with the LIMS ID and forwarded to the administration department for scanning/saving in order to be available for view within LIMS.

10.2 Reagent grade chemicals shall be used in all tests unless otherwise specified. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.3 Methylene chloride (Please read SOP-336 before handling this solvent in our laboratory.) – Trace analysis grade.

10.4 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded on the certificate of analysis sheet. The date they are opened is noted on the label and recorded in LIMS. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the freezer at a temperature of -15°C ± 5°C from the date they are received/prepared. Standards are brought to room temperature before being used to make standards. Sonication is used if precipitation is observed after bringing to room temperature. The freezer temperature is monitored daily with a quarterly calibrated digital min/max thermometer and recorded with calibration correction in the associated temperature/calibration logbook.

10.5 Individual standard makeup is recorded in LIMS with specific details concerning the standard being used, concentration, amount, solvent and expiration date. See Standards Appendix (26.12) for detailed examples. All reagents used for preparation and analysis are entered into LIMS. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the associated samples.

11.0 Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling while soil samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed.

12.0 Quality Control

12.1 Internals - All samples and QC are spiked with internal standards prior to analysis.

12.2 Surrogates - All samples and QC are spiked with surrogates prior to extraction. See **Table 2** for criteria and corrective action.

12.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples to provide accuracy results. See **Table 2** for criteria and corrective action.

12.4 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See **Table 2** for criteria and corrective action.

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, if sample is available. If no sample is available, an LCSD must be extracted to provide precision results. See **Table 2** for criteria and corrective action. Some factors that may affect MS/MSD results are:

12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action may be required in the form of reanalysis or re-extraction if the exceedence is attributed to laboratory factors and not sample matrix.

12.5.2 Original sample concentration - If a spiked compound exceeds recovery limits and the concentration of that compound in the original sample greatly exceeds the spiked concentration, no further corrective action may be necessary other than the generation of a non-conformance report to document the problem. Discuss with your group leader.

12.5.3 MS vs. MSD - If a spiked compound has a similar recovery issues in both the MS and MSD which are not traced to a method problem, no further action may be necessary other than the generation of a non-conformance report to document the problem. Discuss with your group leader.

12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your group leader who should discuss the problem with the data quality manager, lab director and/or client/project manager to determine the action to be taken.

12.6 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Resolution between the peaks should be $\leq$50%. To evaluate the resolution, the standard (CCV) must be processed with the correct method in Chemstation and be evaluated in QEdit. The Chromeval function in QEdit allows for resolution to be evaluated under the Evaluate Resolution function. Once the peaks in question are integrated, a % resolution can be generated and printed.

12.7 Initial Demonstration of Capability (DOC)/Performance (IDP): Each analyst must perform an IDOC/IDP prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples (may be prepared from same source as calibration). The data is calculated for accuracy and precision requirements. The IDOC form is completed by each analyst and then provided to the group leader for further processing and approval. See **Table 2** for acceptance criteria. This also must be done when a new instrument is installed or a significant change to the method has been made.

13.0 Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Initial Calibration - An initial multi-point calibration curve must be analyzed and shown to meet the initial calibration criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. The LOQ-level standard should be back-calculated to the calibration curve for linear calibration curve fits and a recovery calculated to verify linearity. See Table 2 for criteria. Generally, levels for the curve range from 5.0ug/mL to 100ug/mL for regular SVOCs and 0.1µg/mL to 50µg/mL for low-level full-scan SVOCs. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

13.3 Initial Calibration Verification (ICV) - A second source standard at the continuing calibration verification (CCV) level must be analyzed and calculated against the initial calibration curve, then shown to meet the ICV criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. For ICV standard preparation, refer to LIMS. Standards Appendix (26.12) has detailed standard examples. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

13.4 Continuing Calibration Verification (CCV) - Every 12 hours, a CCV must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. For CCV standard preparation, refer to LIMS. . Standards Appendix (26.12) has detailed standard examples. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

13.5 Ending Calibration Verification (HCV) - For QSM 5.0, an ending CCV must be analyzed within the 12 hour tune. Same documentation requirements apply as for the CCV above. See **Table 2** of this SOP for acceptance criteria and corrective action.

14.0 Procedure

Prior to analysis the samples are prepared for chromatography using the appropriate sample preparation methods (generally SW-846 methods 3510, 3520, 3541, 3546 3550, 3580, EPA method 625 or CLP).

14.1 Chromatographic conditions: Refer to corresponding instrument maintenance log for gas chromatograph and mass spectrometer conditions. Note: The Low-Level Full-Scan analysis is performed following all 8270D requirements with sensitivity enhancing adjustments across all analytical components combining to allow increased sensitivity.

14.2 The mass spectrum of DFTPP is acquired by using the auto find DFTPP function in Chemstation (which uses three scans [one of the apex and the scan prior and after

the apex] with background subtraction [instrument selected peak within 20 scans prior to the apex scan]) to evaluate the DFTPP. DFTPP tailing is evaluated in the Chromeval function of Chemstation. After the DFTPP is processed with the correct method, the data is evaluated in QEdit. The Chromeval function in QEdit allows tailing to be evaluated and printed under the Evaluate tailing function and allows for degradation to be evaluated and printed under the Evaluate Degradation function.

14.2 Tuning - Prior to any calibration or analysis, DFTPP tuning criteria must be met for a 50 ng injection of the tuning standard. The injection port performance compounds (pentachlorophenol, benzidine and 4,4'-DDT) are also injected to verify the performance of the injection port . See **Table 2** for criteria and corrective action.

14.3 Extracts - Prior to analysis, 1.0 mL extracts are prepared by verifying volume and spiking with 10uL of the internal standard solution. See Standards Appendix (26.12) for detailed standard examples. Record the amount and ID of internal standard spiked on the sequence log printed from the LIMS.

14.4 Instrument sequence-The instrument sequence log is filled out prior to sample analyses. An example of a typical instrument sequence log follows:

1-SEQ-TUN1 (12:00 am)
2-SEQ-CCV1
3-SEQ-BS1
4-SEQ-BLK1
5-Sample
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample
12-Sample
13-Sample
14-SEQ-MS1
15-SEQ-MSD1
16-SEQ-TUN2 (12:00pm - 12 hours since last DFTPP/CCV)
17-SEQ-CCV2
18-Sample
19-Sample
20-Sample

14.5 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the Chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through Chemstation/Enviroquant. The following must be checked to determine if the sample will need reanalysis or dilution. Criteria and corrective action are found in Table 2. Formal data evaluation is detailed in SOP QS05 and documented using the Analyst Data Review Checklist (see Appendix). Manual integration guidance is found in SOP QS07.

14.5.1 Internal Standard Area Counts and Retention Times
14.6.2 Surrogate Recoveries and Retention Times
14.5.3 Analyte concentration.

14.5.4 Analyte identification based on spectrum and retention time.
14.5.5 Analyte quantitation verification.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:
A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard.

15.2 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\%\ \text{Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where:
Calculated concentration is determined from the initial calibration.
Theoretical concentration is the concentration at which the standard was prepared.

$$\%\ \text{Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where:
CCV RF is the response factor from the analysis of the verification standard
Average RF is the average of the response factors from the initial calibration.

15.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration}\ (\mu g/L) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:
A_s = Area (or height) of the peak for the analyte in the sample.
A_{is} = Area (or height) of the peak for the internal standard.
C_{is} = Concentration of the internal standard in the volume extracted in ug/L.
D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.
$\overline{RF}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

15.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s,

A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

15.3 Any questions left unanswered by this SOP should be clarified by reading the referenced method. If questions still remain unanswered, check with the Group Leader, Technical Director and/or Data Quality Manager.

16.0 Method Performance

See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

16.1 Method Detection Limit Study or Detection Limit Determination
16.2 Limit of Detection Verification
16.3 Limit of Quantitation or Reporting Limit Verification
16.4 Initial Demonstration of Capability (IDOC)/Performance (IDP)
16.5 PT Studies

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Corrective Actions and out-of-control data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for

Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on corrective actions and out of control data.

20.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

21.0 Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

22.0 Equipment/Instrument Maintenance

22.1 Injector Maintenance for split injection port

22.1.1 Replace liner, septum, and possibly o-ring if breakdown is occurring. If more maintenance is required then replace the inlet seal and clip column.

22.2 Injector Maintenance for splitless injection port

22.2.1 Replace liner, septum, all o-rings, and inlet seal. Column will also be clipped.

23. Computer Hardware and Software

23.1 Acquisition Software: HP Chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

23.2 Data Processing Software: HP Chemstation data system. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances of any EICP between specified time or scan-number limits. NIST98.L mass spectral library is installed.

24.0 Troubleshooting-If routine maintenance in section 22.0 did not bring the instrument back into control, more involved maintenance may be required (i.e. detector maintenance, analytical column maintenance, etc.). Bring to the attention of the Group Leader.

25.0 References

40 CFR, Part 136; Appendix A
Test Methods for Evaluating Solid Waste, SW-846
National Environmental Laboratory Accreditation Conference; CH. 5, 2003
USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
DOD Quality Systems Manual for Environmental Laboratories,

26.0 Tables, Diagrams, Flowcharts and Validation Data

26.1 Table 1A, Regular SVOC limits
Table 1B, Low-Level Full-Scan SVOC limits
26.2 Table 2, QA/QC summary table
26.3 Table 3, Data Reviewers Checklist(s)
26.4 Table 4, 625 QC Limits

S/W	Method	Table 1A Analyte (Regular Limits)	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Solid	8270D	1,1'-Biphenyl	92-52-4	333	167	83.3	ug/Kg	45	110	30	40	117	20
Solid	8270D	1,2,4,5-Tetrachlorobenzene	95-94-3	333	167	83.3	ug/Kg	50	150	30	37	119	20
Solid	8270D	1,2,4-Trichlorobenzene	120-82-1	333	167	83.3	ug/Kg	45	110	30	34	118	20
Solid	8270D	1,2-Dichlorobenzene (12DCB)	95-50-1	333	167	83.3	ug/Kg	45	95	30	33	117	20
Solid	8270D	1,2-Diphenylhydrazine	122-66-7	333	167	83.3	ug/Kg	10	110	30	41	125	20
Solid	8270D	1,3-Dichlorobenzene (13DCB)	541-73-1	333	167	83.3	ug/Kg	40	100	30	30	115	20
Solid	8270D	1,4-Dichlorobenzene (14DCB)	106-46-7	333	167	83.3	ug/Kg	35	105	30	31	115	20
Solid	8270D	1,4-Dioxane	123-91-1	333	167	83.3	ug/Kg	10	110	30	43	110	20
Solid	8270D	1-Methylnaphthalene	90-12-0	333	167	83.3	ug/Kg	30	110	30	40	119	20
Solid	8270D	2,2 -Oxybis(1-chloropropane), bis(2-Chloro-1-methylethyl)ether (fka bis(2-Chloroisopropyl) ether)	108-60-1	333	167	83.3	ug/Kg	20	115	30	33	131	20
Solid	8270D	2,3,4,6-Tetrachlorophenol	58-90-2	333	167	83.3	ug/Kg	50	125	30	44	125	20
Solid	8270D	2,4,5-Trichlorophenol	95-95-4	333	167	83.3	ug/Kg	50	110	30	41	124	20
Solid	8270D	2,4,6-Trichlorophenol (TCP)	88-06-2	333	167	83.3	ug/Kg	45	110	30	39	126	20
Solid	8270D	2,4-Dichlorophenol (DCP)	120-83-2	333	167	83.3	ug/Kg	45	110	30	40	122	20
Solid	8270D	2,4-Dimethylphenol	105-67-9	1330	667	333	ug/Kg	30	105	30	30	127	20
Solid	8270D	2,4-Dinitrophenol	51-28-5	3330	1670	833	ug/Kg	15	130	30	48	113	20
Solid	8270D	2,4-Dinitrotoluene (DNT)	121-14-2	333	167	83.3	ug/Kg	50	115	30	48	126	20
Solid	8270D	2,6-Dichlorophenol (DCP)	87-65-0	333	167	83.3	ug/Kg	45	110	30	41	117	20
Solid	8270D	2,6-Dinitrotoluene	606-20-2	333	167	83.3	ug/Kg	50	110	30	46	124	20
Solid	8270D	2.4.6-Tribromophenol (Surrogate)	118-79-6	667	333	167	ug/Kg	35	125		39	132	
Solid	8270D	2-Chloronaphthalene	91-58-7	333	167	83.3	ug/Kg	45	105	30	41	114	20
Solid	8270D	2-Chlorophenol	95-57-8	333	167	83.3	ug/Kg	45	105	30	34	121	20
Solid	8270D	2-Fluorobiphenyl (Surrogate)	321-60-8	333	167	83.3	ug/Kg	45	105		44	115	
Solid	8270D	2-Fluorophenol (Surrogate)	367-12-4	667	333	167	ug/Kg	35	105		35	115	
Solid	8270D	2-Methylnaphthalene	91-57-6	333	167	83.3	ug/Kg	40	110	30	38	122	20
Solid	8270D	2-Methylphenol (o-Cresol)	95-48-7	333	167	83.3	ug/Kg	40	105	30	32	122	20
Solid	8270D	2-Nitroaniline	88-74-4	1330	667	333	ug/Kg	45	120	30	44	127	20
Solid	8270D	2-Nitrophenol (ONP)	88-75-5	333	167	83.3	ug/Kg	40	110	30	36	123	20
Solid	8270D	3,3'-Dichlorobenzidine (DCB)	91-94-1	333	167	83.3	ug/Kg	10	130	30	22	121	20
Solid	8270D	3-Methylphenol/4-Methylphenol	108-39-4/106	333	167	83.3	ug/Kg	40	105	30	34	119	20
Solid	8270D	3-Nitroaniline	99-09-2	1330	667	333	ug/Kg	25	110	30	33	119	20
Solid	8270D	4,6-Dinitro-2-methylphenol (DNOC)	534-52-1	3330	1670	833	ug/Kg	30	135	30	29	132	20
Solid	8270D	4-Bromophenyl phenyl ether	101-55-3	333	167	83.3	ug/Kg	45	115	30	46	124	20
Solid	8270D	4-Chloro-3-methylphenol	59-50-7	333	167	83.3	ug/Kg	45	115	30	45	122	20
Solid	8270D	4-Chloroaniline	106-47-8	333	167	83.3	ug/Kg	10	95	30	17	106	20

S/W	Method	Table 1A Analyte (Regular Limits)	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Solid	8270D	4-Chlorophenyl phenyl ether	7005-72-3	333	167	83.3	ug/Kg	45	110	30	45	121	20
Solid	8270D	4-Nitroaniline (PNA)	100-01-6	1330	667	333	ug/Kg	35	115	30	73	112	20
Solid	8270D	4-Nitrophenol (PNP)	100-02-7	1330	667	333	ug/Kg	15	140	30	30	132	20
Solid	8270D	Acenaphthene	83-32-9	333	167	83.3	ug/Kg	45	110	30	40	123	20
Solid	8270D	Acenaphthylene	208-96-8	333	167	83.3	ug/Kg	45	105	30	32	132	20
Solid	8270D	Acetophenone	98-86-2	333	167	83.3	ug/Kg	35	110	30	33	115	20
Solid	8270D	Aniline	62-53-3	333	167	83.3	ug/Kg	10	110	30	36	110	20
Solid	8270D	Anthracene	120-12-7	333	167	83.3	ug/Kg	55	105	30	47	123	20
Solid	8270D	Atrazine	1912-24-9	333	167	83.3	ug/Kg	55	105	30	47	127	20
Solid	8270D	Benzaldehyde	100-52-7	333	167	83.3	ug/Kg	10	160	30	55	110	20
Solid	8270D	Benzidine	92-87-5	3330	1670	833	ug/Kg	0	200	30	0	110	20
Solid	8270D	Benzo(a)anthracene	56-55-3	333	167	83.3	ug/Kg	50	110	30	49	126	20
Solid	8270D	Benzo(a)pyrene	50-32-8	333	167	83.3	ug/Kg	50	110	30	45	129	20
Solid	8270D	Benzo(b)fluoranthene	205-99-2	333	167	83.3	ug/Kg	45	115	30	45	132	20
Solid	8270D	Benzo(g,h,i)perylene	191-24-2	333	167	83.3	ug/Kg	40	125	30	43	134	20
Solid	8270D	Benzo(k)fluoranthene	207-08-9	333	167	83.3	ug/Kg	45	125	30	47	132	20
Solid	8270D	Benzoic Acid	65-85-0	1330	667	333	ug/Kg	0	110	30	41	136	20
Solid	8270D	Benzyl alcohol	100-51-6	333	167	83.3	ug/Kg	20	125	30	29	122	20
Solid	8270D	bis(2-Chloroethoxy)methane	111-91-1	333	167	83.3	ug/Kg	45	110	30	36	121	20
Solid	8270D	bis(2-Chloroethyl)ether (BCEE)	111-44-4	333	167	83.3	ug/Kg	40	105	30	31	120	20
Solid	8270D	bis(2-Ethylhexyl)phthalate (BEHP)	117-81-7	333	167	83.3	ug/Kg	45	125	30	51	133	20
Solid	8270D	Butyl benzyl phthalate (BBP)	85-68-7	333	167	83.3	ug/Kg	50	125	30	48	132	20
Solid	8270D	Caprolactam	105-60-2	333	167	83.3	ug/Kg	50	110	30	46	117	20
Solid	8270D	Carbazole	86-74-8	333	167	83.3	ug/Kg	45	115	30	50	123	20
Solid	8270D	Chrysene	218-01-9	333	167	83.3	ug/Kg	55	110	30	50	124	20
Solid	8270D	Dibenz(a,h)anthracene	53-70-3	333	167	83.3	ug/Kg	40	125	30	45	134	20
Solid	8270D	Dibenzofuran (DBF)	132-64-9	333	167	83.3	ug/Kg	50	105	30	44	120	20
Solid	8270D	Diethyl phthalate (DEP)	84-66-2	333	167	83.3	ug/Kg	50	115	30	50	124	20
Solid	8270D	Dimethyl phthalate (DMP)	131-11-3	333	167	83.3	ug/Kg	50	110	30	48	124	20
Solid	8270D	Di-n-butyl phthalate (DBP)	84-74-2	333	167	83.3	ug/Kg	55	110	30	51	128	20
Solid	8270D	Di-n-octyl phthalate (DNOP)	117-84-0	333	167	83.3	ug/Kg	40	130	30	45	140	20
Solid	8270D	Fluoranthene	206-44-0	333	167	83.3	ug/Kg	55	115	30	50	127	20
Solid	8270D	Fluorene	86-73-7	333	167	83.3	ug/Kg	50	110	30	43	125	20
Solid	8270D	Hexachlorobenzene (HCB)	118-74-1	333	167	83.3	ug/Kg	45	120	30	45	122	20
Solid	8270D	Hexachlorobutadiene (HCBD)	87-68-3	333	167	83.3	ug/Kg	30	110	30	32	123	20
Solid	8270D	Hexachlorocyclopentadiene (HCCPD)	77-47-4	333	167	83.3	ug/Kg	10	110	30	42	110	20
Solid	8270D	Hexachloroethane (HCE)	67-72-1	333	167	83.3	ug/Kg	35	110	30	28	117	20

S/W	Method	Table 1A Analyte (Regular Limits)	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Solid	8270D	Indeno(1,2,3-cd)pyrene	193-39-5	333	167	83.3	ug/Kg	40	120	30	45	133	20
Solid	8270D	Isophorone	78-59-1	333	167	83.3	ug/Kg	45	110	30	30	122	20
Solid	8270D	Naphthalene	91-20-3	333	167	83.3	ug/Kg	40	105	30	35	123	20
Solid	8270D	Nitrobenzene	98-95-3	333	167	83.3	ug/Kg	40	115	30	34	122	20
Solid	8270D	Nitrobenzene-d5 (Surrogate)	4165-60-0	333	167	83.3	ug/Kg	35	100		37	122	
Solid	8270D	N-Nitrosodimethylamine	62-75-9	333	167	83.3	ug/Kg	20	115	30	23	120	20
Solid	8270D	N-Nitroso-di-n-propylamine (NDPA)	621-64-7	333	167	83.3	ug/Kg	40	115	30	36	120	20
Solid	8270D	N-nitrosodiphenylamine (NDPHA)	86-30-6	333	167	83.3	ug/Kg	50	115	30	38	127	20
Solid	8270D	Pentachlorophenol	87-86-5	1330	667	333	ug/Kg	25	120	30	25	133	20
Solid	8270D	Phenanthrene	85-01-8	333	167	83.3	ug/Kg	50	110	30	50	121	20
Solid	8270D	Phenol	108-95-2	333	167	83.3	ug/Kg	40	100	30	34	121	20
Solid	8270D	Phenol-d6 (Surrogate)	13127-88-3	667	333	167	ug/Kg	40	100		33	122	
Solid	8270D	Pyrene	129-00-0	333	167	83.3	ug/Kg	45	125	30	47	127	20
Solid	8270D	Pyridine	110-86-1	333	167	83.3	ug/Kg	10	110	30	56	110	20
Solid	8270D	Terphenyl-d14 (Surrogate)	1718-51-0	333	167	83.3	ug/Kg	30	125		54	127	
Water	8270D/625	1,1'-Biphenyl	92-52-4	5.0	2.5	1.25	ug/L	45	135	30	49	115	20
Water	8270D/625	1,2,4,5-Tetrachlorobenzene	95-94-3	5.0	2.5	1.25	ug/L	30	135	30	35	121	20
Water	8270D/625	1,2,4-Trichlorobenzene	120-82-1	5.0	2.5	1.25	ug/L	35	105	30	29	116	20
Water	8270D/625	1,2-Dichlorobenzene (12DCB)	95-50-1	5.0	2.5	1.25	ug/L	35	100	30	32	111	20
Water	8270D/625	1,2-Diphenylhydrazine	122-66-7	5.0	2.5	1.25	ug/L	55	115	30	49	122	20
Water	8270D/625	1,3-Dichlorobenzene (13DCB)	541-73-1	5.0	2.5	1.25	ug/L	30	100	30	28	110	20
Water	8270D/625	1,4-Dichlorobenzene (14DCB)	106-46-7	5.0	2.5	1.25	ug/L	30	100	30	29	112	20
Water	8270D/625	1,4-Dioxane	123-91-1	5.0	2.5	1.25	ug/L	5	110	30	19	110	20
Water	8270D/625	1-Methylnaphthalene	90-12-0	5.0	2.5	1.25	ug/L	35	115	30	41	119	20
Water	8270D/625	2,2 -Oxybis(1-chloropropane), bis(2-Chloro-1-methylethyl)ether (fka bis(2-Chloroisopropyl) ether)	108-60-1	5.0	2.5	1.25	ug/L	25	130	30	37	130	20
Water	8270D/625	2,3,4,6-Tetrachlorophenol	58-90-2	10	5.0	2.5	ug/L	25	135	30	50	128	20
Water	8270D/625	2,4,5-Trichlorophenol	95-95-4	5.0	2.5	1.25	ug/L	50	110	30	53	123	20
Water	8270D/625	2,4,6-Trichlorophenol (TCP)	88-06-2	5.0	2.5	1.25	ug/L	50	115	30	50	125	20
Water	8270D/625	2,4-Dichlorophenol (DCP)	120-83-2	5.0	2.5	1.25	ug/L	40	110	30	47	121	20
Water	8270D/625	2,4-Dimethylphenol	105-67-9	5.0	2.5	1.0	ug/L	30	110	30	31	124	20
Water	8270D/625	2,4-Dinitrophenol	51-28-5	40	10	3.0	ug/L	15	140	30	23	143	20
Water	8270D/625	2,4-Dinitrotoluene (DNT)	121-14-2	5.0	2.5	1.25	ug/L	50	120	30	57	128	20
Water	8270D/625	2,6-Dichlorophenol (DCP)	87-65-0	5.0	2.5	1.25	ug/L	30	140	30	50	118	20
Water	8270D/625	2,6-Dinitrotoluene	606-20-2	5.0	2.5	1.25	ug/L	50	115	30	57	124	20
Water	8270D/625	2,4,6-Tribromophenol (Surrogate)	118-79-6	10	5.0	2.5	ug/L	40	125		43	140	

S/W	Method	Table 1A Analyte (Regular Limits)	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Water	8270D/625	2-Chloronaphthalene	91-58-7	5.0	2.5	1.25	ug/L	50	105	30	40	116	20
Water	8270D/625	2-Chlorophenol	95-57-8	5.0	2.5	1.25	ug/L	35	105	30	38	117	20
Water	8270D/625	2-Fluorobiphenyl (Surrogate)	321-60-8	5.0	2.5	1.25	ug/L	50	110		44	119	
Water	8270D/625	2-Fluorophenol (Surrogate)	367-12-4	10	5.0	2.5	ug/L	20	110		19	119	
Water	8270D/625	2-Methylnaphthalene	91-57-6	5.0	2.5	1.25	ug/L	45	105	30	40	121	20
Water	8270D/625	2-Methylphenol (o-Cresol)	95-48-7	5.0	2.5	1.25	ug/L	40	110	30	30	117	20
Water	8270D/625	2-Nitroaniline	88-74-4	20	10	5.0	ug/L	50	115	30	55	127	20
Water	8270D/625	2-Nitrophenol (ONP)	88-75-5	5.0	2.5	1.25	ug/L	40	115	30	47	123	20
Water	8270D/625	3,3'-Dichlorobenzidine (DCB)	91-94-1	5.0	2.5	1.25	ug/L	20	110	30	27	129	20
Water	8270D/625	3-Methylphenol/4-Methylphenol	108-39-4/106	5.0	2.5	1.25	ug/L	30	110	30	29	110	20
Water	8270D/625	3-Nitroaniline	99-09-2	20	10	5.0	ug/L	20	125	30	41	128	20
Water	8270D/625	4,6-Dinitro-2-methylphenol (DNOC)	534-52-1	40	10	3.0	ug/L	40	130	30	44	137	20
Water	8270D/625	4-Bromophenyl phenyl ether	101-55-3	5.0	2.5	1.25	ug/L	50	115	30	55	124	20
Water	8270D/625	4-Chloro-3-methylphenol	59-50-7	5.0	2.5	1.25	ug/L	45	110	30	52	119	20
Water	8270D/625	4-Chloroaniline	106-47-8	5.0	2.5	1.25	ug/L	15	110	30	33	117	20
Water	8270D/625	4-Chlorophenyl phenyl ether	7005-72-3	5.0	2.5	1.25	ug/L	50	110	30	53	121	20
Water	8270D/625	4-Nitroaniline (PNA)	100-01-6	20	10	5.0	ug/L	35	120	30	32	115	20
Water	8270D/625	4-Nitrophenol (PNP)	100-02-7	20	5.0	1.25	ug/L	0	125	30	12	110	20
Water	8270D/625	Acenaphthene	83-32-9	5.0	2.5	1.25	ug/L	45	110	30	47	122	20
Water	8270D/625	Acenaphthylene	208-96-8	5.0	2.5	1.25	ug/L	50	105	30	41	130	20
Water	8270D/625	Acetophenone	98-86-2	5.0	2.5	1.25	ug/L	45	130	30	46	118	20
Water	8270D/625	Aniline	62-53-3	5.0	2.5	1.25	ug/L	25	110	30	26	110	20
Water	8270D/625	Anthracene	120-12-7	5.0	2.5	1.25	ug/L	55	110	30	57	123	20
Water	8270D/625	Atrazine	1912-24-9	5.0	2.5	1.25	ug/L	40	150	30	44	142	20
Water	8270D/625	Benzaldehyde	100-52-7	5.0	2.5	1.25	ug/L	40	125	30	36	115	20
Water	8270D/625	Benzidine	92-87-5	20	5.0	1.25	ug/L	0	110	30	16	110	20
Water	8270D/625	Benzo(a)anthracene	56-55-3	5.0	2.5	1.25	ug/L	55	110	30	58	125	20
Water	8270D/625	Benzo(a)pyrene	50-32-8	5.0	2.5	1.25	ug/L	55	110	30	54	128	20
Water	8270D/625	Benzo(b)fluoranthene	205-99-2	5.0	2.5	1.25	ug/L	45	120	30	53	131	20
Water	8270D/625	Benzo(g,h,i)perylene	191-24-2	5.0	2.5	1.25	ug/L	40	125	30	50	134	20
Water	8270D/625	Benzo(k)fluoranthene	207-08-9	5.0	2.5	1.25	ug/L	45	125	30	57	129	20
Water	8270D/625	Benzoic Acid	65-85-0	40	10	2.5	ug/L	0	125	30	3	110	20
Water	8270D/625	Benzyl alcohol	100-51-6	5.0	2.5	1.25	ug/L	30	110	30	31	112	20
Water	8270D/625	bis(2-Chloroethoxy)methane	111-91-1	5.0	2.5	1.25	ug/L	45	105	30	48	120	20
Water	8270D/625	bis(2-Chloroethyl)ether (BCEE)	111-44-4	5.0	2.5	1.25	ug/L	35	110	30	43	118	20
Water	8270D/625	bis(2-Ethylhexyl)phthalate (BEHP)	117-81-7	5.0	2.5	1.25	ug/L	40	125	30	55	135	20
Water	8270D/625	Butyl benzyl phthalate (BBP)	85-68-7	5.0	2.5	1.25	ug/L	45	115	30	53	134	20

S/W	Method	Table 1A Analyte (Regular Limits)	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Water	8270D/625	Caprolactam	105-60-2	5.0	2.5	1.25	ug/L	5	110	30	7	110	20
Water	8270D/625	Carbazole	86-74-8	5.0	2.5	1.25	ug/L	50	115	30	60	122	20
Water	8270D/625	Chrysene	218-01-9	5.0	2.5	1.25	ug/L	55	110	30	59	123	20
Water	8270D/625	Dibenz(a,h)anthracene	53-70-3	5.0	2.5	1.25	ug/L	40	125	30	51	134	20
Water	8270D/625	Dibenzofuran (DBF)	132-64-9	5.0	2.5	1.25	ug/L	55	105	30	53	118	20
Water	8270D/625	Diethyl phthalate (DEP)	84-66-2	5.0	2.5	1.25	ug/L	40	120	30	56	125	20
Water	8270D/625	Dimethyl phthalate (DMP)	131-11-3	5.0	2.5	1.25	ug/L	25	125	30	45	127	20
Water	8270D/625	Di-n-butyl phthalate (DBP)	84-74-2	5.0	2.5	1.25	ug/L	55	115	30	59	127	20
Water	8270D/625	Di-n-octyl phthalate (DNOP)	117-84-0	5.0	2.5	1.25	ug/L	35	135	30	51	140	20
Water	8270D/625	Fluoranthene	206-44-0	5.0	2.5	1.25	ug/L	55	115	30	57	128	20
Water	8270D/625	Fluorene	86-73-7	5.0	2.5	1.25	ug/L	50	110	30	52	124	20
Water	8270D/625	Hexachlorobenzene (HCB)	118-74-1	5.0	2.5	1.25	ug/L	50	110	30	53	125	20
Water	8270D/625	Hexachlorobutadiene (HCBD)	87-68-3	5.0	2.5	1.25	ug/L	25	105	30	22	124	20
Water	8270D/625	Hexachlorocyclopentadiene (HCCPD)	77-47-4	10	5.0	2.5	ug/L	0	120	30	7	110	20
Water	8270D/625	Hexachloroethane (HCE)	67-72-1	5.0	2.5	1.25	ug/L	30	95	30	21	115	20
Water	8270D/625	Indeno(1,2,3-cd)pyrene	193-39-5	5.0	2.5	1.25	ug/L	45	125	30	52	134	20
Water	8270D/625	Isophorone	78-59-1	5.0	2.5	1.25	ug/L	50	110	30	42	124	20
Water	8270D/625	Naphthalene	91-20-3	5.0	2.5	1.25	ug/L	40	100	30	40	121	20
Water	8270D/625	Nitrobenzene	98-95-3	5.0	2.5	1.25	ug/L	30	110	30	45	121	20
Water	8270D/625	Nitrobenzene-d5 (Surrogate)	4165-60-0	5.0	2.5	1.25	ug/L	40	110		44	120	
Water	8270D/625	N-Nitrosodimethylamine	62-75-9	5.0	2.5	1.25	ug/L	25	110	30	13	110	20
Water	8270D/625	N-Nitroso-di-n-propylamine (NDPA)	621-64-7	5.0	2.5	1.25	ug/L	35	130	30	49	119	20
Water	8270D/625	N-nitrosodiphenylamine (NDPHA)	86-30-6	5.0	2.5	1.25	ug/L	50	110	30	51	123	20
Water	8270D/625	Pentachlorophenol	87-86-5	20	5.0	1.25	ug/L	40	115	30	35	138	20
Water	8270D/625	Phenanthrene	85-01-8	5.0	2.5	1.25	ug/L	50	115	30	59	120	20
Water	8270D/625	Phenol	108-95-2	5.0	2.5	1.25	ug/L	0	115	30	12	110	20
Water	8270D/625	Phenol-d6 (Advisory Surrogate)	13127-88-3	10	5.0	2.50	ug/L	10	115		9	110	
Water	8270D/625	Pyrene	129-00-0	5.0	2.5	1.25	ug/L	50	130	30	57	126	20
Water	8270D/625	Pyridine	110-86-1	10	5.0	2.5	ug/L	5	110	30	8	110	20
Water	8270D/625	Terphenyl-d14 (Surrogate)	1718-51-0	5.0	2.5	1.25	ug/L	50	135		50	134	
Water	8270D/625	Dibenz(a,c)anthracene	215-58-7	5.0	2.5	1.25	ug/L	10	200	30	10	200	20
Water	8270D/625	Perylene	198-55-0	5.0	2.5	1.25	ug/L	10	200	30	10	200	20

LL= Low Limit, UL = Upper Limit, RPD = relative percent difference, LL5/UL5/RPD5 are limits from QSM5, highlighted limits are in-house limits.

S/W	Method	Table 1B Analyte (Low-Level Full Scan)	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Solid	8270D LLFS	1,1'-Biphenyl	92-52-4	333	167	83.3	ug/Kg	45	110	30	40	117	20
Solid	8270D LLFS	1,2,4,5-Tetrachlorobenzene	95-94-3	13.3	6.67	3.33	ug/Kg	50	150	30	37	119	20
Solid	8270D LLFS	1,2,4-Trichlorobenzene	120-82-1	333	167	83.3	ug/Kg	45	110	30	34	118	20
Solid	8270D LLFS	1,2-Dichlorobenzene (12DCB)	95-50-1	333	167	83.3	ug/Kg	45	95	30	33	117	20
Solid	8270D LLFS	1,2-Diphenylhydrazine	122-66-7	333	167	83.3	ug/Kg	10	110	30	41	125	20
Solid	8270D LLFS	1,3-Dichlorobenzene (13DCB)	541-73-1	333	167	83.3	ug/Kg	40	100	30	30	115	20
Solid	8270D LLFS	1,4-Dichlorobenzene (14DCB)	106-46-7	333	167	83.3	ug/Kg	35	105	30	31	115	20
Solid	8270D LLFS	1,4-Dioxane	123-91-1	33.3	16.7	8.33	ug/Kg	10	110	30	43	110	20
Solid	8270D LLFS	1-Methylnaphthalene	90-12-0	6.67	3.33	1.67	ug/Kg	30	110	30	40	119	20
Solid	8270D LLFS	2,2 -Oxybis(1-chloropropane),bis(2-Chloro-1-methylethyl)ether (fka bis(2-Chloroisopropyl) ether)	108-60-1	6.67	3.33	1.67	ug/Kg	20	115	30	33	131	20
Solid	8270D LLFS	2,3,4,6-Tetrachlorophenol	58-90-2	333	167	83.3	ug/Kg	50	125	30	44	125	20
Solid	8270D LLFS	2,4,5-Trichlorophenol	95-95-4	333	167	83.3	ug/Kg	50	110	30	41	124	20
Solid	8270D LLFS	2,4,6-Tribromophenol (Surrogate)	118-79-6	13.3	6.67	1.67	ug/Kg	45	110	30	39	126	20
Solid	8270D LLFS	2,4,6-Trichlorophenol (TCP)	88-06-2	333	167	83.3	ug/Kg	45	110	30	40	122	20
Solid	8270D LLFS	2,4-Dichlorophenol (DCP)	120-83-2	333	167	83.3	ug/Kg	30	105	30	30	127	20
Solid	8270D LLFS	2,4-Dimethylphenol	105-67-9	1330	667	333	ug/Kg	15	130	30	48	113	20
Solid	8270D LLFS	2,4-Dinitrophenol	51-28-5	3330	1670	833	ug/Kg	50	115	30	48	126	20
Solid	8270D LLFS	2,4-Dinitrotoluene (DNT)	121-14-2	6.67	3.33	1.67	ug/Kg	45	110	30	41	117	20
Solid	8270D LLFS	2,6-Dichlorophenol (DCP)	87-65-0	333	167	83.3	ug/Kg	50	110	30	46	124	20
Solid	8270D LLFS	2,6-Dinitrotoluene	606-20-2	6.67	3.33	1.67	ug/Kg	35	125		39	132	
Solid	8270D LLFS	2-Chloronaphthalene	91-58-7	6.67	3.33	1.67	ug/Kg	45	105	30	41	114	20
Solid	8270D LLFS	2-Chlorophenol	95-57-8	333	167	83.3	ug/Kg	45	105	30	34	121	20
Solid	8270D LLFS	2-Fluorobiphenyl (Surrogate)	321-60-8	6.67	3.33	1.67	ug/Kg	45	105		44	115	
Solid	8270D LLFS	2-Fluorophenol (Surrogate)	367-12-4	13.3	6.67	1.67	ug/Kg	35	105		35	115	
Solid	8270D LLFS	2-Methylnaphthalene	91-57-6	6.67	3.33	1.67	ug/Kg	40	110	30	38	122	20
Solid	8270D LLFS	2-Methylphenol (o-Cresol)	95-48-7	333	167	83.3	ug/Kg	40	105	30	32	122	20
Solid	8270D LLFS	2-Nitroaniline	88-74-4	1330	667	333	ug/Kg	45	120	30	44	127	20
Solid	8270D LLFS	2-Nitrophenol (ONP)	88-75-5	333	167	83.3	ug/Kg	40	110	30	36	123	20
Solid	8270D LLFS	3,3'-Dichlorobenzidine (DCB)	91-94-1	6.67	3.33	1.67	ug/Kg	10	130	30	22	121	20
Solid	8270D LLFS	3-Methylphenol/4-Methylphenol	108-39-4/106	333	167	83.3	ug/Kg	40	105	30	34	119	20
Solid	8270D LLFS	3-Nitroaniline	99-09-2	1330	667	333	ug/Kg	25	110	30	33	119	20
Solid	8270D LLFS	4,6-Dinitro-2-methylphenol (DNOC)	534-52-1	33.3	13.3	3.33	ug/Kg	30	135	30	29	132	20
Solid	8270D LLFS	4-Bromophenyl phenyl ether	101-55-3	333	167	83.3	ug/Kg	45	115	30	46	124	20
Solid	8270D LLFS	4-Chloro-3-methylphenol	59-50-7	333	167	83.3	ug/Kg	45	115	30	45	122	20

S/W	Method	Table 1B Analyte (Low-Level Full Scan)	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Solid	8270D LLFS	4-Chloroaniline	106-47-8	6.67	3.33	1.67	ug/Kg	10	95	30	17	106	20
Solid	8270D LLFS	4-Chlorophenyl phenyl ether	7005-72-3	333	167	83.3	ug/Kg	45	110	30	45	121	20
Solid	8270D LLFS	4-Nitroaniline (PNA)	100-01-6	1330	667	333	ug/Kg	35	115	30	73	112	20
Solid	8270D LLFS	4-Nitrophenol (PNP)	100-02-7	1330	667	333	ug/Kg	15	140	30	30	132	20
Solid	8270D LLFS	Acenaphthene	83-32-9	6.67	3.33	1.67	ug/Kg	45	110	30	40	123	20
Solid	8270D LLFS	Acenaphthylene	208-96-8	6.67	3.33	1.67	ug/Kg	45	105	30	32	132	20
Solid	8270D LLFS	Acetophenone	98-86-2	333	167	83.3	ug/Kg	35	110	30	33	115	20
Solid	8270D LLFS	Aniline	62-53-3	333	167	83.3	ug/Kg	10	110	30	36	110	20
Solid	8270D LLFS	Anthracene	120-12-7	6.67	3.33	1.67	ug/Kg	55	105	30	47	123	20
Solid	8270D LLFS	Atrazine	1912-24-9	333	167	83.3	ug/Kg	55	105	30	47	127	20
Solid	8270D LLFS	Benzaldehyde	100-52-7	333	167	83.3	ug/Kg	10	160	30	55	110	20
Solid	8270D LLFS	Benzidine	92-87-5	3330	1670	833	ug/Kg	0	200	30	0	110	20
Solid	8270D LLFS	Benzo(a)anthracene	56-55-3	6.67	3.33	1.67	ug/Kg	50	110	30	49	126	20
Solid	8270D LLFS	Benzo(a)pyrene	50-32-8	6.67	3.33	1.67	ug/Kg	50	110	30	45	129	20
Solid	8270D LLFS	Benzo(b)fluoranthene	205-99-2	6.67	3.33	1.67	ug/Kg	45	115	30	45	132	20
Solid	8270D LLFS	Benzo(g,h,i)perylene	191-24-2	6.67	3.33	1.67	ug/Kg	40	125	30	43	134	20
Solid	8270D LLFS	Benzo(k)fluoranthene	207-08-9	6.67	3.33	1.67	ug/Kg	45	125	30	47	132	20
Solid	8270D LLFS	Benzoic Acid	65-85-0	1330	667	83.3	ug/Kg	0	110	30	41	136	20
Solid	8270D LLFS	Benzyl alcohol	100-51-6	333	167	83.3	ug/Kg	20	125	30	29	122	20
Solid	8270D LLFS	bis(2-Chloroethoxy)methane	111-91-1	333	167	83.3	ug/Kg	45	110	30	36	121	20
Solid	8270D LLFS	bis(2-Chloroethyl)ether (BCEE)	111-44-4	6.67	3.33	1.67	ug/Kg	40	105	30	31	120	20
Solid	8270D LLFS	bis(2-Ethylhexyl)phthalate (BEHP)	117-81-7	33.3	16.7	8.33	ug/Kg	45	125	30	51	133	20
Solid	8270D LLFS	Butyl benzyl phthalate (BBP)	85-68-7	333	167	83.3	ug/Kg	50	125	30	48	132	20
Solid	8270D LLFS	Caprolactam	105-60-2	333	167	83.3	ug/Kg	50	110	30	46	117	20
Solid	8270D LLFS	Carbazole	86-74-8	6.67	3.33	1.67	ug/Kg	45	115	30	50	123	20
Solid	8270D LLFS	Chrysene	218-01-9	6.67	3.33	1.67	ug/Kg	55	110	30	50	124	20
Solid	8270D LLFS	Dibenz(a,h)anthracene	53-70-3	6.67	3.33	1.67	ug/Kg	40	125	30	45	134	20
Solid	8270D LLFS	Dibenzofuran (DBF)	132-64-9	6.67	3.33	1.67	ug/Kg	50	105	30	44	120	20
Solid	8270D LLFS	Diethyl phthalate (DEP)	84-66-2	333	167	83.3	ug/Kg	50	115	30	50	124	20
Solid	8270D LLFS	Dimethyl phthalate (DMP)	131-11-3	333	167	83.3	ug/Kg	50	110	30	48	124	20
Solid	8270D LLFS	Di-n-butyl phthalate (DBP)	84-74-2	333	167	83.3	ug/Kg	55	110	30	51	128	20
Solid	8270D LLFS	Di-n-octyl phthalate (DNOP)	117-84-0	13.3	6.67	3.33	ug/Kg	40	130	30	45	140	20
Solid	8270D LLFS	Fluoranthene	206-44-0	6.67	3.33	1.67	ug/Kg	55	115	30	50	127	20
Solid	8270D LLFS	Fluorene	86-73-7	6.67	3.33	1.67	ug/Kg	50	110	30	43	125	20
Solid	8270D LLFS	Hexachlorobenzene (HCB)	118-74-1	6.67	3.33	1.67	ug/Kg	45	120	30	45	122	20
Solid	8270D LLFS	Hexachlorobutadiene (HCBD)	87-68-3	6.67	3.33	1.67	ug/Kg	30	110	30	32	123	20
Solid	8270D LLFS	Hexachlorocyclopentadiene (HCCPD)	77-47-4	33.3	16.7	8.33	ug/Kg	10	110	30	42	110	20

S/W	Method	Table 1B Analyte (Low-Level Full Scan)	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Solid	8270D LLFS	Hexachloroethane (HCE)	67-72-1	6.67	3.33	1.67	ug/Kg	35	110	30	28	117	20
Solid	8270D LLFS	Indeno(1,2,3-cd)pyrene	193-39-5	6.67	3.33	1.67	ug/Kg	40	120	30	45	133	20
Solid	8270D LLFS	Isophorone	78-59-1	6.67	3.33	1.67	ug/Kg	45	110	30	30	122	20
Solid	8270D LLFS	Naphthalene	91-20-3	6.67	3.33	1.67	ug/Kg	40	105	30	35	123	20
Solid	8270D LLFS	Nitrobenzene	98-95-3	6.67	3.33	1.67	ug/Kg	40	115	30	34	122	20
Solid	8270D LLFS	Nitrobenzene-d5 (Surrogate)	4165-60-0	6.67	3.33	1.67	ug/Kg	35	100		37	122	
Solid	8270D LLFS	N-Nitrosodimethylamine	62-75-9	333	167	83.3	ug/Kg	20	115	30	23	120	20
Solid	8270D LLFS	N-Nitroso-di-n-propylamine (NDPA)	621-64-7	6.67	3.33	1.67	ug/Kg	40	115	30	36	120	20
Solid	8270D LLFS	N-nitrosodiphenylamine (NDPHA)	86-30-6	6.67	3.33	1.67	ug/Kg	50	115	30	38	127	20
Solid	8270D LLFS	Pentachlorophenol	87-86-5	66.7	33.3	16.7	ug/Kg	25	120	30	25	133	20
Solid	8270D LLFS	Phenanthrene	85-01-8	6.67	3.33	1.67	ug/Kg	50	110	30	50	121	20
Solid	8270D LLFS	Phenol	108-95-2	13.3	6.67	3.33	ug/Kg	40	100	30	34	121	20
Solid	8270D LLFS	Phenol-d6 (Surrogate)	13127-88-3	13.3	6.67	1.67	ug/Kg	40	100		33	122	
Solid	8270D LLFS	Pyrene	129-00-0	6.67	3.33	1.67	ug/Kg	45	125	30	47	127	20
Solid	8270D LLFS	Pyridine	110-86-1	1330	667	83.3	ug/Kg	10	110	30	56	110	20
Solid	8270D LLFS	Terphenyl-d14 (Surrogate)	1718-51-0	6.67	3.33	1.67	ug/Kg	30	125		54	127	
Water	8270D LLFS	1,1'-Biphenyl	92-52-4	5.0	2.5	1.25	ug/L	45	135	30	49	115	20
Water	8270D LLFS	1,2,4,5-Tetrachlorobenzene	95-94-3	0.20	0.10	0.050	ug/L	30	135	30	35	121	20
Water	8270D LLFS	1,2,4-Trichlorobenzene	120-82-1	5.0	2.5	1.25	ug/L	35	105	30	29	116	20
Water	8270D LLFS	1,2-Dichlorobenzene (12DCB)	95-50-1	5.0	2.5	1.25	ug/L	35	100	30	32	111	20
Water	8270D LLFS	1,2-Diphenylhydrazine	122-66-7	5.0	2.5	1.25	ug/L	55	115	30	49	122	20
Water	8270D LLFS	1,3-Dichlorobenzene (13DCB)	541-73-1	5.0	2.5	1.25	ug/L	30	100	30	28	110	20
Water	8270D LLFS	1,4-Dichlorobenzene (14DCB)	106-46-7	5.0	2.5	1.25	ug/L	30	100	30	29	112	20
Water	8270D LLFS	1,4-Dioxane	123-91-1	0.50	0.25	0.125	ug/L	5	110	30	19	110	20
Water	8270D LLFS	1-Methylnaphthalene	90-12-0	0.20	0.10	0.050	ug/L	35	115	30	41	119	20
Water	8270D LLFS	2,2 -Oxybis(1-chloropropane),bis(2-Chloro-1-methylethyl)ether (fka bis(2-Chloroisopropyl) ether)	108-60-1	0.20	0.10	0.050	ug/L	25	130	30	37	130	20
Water	8270D LLFS	2,3,4,6-Tetrachlorophenol	58-90-2	10	5.0	1.25	ug/L	25	135	30	50	128	20
Water	8270D LLFS	2,4,5-Trichlorophenol	95-95-4	5.0	2.5	1.25	ug/L	50	110	30	53	123	20
Water	8270D LLFS	2,4,6-Tribromophenol (Surrogate)	118-79-6	0.40	0.20	0.050	ug/L	50	115	30	50	125	20
Water	8270D LLFS	2,4,6-Trichlorophenol (TCP)	88-06-2	5.0	2.5	0.75	ug/L	40	110	30	47	121	20
Water	8270D LLFS	2,4-Dichlorophenol (DCP)	120-83-2	5.0	2.5	1.25	ug/L	30	110	30	31	124	20
Water	8270D LLFS	2,4-Dimethylphenol	105-67-9	5.0	2.5	0.60	ug/L	15	140	30	23	143	20
Water	8270D LLFS	2,4-Dinitrophenol	51-28-5	10	2.5	0.50	ug/L	50	120	30	57	128	20
Water	8270D LLFS	2,4-Dinitrotoluene (DNT)	121-14-2	0.20	0.10	0.050	ug/L	30	140	30	50	118	20
Water	8270D LLFS	2,6-Dichlorophenol (DCP)	87-65-0	5.0	2.5	1.25	ug/L	50	115	30	57	124	20

S/W	Method	Table 1B Analyte (Low-Level Full Scan)	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Water	8270D LLFS	2,6-Dinitrotoluene	606-20-2	0.20	0.10	0.050	ug/L	40	125		43	140	
Water	8270D LLFS	2-Chloronaphthalene	91-58-7	0.20	0.10	0.050	ug/L	50	105	30	40	116	20
Water	8270D LLFS	2-Chlorophenol	95-57-8	5.0	2.5	1.25	ug/L	35	105	30	38	117	20
Water	8270D LLFS	2-Fluorobiphenyl (Surrogate)	321-60-8	0.20	0.10	0.050	ug/L	50	110		44	119	
Water	8270D LLFS	2-Fluorophenol (Surrogate)	367-12-4	0.40	0.20	0.050	ug/L	20	110		19	119	
Water	8270D LLFS	2-Methylnaphthalene	91-57-6	0.20	0.10	0.050	ug/L	45	105	30	40	121	20
Water	8270D LLFS	2-Methylphenol (o-Cresol)	95-48-7	5.0	2.5	1.25	ug/L	40	110	30	30	117	20
Water	8270D LLFS	2-Nitroaniline	88-74-4	20	5.0	1.25	ug/L	50	115	30	55	127	20
Water	8270D LLFS	2-Nitrophenol (ONP)	88-75-5	5.0	2.5	1.25	ug/L	40	115	30	47	123	20
Water	8270D LLFS	3,3'-Dichlorobenzidine (DCB)	91-94-1	0.50	0.25	0.12	ug/L	20	110	30	27	129	20
Water	8270D LLFS	3-Methylphenol/4-Methylphenol	108-39-4/106	5.0	2.5	1.25	ug/L	30	110	30	29	110	20
Water	8270D LLFS	3-Nitroaniline	99-09-2	20	5.0	1.25	ug/L	20	125	30	41	128	20
Water	8270D LLFS	4,6-Dinitro-2-methylphenol (DNOC)	534-52-1	1.0	0.40	0.10	ug/L	40	130	30	44	137	20
Water	8270D LLFS	4-Bromophenyl phenyl ether	101-55-3	5.0	2.5	1.25	ug/L	50	115	30	55	124	20
Water	8270D LLFS	4-Chloro-3-methylphenol	59-50-7	5.0	2.5	1.25	ug/L	45	110	30	52	119	20
Water	8270D LLFS	4-Chloroaniline	106-47-8	0.20	0.10	0.050	ug/L	15	110	30	33	117	20
Water	8270D LLFS	4-Chlorophenyl phenyl ether	7005-72-3	5.0	2.5	1.25	ug/L	50	110	30	53	121	20
Water	8270D LLFS	4-Nitroaniline (PNA)	100-01-6	20	5.0	1.25	ug/L	35	120	30	32	115	20
Water	8270D LLFS	4-Nitrophenol (PNP)	100-02-7	5.0	1.25	0.30	ug/L	0	125	30	12	110	20
Water	8270D LLFS	Acenaphthene	83-32-9	0.20	0.10	0.050	ug/L	45	110	30	47	122	20
Water	8270D LLFS	Acenaphthylene	208-96-8	0.20	0.10	0.050	ug/L	50	105	30	41	130	20
Water	8270D LLFS	Acetophenone	98-86-2	5.0	2.5	1.25	ug/L	45	130	30	46	118	20
Water	8270D LLFS	Aniline	62-53-3	5.0	2.5	1.25	ug/L	25	110	30	26	110	20
Water	8270D LLFS	Anthracene	120-12-7	0.20	0.10	0.050	ug/L	55	110	30	57	123	20
Water	8270D LLFS	Atrazine	1912-24-9	5.0	2.5	1.25	ug/L	40	150	30	44	142	20
Water	8270D LLFS	Benzaldehyde	100-52-7	5.0	2.5	1.25	ug/L	40	125	30	36	115	20
Water	8270D LLFS	Benzidine	92-87-5	20	5.0	1.25	ug/L	0	110	30	16	110	20
Water	8270D LLFS	Benzo(a)anthracene	56-55-3	0.20	0.10	0.025	ug/L	55	110	30	58	125	20
Water	8270D LLFS	Benzo(a)pyrene	50-32-8	0.20	0.10	0.050	ug/L	55	110	30	54	128	20
Water	8270D LLFS	Benzo(b)fluoranthene	205-99-2	0.20	0.10	0.025	ug/L	45	120	30	53	131	20
Water	8270D LLFS	Benzo(g,h,i)perylene	191-24-2	0.20	0.10	0.050	ug/L	40	125	30	50	134	20
Water	8270D LLFS	Benzo(k)fluoranthene	207-08-9	0.20	0.10	0.050	ug/L	45	125	30	57	129	20
Water	8270D LLFS	Benzoic Acid	65-85-0	20	5.0	1.25	ug/L	0	125	30	3	110	20
Water	8270D LLFS	Benzyl alcohol	100-51-6	5.0	2.5	1.25	ug/L	30	110	30	31	112	20
Water	8270D LLFS	bis(2-Chloroethoxy)methane	111-91-1	5.0	2.5	1.25	ug/L	45	105	30	48	120	20
Water	8270D LLFS	bis(2-Chloroethyl)ether (BCEE)	111-44-4	0.20	0.050	0.018	ug/L	35	110	30	43	118	20
Water	8270D LLFS	bis(2-Ethylhexyl)phthalate (BEHP)	117-81-7	5.0	2.5	1.25	ug/L	40	125	30	55	135	20

S/W	Method	Table 1B Analyte (Low-Level Full Scan)	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Water	8270D LLFS	Butyl benzyl phthalate (BBP)	85-68-7	5.0	2.5	1.25	ug/L	45	115	30	53	134	20
Water	8270D LLFS	Caprolactam	105-60-2	5.0	2.5	1.25	ug/L	5	110	30	7	110	20
Water	8270D LLFS	Carbazole	86-74-8	0.20	0.10	0.050	ug/L	50	115	30	60	122	20
Water	8270D LLFS	Chrysene	218-01-9	0.20	0.10	0.050	ug/L	55	110	30	59	123	20
Water	8270D LLFS	Dibenz(a,h)anthracene	53-70-3	0.20	0.050	0.016	ug/L	40	125	30	51	134	20
Water	8270D LLFS	Dibenzofuran (DBF)	132-64-9	0.20	0.10	0.050	ug/L	55	105	30	53	118	20
Water	8270D LLFS	Diethyl phthalate (DEP)	84-66-2	5.0	2.5	1.25	ug/L	40	120	30	56	125	20
Water	8270D LLFS	Dimethyl phthalate (DMP)	131-11-3	5.0	2.5	1.25	ug/L	25	125	30	45	127	20
Water	8270D LLFS	Di-n-butyl phthalate (DBP)	84-74-2	5.0	2.5	1.25	ug/L	55	115	30	59	127	20
Water	8270D LLFS	Di-n-octyl phthalate (DNOP)	117-84-0	5.0	2.5	1.25	ug/L	35	135	30	51	140	20
Water	8270D LLFS	Fluoranthene	206-44-0	0.20	0.10	0.050	ug/L	55	115	30	57	128	20
Water	8270D LLFS	Fluorene	86-73-7	0.20	0.10	0.050	ug/L	50	110	30	52	124	20
Water	8270D LLFS	Hexachlorobenzene (HCB)	118-74-1	0.20	0.10	0.050	ug/L	50	110	30	53	125	20
Water	8270D LLFS	Hexachlorobutadiene (HCBD)	87-68-3	0.20	0.10	0.050	ug/L	25	105	30	22	124	20
Water	8270D LLFS	Hexachlorocyclopentadiene (HCCPD)	77-47-4	0.50	0.25	0.125	ug/L	0	120	30	7	110	20
Water	8270D LLFS	Hexachloroethane (HCE)	67-72-1	0.20	0.10	0.050	ug/L	30	95	30	21	115	20
Water	8270D LLFS	Indeno(1,2,3-cd)pyrene	193-39-5	0.20	0.10	0.025	ug/L	45	125	30	52	134	20
Water	8270D LLFS	Isophorone	78-59-1	0.20	0.10	0.050	ug/L	50	110	30	42	124	20
Water	8270D LLFS	Naphthalene	91-20-3	0.20	0.10	0.050	ug/L	40	100	30	40	121	20
Water	8270D LLFS	Nitrobenzene	98-95-3	0.20	0.10	0.050	ug/L	30	110	30	45	121	20
Water	8270D LLFS	Nitrobenzene-d5 (Surrogate)	4165-60-0	0.20	0.10	0.050	ug/L	40	110		44	120	
Water	8270D LLFS	N-Nitrosodimethylamine	62-75-9	5.0	2.5	1.25	ug/L	25	110	30	13	110	20
Water	8270D LLFS	N-Nitroso-di-n-propylamine (NDPA)	621-64-7	0.20	0.10	0.028	ug/L	35	130	30	49	119	20
Water	8270D LLFS	N-nitrosodiphenylamine (NDPHA)	86-30-6	0.50	0.25	0.13	ug/L	50	110	30	51	123	20
Water	8270D LLFS	Pentachlorophenol	87-86-5	1.0	0.50	0.25	ug/L	40	115	30	35	138	20
Water	8270D LLFS	Phenanthrene	85-01-8	0.40	0.20	0.050	ug/L	50	115	30	59	120	20
Water	8270D LLFS	Phenol	108-95-2	0.40	0.20	0.10	ug/L	0	115	30	12	110	20
Water	8270D LLFS	Phenol-d6 (Advisory Surrogate)	13127-88-3	0.40	0.20	0.050	ug/L	10	115		9	110	
Water	8270D LLFS	Pyrene	129-00-0	0.20	0.10	0.050	ug/L	50	130	30	57	126	20
Water	8270D LLFS	Pyridine	110-86-1	10	5.0	1.25	ug/L	5	110	30	8	110	20
Water	8270D LLFS	Terphenyl-d14 (Surrogate)	1718-51-0	0.20	0.10	0.050	ug/L	50	135		50	134	

LL= Low Limit, UL = Upper Limit, RPD = relative percent difference, LL5/UL5/RPD5 are limits from QSM5, highlighted limits are in-house limits.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available (see table 1); otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in SOP QS03. No analysis shall be allowed by analyst until successful demonstration of capability/ performance is complete.
MDL determination	Initial demonstration with quarterly verification from LOD, LOQ. May be required annually for specific certifications.	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis with quarterly verification.	Refer to SOP QS08.			
LOQ establishment and verification	Prior to initial analysis with quarterly verification.	Refer to SOP QS08.			
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to table 8 of this SOP.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2. [Method 625 – benzidine and pentachlorophenol tailing limits are 3 and 5, respectively, when benzidine or acids are target analytes. Benzidine tailing is specific to benzidine analysis and pentachlorophenol tailing is specific to acid analyte analyses according to 625.]	Correct problem then repeat breakdown checks.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	**1.** Average response factor (RF) for SPCCs: See table 9 for 8270D SPCC analytes and limits. **2.** One option below: **Option 1:** RSD for each analyte ≤ 15%; [≤ 20% for non-DoD 8270D; or, ≤35% for non-DoD 625] **Option 2:** linear least squares regression $r^2 \geq 0.990$; **Option 3:** non-linear regression–coefficient of determination (COD) $r^2 \geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order). 3. Linear curve fits should have LOQ standard level within 70%-130% when back calculated against curve.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value [± 25% for non-DoD 8270C; or, ± 30% for non-DoD 8270D]	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the sequence CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units. Note - retention times may be updated based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping).	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time. For DoD QSM 5.0, ending CCV required within 12 hour tune time.	**1.** RF for SPCCs: SVOCs ≥ 0.050 [2,4-dinitrophenol, hexachlorocyclopentadiene, N-Nitrosodi-n-propylamine, 4-nitrophenol] **Note 1:** See table 9 for 8270D SPCC analytes and limits. **Note 2:** ≥ 0.050 for all low-level targets **2.** %Difference/Drift for all target compounds and surrogates: SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). **For QSM 5.0, ending CCV required within 12 hour tune with analyte recoveries ±50%D.**	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV. For QSM, immediate analysis of 2 passing CCVs can be used to report unqualified data.	If reanalysis cannot be performed, data should be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem should be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed, holding time has been exceeded or client has approved reporting.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or daily CCV (**± 10 seconds QSM 5.0**); EICP area within -50% to +100% of ICAL midpoint standard or daily CCV.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply qualifier to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected ≥ ½ LOQ or ≥ 1/10 the amount measured in any sample or ≥1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants (phthalates), no analytes detected ≥ LOQ.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by client or DoD (Table 1), if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch per matrix	Use LCS criteria, above.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix	MSD: For matrix evaluation, use LCS acceptance criteria above. MSD or sample duplicate: RPD ≤ 30% QSM4.2, **≤ 20% QSM 5.0** or client specified limit (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD (Table 1 above) or Client. No limits specified for Method 625.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply qualifier to all associated analytes if acceptance criteria are not met. For acid surrogate, qualify acid analytes, for base/neutral surrogates, qualify base/neutral analytes.	
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 3, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):		
Batch Number(s):		
Sequence ID:	**Run Date:**	**Instrument ID:**
Method: 8270D/8270D Low/625	**Calibration#:**	**NCR#:**

QA/QC Item	Yes	No	NA	2nd Check
1. Was the autosampler tray verified against the sequence file?				
2. Is the BFB/DFTPP tune performed every 12 hours and are the tuning criteria met? Have tailing and breakdown criteria been met?				
3. Are the % RSDs within 15% or $\geq$0.99 r2 linear corr or $\geq$0.99 quadratic COD for all analytes in the initial calibration? Are SPCC response factor criteria met? Were retention times checked for compounds with the same spectra. Were concentrations checked for compounds with different concentrations? $\leq$50% mass resolution verified for all structural isomers?				
4. Is recalculation of low standard for linear curve fits within 70%-130% (should)? List exceptions and discuss with management prior to use.				
5. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met per Table 2 of the SOP? Are SPCC response factor criteria met?				
6. Does the Continuing Calibration Standard (CCV) meet SOP Table 2 criteria and is IS within 50% to +100% of calibration curve midpoint? **QSM5.0 ending CCV (HCV) within 12 hours and analyte recoveries within $\pm$ 50%?**				
7. QSM5.0 ending CCV (HCV) within 12 hours and analyte recoveries within $\pm$ 50%?				
8. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the RL (LOD for DoD except phthalates)?				
9. Are the BS, BSD, MS, MSD within control limits and run at the desired frequency?				
10. Are all sample holding times met, analytes within calibration range, IS areas within 50% to +100% of CCV response and surrogate recoveries within limits?				
11. Sample_____shows calculation verified from raw data to final LIMS concentration.				
12. Data uploaded to LIMS with correct analyst/instrument ID reflected?				
13. Reagents/Standards verified accurate on bench sheets and in LIMS?				

Comments on any "No" response:

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Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 4 – 625 QC limits

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	100.00	0.0000	100.00	100	47-145
Acenaphthylene	100.00	0.0000	100.00	100	33-145
Anthracene	100.00	0.0000	100.00	100	27-133
Benzidine	100.00	0.0000	100.00	100	D-110
Benzo(a)anthracene	100.00	0.0000	100.00	100	33-143
Benzo(b)fluoranthene	100.00	0.0000	100.00	100	24-159
Benzo(k)fluoranthene	100.00	0.0000	100.00	100	11-162
Benzo(g,h,i)perylene	100.00	0.0000	100.00	100	D-219
Benzo(a)pyrene	100.00	0.0000	100.00	100	17-163
bis(2-Chloroethoxy)meth	100.00	0.0000	100.00	100	33-184
bis(2-Chloroethyl)ether	100.00	0.0000	100.00	100	12-158
bis(2-Chloroisopropyl)e	100.00	0.0000	100.00	100	36-166
Bis(2-ethylhexyl)phthal	100.00	0.0000	100.00	100	8-158
4-Bromophenyl-phenyleth	100.00	0.0000	100.00	100	53-127
Butylbenzylphthalate	100.00	0.0000	100.00	100	D-152
4-Chloro-3-methylphenol	100.00	0.0000	100.00	100	22-147
2-Chloronaphthalene	100.00	0.0000	100.00	100	60-118
2-Chlorophenol	100.00	0.0000	100.00	100	23-134
4-Chlorophenyl-phenylet	100.00	0.0000	100.00	100	25-158
Chrysene	100.00	0.0000	100.00	100	17-168
Dibenz(a,h)anthracene	100.00	0.0000	100.00	100	D-227
1,2-Dichlorobenzene	100.00	0.0000	100.00	100	32-129
1,3-Dichlorobenzene	100.00	0.0000	100.00	100	D-172
1,4-Dichlorobenzene	100.00	0.0000	100.00	100	20-124
3,3'-Dichlorobenzidine	100.00	0.0000	100.00	100	D-262
2,4-Dichlorophenol	100.00	0.0000	100.00	100	39-135
Diethylphthalate	100.00	0.0000	100.00	100	D-114
2,4-Dimethylphenol	100.00	0.0000	100.00	100	32-119
Dimethylphthalate	100.00	0.0000	100.00	100	D-112
Di-n-butylphthalate	100.00	0.0000	100.00	100	1-118
4,6-Dinitro-2-methylphe	100.00	0.0000	100.00	100	D-181
2,4-Dinitrophenol	100.00	0.0000	100.00	100	D-191
2,4-Dinitrotoluene	100.00	0.0000	100.00	100	39-139
2,6-Dinitrotoluene	100.00	0.0000	100.00	100	50-158
Di-n-octylphthalate	100.00	0.0000	100.00	100	4-146
Fluoranthene	100.00	0.0000	100.00	100	26-137
Fluorene	100.00	0.0000	100.00	100	59-121
Hexachlorobenzene	100.00	0.0000	100.00	100	D-152
Hexachlorobutadiene	100.00	0.0000	100.00	100	24-116
Hexachlorocyclopentadie	100.00	0.0000	100.00	100	15- 70
Hexachloroethane	100.00	0.0000	100.00	100	40-113
Indeno(1,2,3-cd)pyrene	100.00	0.0000	100.00	100	D-171
Isophorone	100.00	0.0000	100.00	100	21-196
Naphthalene	100.00	0.0000	100.00	100	21-133
Nitrobenzene	100.00	0.0000	100.00	100	35-180
2-Nitrophenol	100.00	0.0000	100.00	100	29-182
4-Nitrophenol	100.00	0.0000	100.00	100	D-132
N-Nitroso-di-methylamin	100.00	0.0000	100.00	100	29- 66
N-Nitrosodiphenylamine	100.00	0.0000	100.00	100	23-100
N-Nitroso-di-n-propylam	100.00	0.0000	100.00	100	D-230
Pentachlorophenol	100.00	0.0000	100.00	100	14-176
Phenanthrene	100.00	0.0000	100.00	100	54-120
Phenol	100.00	0.0000	100.00	100	5-112
Pyrene	100.00	0.0000	100.00	100	52-115
1,2,4-Trichlorobenzene	100.00	0.0000	100.00	100	44-142
2,4,6-Trichlorophenol	100.00	0.0000	100.00	100	37-144

Table 5 - BNA STANDARDS USED

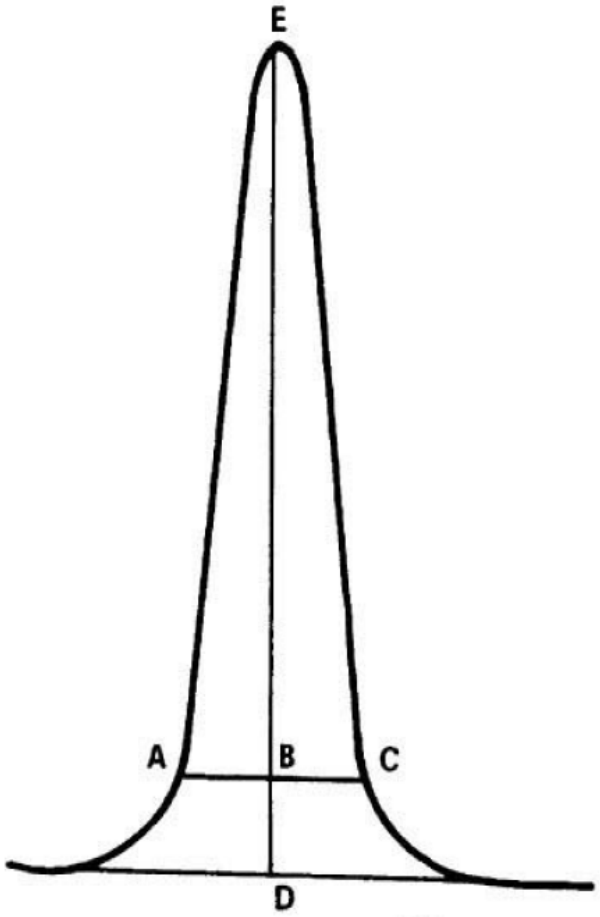
base/neutral mix (2000ppm)		acids mix (2000ppm)
bis(2-Chloroethyl)ether		2,4-Dinitrophenol
bis(2-Chloroisopropyl)ether		2-Methylphenol
1,3-Dichlorobenzene		4-Methylphenol (as 3&4-Methylphenol)
1,2-Dichlorobenzene		Benzoic acid
1,4-Dichlorobenzene		4,6-Dinitro-2-methylphenol
Hexachloroethane		4-Nitrophenol
N-Nitroso-di-methylamine		2,4,5-Trichlorophenol
N-Nitroso-di-n-propylamine		2,4,6-Trichlorophenol
2,4-Dinitrotoluene		Phenol
2,6-Dinitrotoluene		Pentachlorophenol
Fluorene		2-Nitrophenol
Dimethylphthalate		4-Chloro-3-methylphenol
Hexachlorocyclopentadiene		2,4-Dichlorophenol
Anthracene		2,4-Dimethylphenol
4-Bromophenyl-phenylether		Benzoic acid
Di-n-butylphthalate		
bis(2-Chloroethoxy)methane		
1,2-Diphenylhydrazine		**semivoa misc. mix(2000ppm)**
Fluoranthene		Aniline
Hexachlorobenzene		Benzyl alcohol
N-Nitrosodiphenylamine		Carbazole
Phenanthrene		4-Chloroaniline
Hexachlorobutadiene		Dibenzofuran
Isophorone		2-Methylnaphthalene
Naphthalene		2-Nitroaniline
Nitrobenzene		3-Nitroaniline
1,2,4-Trichlorobenzene		4-Nitroaniline
Acenaphthene		Pyridine
Acenaphthylene		
2-Chloronaphthalene		
4-Chlorophenyl-phenylether		**Benzidine mix (2000ppm)**
Diethylphthalate		Benzidine
Benzo(a)anthracene		3,3'-Dichlorobenzidine
Bis(2-ethylhexyl)phthalate		
Butylbenzylphthalate		
Chrysene		**Individual or misc. mixes (2000/5000/20,000ppm)**
p-(Dimethylamino)azobenzene		Caprolactam
Pyrene		Benzaldehyde
Benzo(b)fluoranthene		Atrazine
Benzo(k)fluoranthene		1,1'-Biphenyl
Benzo(g,h,i)perylene		1,4-Dioxane
Benzo(a)pyrene		1-methylnapthalene
Dibenz(a,h)anthracene		2,6-dichlorophenol
Di-n-octylphthalate		2,3,4,6-tetrachlorophenol
Indeno(1,2,3-cd)pyrene		

BNA internals (2000ppm)		Acid surrogate (7500ppm)
1,4-Dichlorobenzene-d4 (I.S)(1)		2-Fluorophenol (S)
Naphthalene-d8 (I.S)(35)		Phenol-d6 (S)
Acenaphthene-d10 (I.S) (59)		2,4,6-Tribromophenol (S)
Phenanthrene-d10 (I.S) (79)		2,-Chlorophenol-d4 (S)
Chrysene-d12 (I.S) (92))		BN surrogate (5000ppm)
Perylene-d12 (I.S) (101)		Nitrobenzene-d5 (S)
		Terphenyl-d14 (S)
		2-Fluorobiphenyl (S)
		1,2-Dichlorobenzene-d4 (S)

Table 6 - Analyte	ISRef	QIon
1,1-Biphenyl	902	154
1,2,4,5-Tetrachlorobenzene	901	216
1,2,4-Trichlorobenzene	901	180
1,2-Dichlorobenzene	900	146
1,2-Diphenylhydrazine	903	77
1,3-Dichlorobenzene	900	146
1,4-Dichlorobenzene	900	146
1,4-Dichlorobenzene-d4	**900**	152
1,4-Dioxane	900	88
1-Methylnaphthalene	901	141
2,2'-Oxybis-1-chloropropane	900	45
2,3,4,6-Tetrachlorophenol	902	232
2,4,5-Trichlorophenol	902	196
2,4,6-Tribromophenol (surr)	903	330
2,4,6-Trichlorophenol	902	196
2,4-Dichlorophenol	901	162
2,4-Dimethylphenol	901	107
2,4-Dinitrophenol	902	184
2,4-Dinitrotoluene	902	165
2,6-Dichlorophenol	901	162
2,6-Dinitrotoluene	902	165
2-Chloronaphthalene	902	162
2-Chlorophenol	900	128
2-Fluorobiphenyl (surr)	902	172
2-Fluorophenol (surr)	900	112
2-Methylnaphthalene	901	141
2-Methylphenol	900	108
2-Nitroaniline	902	65
2-Nitrophenol	901	139
3,3'-Dichlorobenzidine	904	252
3-Methylphenol/4-Methylphenol	900	108
3-Nitroaniline	902	138
4,6-Dinitro-2-methylphenol	903	198
4-Bromophenyl-phenylether	903	248
4-Chloro-3-methylphenol	901	107
4-Chloroaniline	901	127
4-Chlorophenyl phenyl ether	902	204
4-Nitroaniline	902	138
4-Nitrophenol	902	65
Acenaphthene	902	153
Acenaphthene-d10	**902**	164
Acenaphthylene	902	152
Acetophenone	900	105
Aniline	900	93
Anthracene	903	178
Atrazine	903	200
Benzaldehyde	900	106
Benzidine	904	184

Table 6 - Analyte	ISRef	QIon
Benzo(a)anthracene	904	228
Benzo(a)pyrene	905	252
Benzo(b)fluoranthene	905	252
Benzo(g,h,i)perylene	905	276
Benzo(k)fluoranthene	905	252
Benzoic acid	901	105
Benzyl alcohol	900	108
Bis(2-chloroethoxy)methane	901	93
Bis(2-chloroethyl)ether	900	93
Bis(2-ethylhexyl)phthalate	904	149
Butylbenzylphthalate	904	149
Caprolactam	901	113
Carbazole	903	167
Chrysene	904	228
Chrysene-d12	**904**	240
Dibenz(a,c)anthracene	905	278
Dibenz(a,h)anthracene	905	278
Dibenzofuran	902	168
Diethylphthalate	902	149
Dimethyl phthalate	902	163
Di-n-butylphthalate	903	149
Di-n-octylphthalate	905	149
Fluoranthene	903	202
Fluorene	902	166
Hexachlorobenzene	903	284
Hexachlorobutadiene	901	225
Hexachlorocyclopentadiene	902	237
Hexachloroethane	900	117
Indeno(1,2,3-cd)pyrene	905	276
Isophorone	901	82
Naphthalene	901	128
Naphthalene-d8	**901**	136
Nitrobenzene	901	77
Nitrobenzene-d5 (surr)	901	82
N-Nitrosodimethylamine	900	42
N-Nitroso-di-n-propylamine	900	70
N-Nitrosodiphenylamine	903	169
Pentachlorophenol	903	266
Perylene-d12	**905**	264
Perylene	905	252
Phenanthrene	903	178
Phenanthrene-d10	**903**	188
Phenol	900	94
Phenol-d6 (surr)	900	99
Pyrene	904	202
Pyridine	900	79
Terphenyl-d14 (surr)	904	244

FIGURE 1
TAILING FACTOR CALCULATION



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

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

Table 8, DFTPP Tuning Criteria

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Note: While 8270D table 3 indicates different criteria, section 11.3.1.2 allows the use of alternate criteria.

Table 9, 8270D SPCC limits

Analyte	SPCC
1,1-Biphenyl	0.010
1,2,4,5-Tetrachlorobenzene	0.010
1,2,4-Trichlorobenzene	0.010
1,2-Dichlorobenzene	0.010
1,2-Diphenylhydrazine	0.010
1,3-Dichlorobenzene	0.010
1,4-Dichlorobenzene	0.010
1,4-Dichlorobenzene-d4	0.010
1,4-Dioxane	0.010
1-Methylnaphthalene	0.010
2,2'-Oxybis-1-chloropropane	0.010
2,3,4,6-Tetrachlorophenol	0.010
2,4,5-Trichlorophenol	0.200
2,4,6-Tribromophenol	0.010
2,4,6-Trichlorophenol	0.200
2,4-Dichlorophenol	0.200
2,4-Dimethylphenol	0.200
2,4-Dinitrophenol **(8270C 0.050 used)**	*0.010*
2,4-Dinitrotoluene	0.200
2,6-Dichlorophenol	0.010
2,6-Dinitrotoluene	0.200
2-Chloronaphthalene	0.800
2-Chlorophenol	0.800
2-Fluorobiphenyl	0.010
2-Fluorophenol	0.010
2-Methylnaphthalene	0.400
2-Methylphenol	0.700
2-Nitroaniline	0.010
2-Nitrophenol	0.100
3,3'-Dichlorobenzidine	0.010
3-Methylphenol/4-Methylphenol	0.010
3-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenylether	0.100
4-Chloro-3-methylphenol	0.200
4-Chloroaniline	0.010
4-Chlorophenyl phenyl ether	0.400
4-Nitroaniline	0.010
4-Nitrophenol **(8270C 0.050 used)**	*0.010*
Acenaphthene	0.900
Acenaphthene-d10	0.010
Acenaphthylene	0.900
Acetophenone	0.010
Aniline	0.010
Anthracene	0.700
Atrazine	0.010
Benzaldehyde	0.010
Benzidine	0.010
Benzo(a)anthracene	0.800
Benzo(a)pyrene	0.700
Benzo(b)fluoranthene	0.700
Benzo(g,h,i)perylene	0.500
Benzo(k)fluoranthene	0.700
Benzoic acid	0.010
Benzyl alcohol	0.010
Bis(2-chloroethoxy)methane	0.300
Bis(2-chloroethyl)ether	0.700
Bis(2-ethylhexyl)phthalate	0.010
Butylbenzylphthalate	0.010
Caprolactam	0.010
Carbazole	0.010
Chrysene	0.700
Chrysene-d12	0.010
Dibenz(a,h)anthracene	0.400
Dibenzofuran	0.800
Diethylphthalate	0.010
Dimethyl phthalate	0.010
Di-n-butylphthalate	0.010
Di-n-octylphthalate	0.010
Fluoranthene	0.600
Fluorene	0.900
Hexachlorobenzene	0.100
Hexachlorobutadiene	0.010
Hexachlorocyclopentadiene	0.050
Hexachloroethane	0.300
Indeno(1,2,3-cd)pyrene	0.500
Isophorone	0.400
Naphthalene	0.700
Naphthalene-d8	0.010
Nitrobenzene	0.200
Nitrobenzene-d5	0.010
N-Nitrosodimethylamine	0.010
N-Nitroso-di-n-propylamine	0.500
N-Nitrosodiphenylamine	0.010
Pentachlorophenol	0.050
Perylene-d12	0.010
Phenanthrene	0.700
Phenanthrene-d10	0.010
Phenol	0.800
Phenol-d6	0.010
Pyrene	0.600
Pyridine	0.010
Terphenyl-d14	0.010

Table 10, Poor Performing Analytes
1,1-Biphenyl
1,2,4,5-Tetrachlorobenzene
1,2-Diphenyl hydrazine
2,2'-Oxybis-1-chloropropane
2,4-Dinitrophenol,
2,6-Dichlorophenol
2-Nitroaniline
3,3'-Dichlorobenzidine
3-Nitroaniline
4,6-Dinitro-2-methylphenol
4-Chloro-3-methylphenol,
4-Chloroaniline
4-Nitroaniline
4-Nitrophenol
Acetophenone
Atrazine
Benzaldehyde
Benzidine
Benzoic acid,
Benzyl alcohol
Bis(2-ethylhexyl)phthalate
Butylbenzylphthalate
Caprolactam
Carbazole
Diethylphthalate
Dimethyl phthalate
Di-n-butylphthalate
Di-n-octylphthalate
Hexachlorobutadiene
Hexachlorocyclopentadiene
N-Nitrosodimethylamine
N-Nitroso-di-n-propylamine
N-Nitrosodiphenylamine
Pentachlorophenol,
Pyridine

Analytical Standard Record

Empirical Laboratories, LLC

15G0553

Description:	SV_DFTPP TUNE	Expires:	01/25/2016
Standard Type:	MS Tune Solution	Prepared:	07/25/2015
Solvent:	dichloromethane	Prepared By:	Randy Ward
Final Volume (mls):	1	Department:	MS
Vials:	1	Last Edit:	07/27/2015 16:03 by RDW

Analyte	CAS Number	Concentration	Units
Pentachlorophenol	87-86-5	50	ug/mL
DFTPP	5074-71-5	50	ug/mL
DDT	50-29-3	50	ug/mL
Benzidine	92-87-5	50	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
15G0496	8270 Tuning Mix	08/12/201	Mark Cobb	01/23/201	07/23/2015 13:45 by MEC	0.1

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15A0169

Description:	SV_ Internal Std Mix @ 4000	Expires:	09/30/2020
Standard Type:	Internal Standard	Prepared:	01/10/2015
Solvent:	Methylene Chloride	Prepared By:	Randy Ward
Final Volume (mls):	1	Department:	MS
Vials:	1	Last Edit:	01/10/2015 14:14 by RDW

Restek cat# 31006

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	4000	ug/mL
Perylene-d12	NA	4000	ug/mL
Naphthalene-d8	NA	4000	ug/mL
Chrysene-d12	NA	4000	ug/mL
Acenaphthene-d10	NA	4000	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	4000	ug/mL

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0299

Description:	bna_cal1_5.0 #1	Expires:	06/11/2015
Standard Type:	Calibration Standa	Prepared:	04/14/2015
Solvent:	dichloromethane	Prepared By:	Randy Ward
Final Volume (mls):	1	Department:	MS
Vials:	1	Last Edit:	04/21/2015 14:39 by RDW

Analyte	CAS Number	Concentration	Units
4-Bromophenyl-phenylether	101-55-3	5	ug/mL
Aniline	62-53-3	5	ug/mL
2-Fluorophenol	367-12-4	10	ug/mL
2-Methylnaphthalene	91-57-6	5	ug/mL
2-Methylphenol	95-48-7	5	ug/mL
2-Nitroaniline	88-74-4	5	ug/mL
2-Nitrophenol	88-75-5	5	ug/mL
3-Methylphenol	108-39-4	5	ug/mL
3-Methylphenol/4-Methylphenol	108-39-4/106	5	ug/mL
2-Chlorophenol	95-57-8	5	ug/mL
4,6-Dinitro-2-methylphenol	534-52-1	5	ug/mL
2-Chloronaphthalene	91-58-7	5	ug/mL
4-Chloro-3-methylphenol	59-50-7	5	ug/mL
4-Chloroaniline	106-47-8	5	ug/mL
4-Chlorophenyl phenyl ether	7005-72-3	5	ug/mL
4-Methylphenol	106-44-5	5	ug/mL
4-Nitroaniline	100-01-6	5	ug/mL
4-Nitrophenol	100-02-7	5	ug/mL
Acenaphthene	83-32-9	5	ug/mL
1,2,4-Trichlorobenzene	120-82-1	5	ug/mL
3-Nitroaniline	99-09-2	5	ug/mL
2,3,5,6-Tetrachlorophenol	935-95-5	5	ug/mL
1,2-Dichlorobenzene	95-50-1	5	ug/mL
1,2-Dinitrobenzene	528-29-0	5	ug/mL
1,2-Diphenylhydrazine	122-66-7	5	ug/mL
1,3-Dichlorobenzene	541-73-1	5	ug/mL
1,3-Dinitrobenzene	99-65-0	5	ug/mL
1,4-Dichlorobenzene	106-46-7	5	ug/mL
1,4-Dinitrobenzene	100-25-4	5	ug/mL
1-Methylnaphthalene	90-12-0	5	ug/mL

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0299

2-Fluorobiphenyl	321-60-8	5	ug/mL
2,3,4,6-Tetrachlorophenol	58-90-2	5	ug/mL
Anthracene	120-12-7	5	ug/mL
2,4,5-Trichlorophenol	95-95-4	5	ug/mL
2,4,6-Tribromophenol	118-79-6	10	ug/mL
2,4,6-Trichlorophenol	88-06-2	5	ug/mL
2,4-Dichlorophenol	120-83-2	5	ug/mL
2,4-Dimethylphenol	105-67-9	5	ug/mL
2,4-Dinitrophenol	51-28-5	5	ug/mL
2,4-Dinitrotoluene	121-14-2	5	ug/mL
2,6-Dinitrotoluene	606-20-2	5	ug/mL
2,2'-Oxybis-1-chloropropane	108-60-1	5	ug/mL
Naphthalene	91-20-3	5	ug/mL
Acenaphthylene	208-96-8	5	ug/mL
Hexachlorobenzene	118-74-1	5	ug/mL
Hexachlorobutadiene	87-68-3	5	ug/mL
Hexachlorocyclopentadiene	77-47-4	5	ug/mL
Hexachloroethane	67-72-1	5	ug/mL
Indeno(1,2,3-cd)pyrene	193-39-5	5	ug/mL
Isophorone	78-59-1	5	ug/mL
N-Nitroso-di-n-propylamine	621-64-7	5	ug/mL
Fluoranthene	206-44-0	5	ug/mL
N-Nitrosodiphenylamine	86-30-6	5	ug/mL
Dimethyl phthalate	131-11-3	5	ug/mL
Nitrobenzene	98-95-3	5	ug/mL
Nitrobenzene-d5	4165-60-0	5	ug/mL
Pentachlorophenol	87-86-5	5	ug/mL
Phenanthrene	85-01-8	5	ug/mL
Phenol	108-95-2	5	ug/mL
Phenol-d6	13127-88-3	10	ug/mL
Pyrene	129-00-0	5	ug/mL
Pyridine	110-86-1	5	ug/mL
N-Nitrosodimethylamine	62-75-9	5	ug/mL
Bis(2-ethylhexyl)phthalate	117-81-7	5	ug/mL
Benzo(a)anthracene	56-55-3	5	ug/mL
Benzo(a)pyrene	50-32-8	5	ug/mL
Benzo(b)fluoranthene	205-99-2	5	ug/mL
Benzo(g,h,i)perylene	191-24-2	5	ug/mL
Benzo(k)fluoranthene	207-08-9	5	ug/mL

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0299

Benzoic acid	65-85-0	5	ug/mL
Benzyl alcohol	100-51-6	5	ug/mL
Bis(2-chloroethoxy)methane	111-91-1	5	ug/mL
Fluorene	86-73-7	5	ug/mL
Bis(2-ethylhexyl)adipate (Outside Certif	103-23-1	5	ug/mL
Terphenyl-d14	1718-51-0	5	ug/mL
Butylbenzylphthalate	85-68-7	5	ug/mL
Carbazole	86-74-8	5	ug/mL
Chrysene	218-01-9	5	ug/mL
Di-n-butylphthalate	84-74-2	5	ug/mL
Di-n-octylphthalate	117-84-0	5	ug/mL
Dibenz(a,h)anthracene	53-70-3	5	ug/mL
Dibenzofuran	132-64-9	5	ug/mL
Diethylphthalate	84-66-2	5	ug/mL
Bis(2-chloroethyl)ether	111-44-4	5	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
15D0139	SV_Intermediate BNA Std-1	04/07/201	Randy Ward	06/11/201	04/21/2015 14:39 by RDW	0.025

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0308

Description:	bna_cal9_5.0 #2	Expires:	10/07/2015
Standard Type:	Calibration Standa	Prepared:	04/14/2015
Solvent:	dichloromethane	Prepared By:	Randy Ward
Final Volume (mls):	1	Department:	MS
Vials:	1	Last Edit:	04/24/2015 17:17 by RDW

Analyte	CAS Number	Concentration	Units
Caprolactam	105-60-2	5	ug/mL
1,2,4,5-Tetrachlorobenzene	95-94-3	5	ug/mL
1,4-Dioxane	123-91-1	5	ug/mL
2,6-Dichlorophenol	87-65-0	5	ug/mL
2,6-Dimethylphenol (Outside Certificatio	576-26-1	5	ug/mL
3,3'-Dichlorobenzidine	91-94-1	5	ug/mL
3,4-Dimethylphenol (Outside Certificatio	95-65-8	5	ug/mL
3-Chlorophenol (Outside Certification)	108-43-0	5	ug/mL
Acetophenone	98-86-2	5	ug/mL
Alachlor (Outside Certification)	15972-60-8	5	ug/mL
Atrazine	1912-24-9	5	ug/mL
Benzaldehyde	100-52-7	5	ug/mL
1,1-Biphenyl	92-52-4	5	ug/mL
Bis(2-ethylhexyl)adipate (Outside Certif	103-23-1	5	ug/mL
Sulfotep (Outside Certification)	3689-24-5	5	ug/mL
Chlorobenzilate (Outside Certification)	510-15-6	5	ug/mL
Chlorpyrifos (Outside Certification)	2921-88-2	5	ug/mL
Diallate (Outside Certification)	2303-16-4	5	ug/mL
Dimethoate (Outside Certification)	60-51-5	5	ug/mL
Disulfoton (Outside Certification)	298-04-4	5	ug/mL
Malathion (Outside Certification)	121-75-5	5	ug/mL
Methyl parathion (Outside Certification)	298-00-0	5	ug/mL
Parathion (Outside Certification)	56-38-2	5	ug/mL
Pentachlorobenzene (Outside Certificatio	608-93-5	5	ug/mL
Phorate (Outside Certification)	298-02-2	5	ug/mL
Pronamide (Outside Certification)	23950-58-5	5	ug/mL
Simazine (Outside Certification)	122-34-9	5	ug/mL
Benzidine	92-87-5	5	ug/mL

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0308

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
15B0354	Benzidine Mix	02/18/201	Randy Ward	10/07/201	04/10/2015 16:45 by RDW	0.0025
15C0648	Alachlor	03/27/201	Randy Ward	09/01/201	04/24/2015 17:16 by RDW	0.005
15C0650	Chlorobenzilate	03/27/201	Randy Ward	10/01/201	04/24/2015 17:17 by RDW	0.005
15D0140	SV_Intermediate BNA Std-2	04/07/201	Randy Ward	10/07/201	04/07/2015 14:31 by RDW	0.025
15D0141	SV_ Custom Pest Mix	04/07/201	Randy Ward	03/23/201	04/24/2015 16:28 by RDW	0.005

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15F0354

Description:	SV_low IS virtual	Expires:	12/04/2015
Standard Type:	Internal Standard	Prepared:	06/04/2015
Solvent:	dichloromethane	Prepared By:	Mark Cobb
Final Volume (mls):	1	Department:	MS
Vials:	1	Last Edit:	06/11/2015 11:21 by MEC

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	2	ug/mL
Perylene-d12	NA	2	ug/mL
Naphthalene-d8	NA	2	ug/mL
Chrysene-d12	NA	2	ug/mL
Acenaphthene-d10	NA	2	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	2	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
15F0133	SV_IS_LOW@200	06/04/201	Mark Cobb	12/04/201	06/04/2015 16:45 by MEC	0.01

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0690

Description:	bna_cal1	Expires:	10/20/2015
Standard Type:	Calibration Standa	Prepared:	04/23/2015
Solvent:	dichloromethane	Prepared By:	Mark Cobb
Final Volume (mls):	1	Department:	MS
Vials:	1	Last Edit:	07/21/2015 14:06 by MKM

Analyte	CAS Number	Concentration	Units
3-Methylphenol/4-Methylphenol	108-39-4/106	0.1	ug/mL
2-Chloronaphthalene	91-58-7	0.1	ug/mL
2-Chlorophenol	95-57-8	0.1	ug/mL
2-Fluorobiphenyl	321-60-8	0.1	ug/mL
2-Fluorophenol	367-12-4	0.2	ug/mL
2-Methylnaphthalene	91-57-6	0.1	ug/mL
2-Methylphenol	95-48-7	0.1	ug/mL
2-Nitroaniline	88-74-4	0.1	ug/mL
2-Nitrophenol	88-75-5	0.1	ug/mL
1,1-Biphenyl	92-52-4	0.1	ug/mL
3-Methylphenol	108-39-4	0.1	ug/mL
2,4-Dinitrotoluene	121-14-2	0.1	ug/mL
3-Nitroaniline	99-09-2	0.1	ug/mL
4,6-Dinitro-2-methylphenol	534-52-1	0.1	ug/mL
4-Bromophenyl-phenylether	101-55-3	0.1	ug/mL
4-Chloro-3-methylphenol	59-50-7	0.1	ug/mL
4-Chloroaniline	106-47-8	0.1	ug/mL
4-Chlorophenyl phenyl ether	7005-72-3	0.1	ug/mL
4-Methylphenol	106-44-5	0.1	ug/mL
4-Nitroaniline	100-01-6	0.1	ug/mL
4-Nitrophenol	100-02-7	0.1	ug/mL
3,3'-Dichlorobenzidine	91-94-1	0.1	ug/mL
2,2'-Oxybis-1-chloropropane	108-60-1	0.1	ug/mL
1,2,4,5-Tetrachlorobenzene	95-94-3	0.1	ug/mL
1,2,4-Trichlorobenzene	120-82-1	0.1	ug/mL
1,2-Dichlorobenzene	95-50-1	0.1	ug/mL
1,2-Dinitrobenzene	528-29-0	0.1	ug/mL
1,2-Diphenylhydrazine	122-66-7	0.1	ug/mL
1,3-Dichlorobenzene	541-73-1	0.1	ug/mL
1,3-Dinitrobenzene	99-65-0	0.1	ug/mL

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0690

1,4-Dichlorobenzene	106-46-7	0.1	ug/mL
1,4-Dinitrobenzene	100-25-4	0.1	ug/mL
2,6-Dinitrotoluene	606-20-2	0.1	ug/mL
1-Methylnaphthalene	90-12-0	0.1	ug/mL
2,6-Dichlorophenol	87-65-0	0.1	ug/mL
2,3,4,6-Tetrachlorophenol	58-90-2	0.1	ug/mL
2,3,5,6-Tetrachlorophenol	935-95-5	0.1	ug/mL
2,4,5-Trichlorophenol	95-95-4	0.1	ug/mL
2,4,6-Tribromophenol	118-79-6	0.2	ug/mL
2,4,6-Trichlorophenol	88-06-2	0.1	ug/mL
2,4-Dichlorophenol	120-83-2	0.1	ug/mL
2,4-Dimethylphenol	105-67-9	0.1	ug/mL
2,4-Dinitrophenol	51-28-5	0.1	ug/mL
Acetophenone	98-86-2	0.1	ug/mL
1,4-Dioxane	123-91-1	0.1	ug/mL
Naphthalene	91-20-3	0.1	ug/mL
Acenaphthene	83-32-9	0.1	ug/mL
Fluorene	86-73-7	0.1	ug/mL
Hexachlorobenzene	118-74-1	0.1	ug/mL
Hexachlorobutadiene	87-68-3	0.1	ug/mL
Hexachlorocyclopentadiene	77-47-4	0.1	ug/mL
Hexachloroethane	67-72-1	0.1	ug/mL
Indeno(1,2,3-cd)pyrene	193-39-5	0.1	ug/mL
Isophorone	78-59-1	0.1	ug/mL
N-Nitroso-di-n-propylamine	621-64-7	0.1	ug/mL
Dimethyl phthalate	131-11-3	0.1	ug/mL
N-Nitrosodiphenylamine	86-30-6	0.1	ug/mL
Diethylphthalate	84-66-2	0.1	ug/mL
Nitrobenzene	98-95-3	0.1	ug/mL
Nitrobenzene-d5	4165-60-0	0.1	ug/mL
Pentachlorophenol	87-86-5	0.1	ug/mL
Phenanthrene	85-01-8	0.1	ug/mL
Phenol	108-95-2	0.1	ug/mL
Phenol-d6	13127-88-3	0.2	ug/mL
Phenol-d6 (advisory)	13127-88-3	0.2	ug/mL
Pyrene	129-00-0	0.1	ug/mL
Pyridine	110-86-1	0.1	ug/mL
N-Nitrosodimethylamine	62-75-9	0.1	ug/mL
Bis(2-chloroethyl)ether	111-44-4	0.1	ug/mL

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0690

Terphenyl-d14	1718-51-0	0.1	ug/mL
Aniline	62-53-3	0.1	ug/mL
Anthracene	120-12-7	0.1	ug/mL
Benzidine	92-87-5	0.1	ug/mL
Benzo(a)anthracene	56-55-3	0.1	ug/mL
Benzo(a)pyrene	50-32-8	0.1	ug/mL
Benzo(b)fluoranthene	205-99-2	0.1	ug/mL
Benzo(g,h,i)perylene	191-24-2	0.1	ug/mL
Benzo(k)fluoranthene	207-08-9	0.1	ug/mL
Fluoranthene	206-44-0	0.1	ug/mL
Bis(2-chloroethoxy)methane	111-91-1	0.1	ug/mL
Acenaphthylene	208-96-8	0.1	ug/mL
Bis(2-ethylhexyl)phthalate	117-81-7	0.1	ug/mL
Bis-(2-Ethylhexyl) Adipate	103-23-1	0.1	ug/mL
Butylbenzylphthalate	85-68-7	0.1	ug/mL
Carbazole	86-74-8	0.1	ug/mL
Chrysene	218-01-9	0.1	ug/mL
Di-n-butylphthalate	84-74-2	0.1	ug/mL
Di-n-octylphthalate	117-84-0	0.1	ug/mL
Dibenz(a,h)anthracene	53-70-3	0.1	ug/mL
Dibenzofuran	132-64-9	0.1	ug/mL
Benzyl alcohol	100-51-6	0.1	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
15D0510	Semi-volatile bna low intermediate std.	04/20/201	Mark Cobb	10/20/201	07/21/2015 14:06 by MKM	0.001

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0699

Description:	bna_calA	Expires:	10/20/2015
Standard Type:	Calibration Standa	Prepared:	04/23/2015
Solvent:	dichloromethane	Prepared By:	Mark Cobb
Final Volume (mls):	1	Department:	MS
Vials:	1	Last Edit:	04/28/2015 14:00 by MEC

Analyte	CAS Number	Concentration	Units
Caprolactam	105-60-2	0.1	ug/mL
Benzoic acid	65-85-0	0.1	ug/mL
Benzaldehyde	100-52-7	0.1	ug/mL
Atrazine	1912-24-9	0.1	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
15D0511	SV_Intermediate BNA low Std 2	04/20/201	Mark Cobb	10/20/201	04/20/2015 16:31 by MEC	0.001

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0711

Description:	SV_BNA_LOW_ICV1	Expires:	07/27/2015
Standard Type:	Calibration Standa	Prepared:	04/28/2015
Solvent:	dichloromethane	Prepared By:	Mark Cobb
Final Volume (mls):	1	Department:	MS
Vials:	1	Last Edit:	07/21/2015 14:05 by MKM

Analyte	CAS Number	Concentration	Units
4-Bromophenyl-phenylether	101-55-3	10	ug/mL
Aniline	62-53-3	10	ug/mL
2-Fluorophenol	367-12-4	20	ug/mL
2-Methylnaphthalene	91-57-6	10	ug/mL
2-Methylphenol	95-48-7	10	ug/mL
2-Nitroaniline	88-74-4	10	ug/mL
2-Nitrophenol	88-75-5	10	ug/mL
3-Methylphenol	108-39-4	10	ug/mL
3-Methylphenol/4-Methylphenol	108-39-4/106	10	ug/mL
2-Chlorophenol	95-57-8	10	ug/mL
4,6-Dinitro-2-methylphenol	534-52-1	10	ug/mL
2-Chloronaphthalene	91-58-7	10	ug/mL
4-Chloro-3-methylphenol	59-50-7	10	ug/mL
4-Chloroaniline	106-47-8	10	ug/mL
4-Chlorophenyl phenyl ether	7005-72-3	10	ug/mL
4-Methylphenol	106-44-5	10	ug/mL
4-Nitroaniline	100-01-6	10	ug/mL
4-Nitrophenol	100-02-7	10	ug/mL
Acenaphthene	83-32-9	10	ug/mL
1,2,4-Trichlorobenzene	120-82-1	10	ug/mL
3-Nitroaniline	99-09-2	10	ug/mL
2,3,5,6-Tetrachlorophenol	935-95-5	10	ug/mL
1,2-Dichlorobenzene	95-50-1	10	ug/mL
1,2-Dinitrobenzene	528-29-0	10	ug/mL
1,2-Diphenylhydrazine	122-66-7	10	ug/mL
1,3-Dichlorobenzene	541-73-1	10	ug/mL
1,3-Dinitrobenzene	99-65-0	10	ug/mL
1,4-Dichlorobenzene	106-46-7	10	ug/mL
1,4-Dinitrobenzene	100-25-4	10	ug/mL
1-Methylnaphthalene	90-12-0	10	ug/mL

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0711

2-Fluorobiphenyl	321-60-8	10	ug/mL
2,3,4,6-Tetrachlorophenol	58-90-2	10	ug/mL
Anthracene	120-12-7	10	ug/mL
2,4,5-Trichlorophenol	95-95-4	10	ug/mL
2,4,6-Tribromophenol	118-79-6	20	ug/mL
2,4,6-Trichlorophenol	88-06-2	10	ug/mL
2,4-Dichlorophenol	120-83-2	10	ug/mL
2,4-Dimethylphenol	105-67-9	10	ug/mL
2,4-Dinitrophenol	51-28-5	10	ug/mL
2,4-Dinitrotoluene	121-14-2	10	ug/mL
2,6-Dinitrotoluene	606-20-2	10	ug/mL
2,2'-Oxybis-1-chloropropane	108-60-1	10	ug/mL
Nitrobenzene	98-95-3	10	ug/mL
Acenaphthylene	208-96-8	10	ug/mL
Hexachlorobutadiene	87-68-3	10	ug/mL
Hexachlorocyclopentadiene	77-47-4	10	ug/mL
Hexachloroethane	67-72-1	10	ug/mL
Indeno(1,2,3-cd)pyrene	193-39-5	10	ug/mL
Isophorone	78-59-1	10	ug/mL
N-Nitroso-di-n-propylamine	621-64-7	10	ug/mL
N-Nitrosodimethylamine	62-75-9	10	ug/mL
Fluorene	86-73-7	10	ug/mL
Naphthalene	91-20-3	10	ug/mL
Fluoranthene	206-44-0	10	ug/mL
Nitrobenzene-d5	4165-60-0	10	ug/mL
Pentachlorophenol	87-86-5	10	ug/mL
Phenanthrene	85-01-8	10	ug/mL
Phenol	108-95-2	10	ug/mL
Phenol-d6	13127-88-3	20	ug/mL
Phenol-d6 (advisory)	13127-88-3	20	ug/mL
Pyrene	129-00-0	10	ug/mL
Pyridine	110-86-1	10	ug/mL
N-Nitrosodiphenylamine	86-30-6	10	ug/mL
Butylbenzylphthalate	85-68-7	10	ug/mL
Benzo(a)anthracene	56-55-3	10	ug/mL
Benzo(a)pyrene	50-32-8	10	ug/mL
Benzo(b)fluoranthene	205-99-2	10	ug/mL
Benzo(g,h,i)perylene	191-24-2	10	ug/mL
Benzo(k)fluoranthene	207-08-9	10	ug/mL

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0711

Benzyl alcohol	100-51-6	10	ug/mL
Bis(2-chloroethoxy)methane	111-91-1	10	ug/mL
Bis(2-chloroethyl)ether	111-44-4	10	ug/mL
Hexachlorobenzene	118-74-1	10	ug/mL
Bis(2-ethylhexyl)phthalate	117-81-7	10	ug/mL
Terphenyl-d14	1718-51-0	10	ug/mL
Carbazole	86-74-8	10	ug/mL
Chrysene	218-01-9	10	ug/mL
Di-n-butylphthalate	84-74-2	10	ug/mL
Di-n-octylphthalate	117-84-0	10	ug/mL
Dibenz(a,h)anthracene	53-70-3	10	ug/mL
Dibenzofuran	132-64-9	10	ug/mL
Diethylphthalate	84-66-2	10	ug/mL
Dimethyl phthalate	131-11-3	10	ug/mL
Bis(2-ethylhexyl)adipate (Outside Certif	103-23-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
15A0267	8270 BNA Mix	08/12/201	Mark Cobb	08/31/201	04/27/2015 08:47 by RDW	0.01
15A0269	8270 Acid Surrogates Mix	10/11/201	Mark Cobb	10/31/202	07/21/2015 14:05 by MKM	0.002
15A0272	Base / Neutrals Surrogates	10/21/201	Mark Cobb	10/31/201	01/15/2015 09:45 by MEC	0.002

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0712

Description:	SV_BNA_LOW_ICV2	Expires:	07/27/2015
Standard Type:	Calibration Standa	Prepared:	04/28/2015
Solvent:	dichloromethane	Prepared By:	Mark Cobb
Final Volume (mls):	1	Department:	MS
Vials:	1	Last Edit:	04/28/2015 14:15 by MEC

Analyte	CAS Number	Concentration	Units
Caprolactam	105-60-2	10	ug/mL
Benzoic acid	65-85-0	10	ug/mL
Benzidine	92-87-5	10	ug/mL
Benzaldehyde	100-52-7	10	ug/mL
Atrazine	1912-24-9	10	ug/mL
Acetophenone	98-86-2	10	ug/mL
3,3'-Dichlorobenzidine	91-94-1	10	ug/mL
2,6-Dichlorophenol	87-65-0	10	ug/mL
2,3,4,6-Tetrachlorophenol	58-90-2	10	ug/mL
1,4-Dioxane	123-91-1	10	ug/mL
1,2,4,5-Tetrachlorobenzene	95-94-3	10	ug/mL
1,1-Biphenyl	92-52-4	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
14B0334	Custom Mix for LCS#2	02/18/201	Jacque Galland	05/31/201	01/27/2015 13:33 by RDW	0.002
15A0273	Benzoic Acid	11/21/201	Mark Cobb	11/30/201	01/15/2015 09:49 by MEC	0.005
15A0275	Benzidines Mix	10/08/201	Mark Cobb	10/31/201	01/15/2015 10:53 by MEC	0.005

Reviewed By Date

Analytical Standard Record

Empirical Laboratories, LLC

15D0713

Description:	SV_BNA_LOW_ICV3	Expires:	07/27/2015
Standard Type:	Calibration Standa	Prepared:	04/28/2015
Solvent:	dichloromethane	Prepared By:	Mark Cobb
Final Volume (mls):	1	Department:	MS
Vials:	1	Last Edit:	04/28/2015 14:17 by MEC

Analyte	CAS Number	Concentration	Units
Benzidine	92-87-5	10	ug/mL
3,3'-Dichlorobenzidine	91-94-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
15A0275	Benzidines Mix	10/08/201	Mark Cobb	10/31/201	01/15/2015 10:53 by MEC	0.005

Reviewed By Date

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 202 **REVISION #: 38** **EFFECTIVE DATE: 20170427**

GC/MS VOLATILES BY EPA METHOD EPA 624 and SW846 METHODS 8260B/8260C INCLUDING APPENDIX IX COMPOUNDS

APPROVALS:

Lab Director: _____ Date: **20170427**

Data Quality Manager: _____ Date: **20170427**

Group Leader: _____ Date: **20170427**
Antonio Monteiro

Changes Summary

SOP202_R38_20170427_VOA
- Soil blank and blank spike preparation updated to include use of glass beads with methanol extractions in sections 12.3, 12.4, 14.1 and 14.2.
- Updated part number for glass beads in section 9.

SOP202_R37_20161121_VOA
- Glass beads added to reagents as 9.16 while Hach test strips were added as 9.17.
- Soil blank and blank spike preparation updated to include use of glass beads in sections 12.3, 12.4, 13.5 and 14.1.
- Holding time for EPA624 samples updated in section 11.2.
- Testing for residual chlorine added as section 14.3.4.

SOP202_R36_20160812_VOA
- 8260C SPCC criteria updated in table 6 for acetone and 2-butanone.
- Criteria for spectrum to support low response factor changed in section 13.5.1.
- Table 1 updated for in-house limits and to include header definitions.
- MDL/LOD/LOQ references simplified in table 2.
- Updated checklist for low-standard read-back to include list/discuss.
- LLOQ and IDP terminology added from SW-846 update V.

SOP202_R35_20151221_VOA
- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.

SOP202_R34_20150922_VOA
- Section 10 updated to reference QS04.
- References to internal standards -50% to 200% changed to -50% to +100%
- Sections 13 updated to indicate LOQ-level standard back-calculation on linear curve fit
- Table 2 updated for MDL, LOD, LOQ, linear curve fit verification and immediate analysis of 2 passing CCVs for QSM.
- Analyst Review Checklist updated.

SOP202_R33_20150427_VOA
- Updated 8260C RF criteria for acetone in table 6 with note concerning the exception.

SOP202_R32_20150116_VOA
- Added references to Atomx system in section 9.4 and 14.3.2.
- Updated internal standard expiration date in section 10.6.2,
- Updated standard makeup information in sections 10.6.3 and 10.6.4.

SOP202_R31_20150113_VOA
- References to section supervisor revised to group leader.
- TOC updated to reflect 26 components (consistent with SOP) instead of old 23 components.
- Checklist updated to change correlation coefficient criteria to read $\geq$ instead of $\leq$, add verification of pH entry in LIMS bench sheet, and add verification of instrument ID to LIMS upload verification.

SOP202_R30_20140929_VOA

- Section 12.6 inserted and data reviewer's checklist updated to address structural isomers.
- Section 13.4 updated to address spectral selection for BFB tune.

SOP202_R29_20140827_VOA

- Added ATOMX autosampler/concentrator to equipment listing section 9.4

SOP202_R28_20140825_VOA

- Checklist updated to correct calibration criteria.

SOP202_R27_20140317_VOA

- Updated to reflect 26 components of QSM5.0.
- 8260C specifications have been added including table 6 for SPCC criteria.
- Updated Data Review Checklist to indicate linear and quadratic correlation coefficient (r2) requirement of 0.990 and remove BNA method questions.
- Update Table 2 to reflect method requirements for 8260C.
- Tables 1 and Table 5 updated to include all calibrated analytes – added Diethyl Ether, 1,3,5-trichlorobenzene, tert-Amyl Alcohol and tert-Amyl ethyl ether.
- Table 1 updated to include QSM4.2 and QSM5.0 limits.
- Table 2 updated to include QSM5.0 requirements
- Tables 3 and 4 removed as they are covered with the analyst review checklist.
- Tables 5-8 renumbered to tables 3-6 with main body references updated appropriately
- References to helium as purge and trap carrier gas updated to reflect nitrogen.
- Computer hardware/software references moved to section 23
- Updated refrigerator/freezer references in section 11.2.
- Added references to BSD in section 12, 14 and Table 2.
- Added details concerning purged BFB standard makeup and use.

Revision 26, 01/08/13

- Addition to sections 10.6.1 and 13.4: **Note: While this concentration is a recommendation for SW-846 8260B/8260C (lesser amount could be used), it is required for USEPA 624. Any deviation from this concentration must be narrated in the reported data package.**

Revision 25, 09/26/12

- All references to Target have been updated to reflect Chemstation for data processing.
- Library reference has been updated to NIST98.L.
- Data review checklist has been updated to include a place for the sample number used to recalculate the concentration from raw area counts all the way to the final LIMS concentration.
- References to LCS have been updated to reflect BS.

Revision 24, 09/13/11

- This SOP is an update from Revision 23 dated 09/09/10.
- Section 9 has been updated with column and concentrator information.
- Section 10 has been updated with current standards mixtures.
- Section 13.1 has been updated to reflect calibration curve for analytes and surrogates.

- References to QSM 4.1 have been updated to QSM 4.2.

Revision 23, 09/09/10

- This SOP is an update from Revision 22 dated 09/30/09.
- Tables 1 and 2 have been updated with appropriate reference updates.
- Tables 5-7 have been added.

Revision 22, 9/30/09

- The SOP is an update from Revision 21 dated 09/11/08
- The SOP is formatted to include all 22-elements required per the NELAC standards.
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.2, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. **Identification of the Test Method**
   This SOP is compliant with methods – EPA Method 624 and SW-846 Methods 8260B/8260C.

2. **Applicable Matrix or Matrices**
   This SOP is applicable to – The analysis of volatile organic compounds in a variety of matrices including but not limited to soils, sediments, ground and surface waters, aqueous sludge, oily wastes, etc.

3. **Limits of detection and quantitation**: See **Table 1** of this SOP.

4. **Scope of Application, Including Parameters to be Analyzed**
   This SOP is based primarily on SW-846 Methods 8260B/8260C. Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

5. **Summary of the Test Method**
   After sample preparation, the sample is introduced into the GC/MS generally using purge and trap but sometimes using direct injection (see SW-846 Methods 5030B, 5035/5035A and 3585 for preparation). In purge and trap, the analytes are stripped from the sample using nitrogen and trapped on an adsorbent tube. The tube is heated while being backflushed with nitrogen to carry the analytes to the GC/MS system. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra of the sample. Analytes are quantitated relative to known standards using the internal standard method.

6. **Definitions**
   Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7. **Interferences**
   Section 3.0 of SW-846 Method 8260B details interferences and potential problems which may be encountered when dealing with volatile analyses.

8. **Safety**
   Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab wide.

9. **Equipment & Supplies**

   9.1 GC: HP 5890 or 6890, temperature programmable, suitable for split or splitless injection.

   9.2 Column: HP-VOC, 30 meter x 0.2 mm I.D. 1.12 µm film thickness silicon coated fused silica capillary column or equivalent.

9.3 M.S.: HP 5971, 5972 or 5973 capable of scanning 35 to 500 amu every one second or less, using 70 volts electron energy in electron impact ionization mode. The MS is capable of producing a mass spectrum for p-Bromofluorobenzene, BFB, which meets all tuning criteria for EPA methods [when 1 µL (50 ng) of the GC/MS tuning standard is introduced to the GC.]

9.4 Purge and Trap Unit

9.4.1 Concentrators: Tekmar/Dohrmann 3000/3100 Sample Concentrator equipped with Supelco trap number 2-4920-U VOCARB 3000, or equivalent, providing good delivery for all target compounds. Atomx Trap #14-5864-403.

9.4.2 Autosamplers: Varian Archon 51 position programmable autosampler and Tekmar Atomx 80-position autosampler with 5ml to 25ml water and heated soil capability.

9.7 Microsyringes – 1.0, 5.0,10, 25, 50, 100, 250, 500 and 1000 µL.

9.8 Syringes – 5, 25 and 50 mL, gas-tight with Luer end.

9.9 Balance - analytical, 0.0001 g; top-loading, 0.01 g.

9.10 Disposable Pasteur pipets.

9.11 Volumetric flasks, Class A - 2 mL, 5 mL, 10 mL, 50 mL, 100 mL and 250 mL with ground-glass stoppers.

9.12 Wooden tongue depressors

9.13 Glass scintillation vials - 20mL with screw caps.

9.14 Latex or Nitrile Gloves

9.15 pH paper (measures pH from 0-14).

9.16 Glass Beads (Fisher Cat#11-312A, or equivalent)

9.17 AquaCheck Free Chlorine Test Strips (Hach 27450-50, or equivalent)

10. Reagents and Standards

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST - RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

10.2 Organic-free reagent water - obtained from the charcoal filter system in the VOA laboratory.

10.3 Methanol - Purge and trap grade (EM-Omnisolv EM-0482-6 or equivalent)

10.4 Methanol - suitable for use in gas chromatography (B&J Omnisolv MX0484-1, or equivalent)

10.5 Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).

10.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the LIMS system along with their lot number and vendor and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date**. All stocks and standards are stored in the freezer at a temperature of -15°C $\pm$ 5°C or less from the date they are received/prepared. The freezer temperature is monitored daily with a calibrated thermometer (annual calibration for liquid in glass and quarterly calibration

for digital) and recorded with calibration correction in the VOA refrigerator/freezer logbook. Minimum and maximum temperatures are recorded after weekends/holidays. Makeup of common standards is detailed below. See standard information in LIMS system for makeup of other standards.

10.6.1 The Bromofluorobenzene (BFB) tuning standard is prepared as follows: Using a 50µL syringe, 25µL of standard (BFB @ 10000ng/µL) is injected into a 5mL volumetric flask containing approximately 4.0mL P&T methanol (Vendor, Lot) and diluted to volume with same making a 50ng/µL standard. After capping and inverting 3 times, the solution is transferred to a labeled Teflon-lined, screw-capped vial and stored in the freezer at -15°C ± 5°C or less for up to a year **(1 week for South Carolina samples)**. A direct injection of 1µL (or equivalent purge delivering 50ng) is used to tune the instrument. **Note: While this concentration is a recommendation for SW-846 8260B/8260C (a lesser amount could be used), it is required for USEPA 624. Any deviation from this concentration must be narrated in the reported data package.**

Note: For purging, 1 µL of this standard is added to 5mL lab water in a 40mL VOA vial to deliver 50ng BFB for soils or 10uL of this standard is added to 50mL lab water in a volumetric flask, inverted 3 times to mix then transferred to a 40mL VOA vial for a 5ml purge to deliver 50ng BFB for waters.

10.6.2 The internal and surrogate standards are prepared as follows: Using the indicated syringe, the indicated amount of standard is injected into a 50 mL volumetric flask containing P&T methanol (Vendor, Lot) and diluted to volume with same making a 150ng/µL standard. After capping and inverting 3 times, the solution is transferred to 40mL VOA vial and assigned a 1 year expiration. As needed, the solution is transferred to the autosampler where it is maintained under nitrogen. Each 8260/624 sample is automatically injected with 1µL of this standard. (The internal standard/surrogate solution may be replaced if the –50% to +100% criteria exceeds in the CCV when calculated against the midpoint of the ICAL or previous CCV.)

Standard	Conc. (ng/µL)	Syringe (µL)	Amount (µL)
8260 ISTD Mix	2500	1000	3000
Surr. Mix	2500	1000	3000

NOTE: for purposes of LIMS documentation, virtual standards are created for the internal standards and surrogates for use in the sequence and the bench sheet. These virtual standards are traceable to the actual standard being used.

10.6.3 Calibration standards are prepared from the vendor stock standards at appropriate concentrations as follows. Occasionally unusual compounds are added to the mix so it is best to check the LIMS for exact standard makeup. Note: for laboratory blank spikes (BS), alternate sources or lot numbers from the main calibration standard are used to make the BS standard.

10.6.3.1 Primary Standard: Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same to make a 200-4000ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ±

5°C for 1 week. A 100-2000µg/L (5mL purge) standard is made using 25µL of this standard to 50mLof reagent water.

Stock Standard(CCV)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
Custom 2 Mix	2000-40000	250	200	200-4000
Ketones+Misc Mix	2000-10000	250	200	200-1000
Liquid mix	2000	250	200	200
Custom mix	2000-10000	250	200	200-1000
Gases **(cat#30042)**	2000	250	200	200
Oxygenates **(CC2098.10)**	2000-10000	250	200	200-1000

Additional compounds may be added such as Appendix IX. Refer to standard ID in LIMS system.

10.6.4 ICV/BS/Matrix Spike Mix: A second source standard is used to check the validity of the gas and primary calibration standards used in analyzing the calibration curve. Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same to make a 100-2000ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50µg/L ICV/BS/Matrix Spike is made using 25µL of this standard to 50mL of reagent water/Sample Matrix.

Stock Standard(ICV/BS)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
Custom 2 mix	2000-40000	100	100	100-2000
Oxygenates	2000-10000	100	100	100-500
Ketones+Misc Mix	2000-10000	100	100	100-500
Liquid mix	2000	100	100	100
Custom Mix	2000-10000	100	100	100-500
Gases	2000	100	100	100

11. Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 All water samples are stored in the VOA water refrigerator in the VOA lab at a temperature of 0°C-6°C. All unpreserved soil samples in Terracore or encores are stored in the VOA soil freezer at ≤10°C. All soil samples in bulk jars or chemically preserved Terracore are stored in the VOA soil refrigerator at a temperature of 0°C-6°C. Non-preserved water volatile samples (including EPA 624 samples treated with sodium thiosulfate) have a holding time of 7 days from date of sampling. Preserved water samples and soil volatile samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). The temperatures are monitored daily with calibrated thermometers (annual calibration for liquid in glass and quarterly calibration for digital) and recorded with calibration correction in the appropriate logbooks. The weekend temperature is monitored with Min/Max thermometers and recorded upon arrival next business day.

12. Quality Control

12.1 Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.1 Internal Standards - All samples and QC are spiked with internals. See **Table 2** for acceptance criteria and corrective action.

12.2 Surrogates - All samples and QC are spiked with surrogates. See **Table 2** of this SOP for acceptance criteria and corrective action.

12.3 BS (BSD) Sample - A BS (BSD if no MS/MSD) is analyzed every 12 hour tune. To prepare the BS (BSD), a blank (5ml water, 5g glass beads with 5mL water or 100ul of methanol extract prepared with 5g glass beads added to 5mL water) is spiked with standards prepared from an alternate vendor or lot number from the calibration standards. Note: the concentration of the BS will be 20 µg/L when analyzing 624 samples (QC Check Sample). See **Table 2** of this SOP for acceptance criteria and corrective action. **When analyzing samples for South Carolina the limits are 70-130% except for poor purgers which are 60-140%.**

12.4 Method Blanks - A method blank (5ml water, 5g glass beads with 5mL water or 100ul of methanol extract prepared with 5g glass beads added to 5mL water) is analyzed every 12 hour tune. See **Table 2** of this SOP for acceptance criteria and corrective action.

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for an MS/MSD with the BS standard. See **Table 2** of this SOP for acceptance criteria and corrective action. MS data evaluation must include the consideration of the following factors.

12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. A water sample which was taken from the same VOA vial for the original sample and the MS/MSD should have very good RPDs unless there has been a method problem. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

12.5.3 MS vs. MSD - If a spiked compound has a problem in both the MS and MSD, review the BS and if acceptable no further action may be necessary since it is attributable to matrix effect.

12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your group leader who should discuss the problem with the client/project manager to determine the action to be taken.

12.6 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Resolution between the peaks should be ≤50%. To evaluate the resolution, the standard (CCV) must be processed with the correct method in Chemstation and be evaluated in QEdit. The Chroeval function in QEdit allows for resolution to be evaluated under the Evaluate Resolution function. Once the peaks in question are integrated, a % resolution can be generated and printed.

13. Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Chromatographic conditions – Refer to corresponding instrument maintenance log for current gas chromatograph, mass spectrometer, and concentrator conditions.

13.3 System Bakeout - Prior to analysis an instrument blank is analyzed.
NOTE: Further cleaning may be accomplished by backflushing the lines with methanol and then analyzing blanks overnight.

13.4 Tuning - Prior to any calibration or analysis, BFB tuning criteria must be met for a 1.0µL injection (or equivalent purging to deliver 50ng BFB) of the tuning standard. See **Table 3** of this SOP for acceptance criteria. Tune must be met every 12 hours sample analysis is to be performed **(every 24 hours for *Federal Register* Method 624 except for South Carolina which only allows 12 hours).** The mass spectrum of BFB is acquired as follows: by using the BFB method in Target (which uses three scans with background subtraction) to process the BFB data file. If the BFB tune does not pass criteria corrective action should be taken. **Note: While this concentration is a recommendation for SW-846 8260B/8260C (lesser amount could be used), it is required for USEPA 624. Any deviation from this concentration must be narrated in the reported data package.**

13.4 The mass spectrum of BFB is acquired by using the autofind BFB function in Chemstation (which uses three scans [one of the apex and the scan prior and after the apex] with background subtraction [instrument selected peak within 20 scans prior to the apex scan]) to evaluate the BFB.

13.5 **Calibration:** Calibration standards are made up in water (or water and glass beads) using the appropriate amount of the methanol standard. See the LIMS for preparation of standards. **Calibration for soils for South Carolina requires that 5mL of sodium bisulfate solution is added to each calibration standard made if the samples will be preserved with sodium bisulfate.** All manual calibration integrations must be approved by the section manager or designated peer reviewer.

13.5.1 Initial Calibration - An initial calibration curve at no less than five (six if using a quadratic curve fit) concentration levels for analytes and surrogates must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. The LOQ-level standard should be back-calculated to the calibration curve for linear calibration curve fits and a recovery calculated to verify linearity. See Table 2 for criteria. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual calibration integrations must be approved by the section manager or designated peer reviewer. Any response factors less than 0.010 must be supported by the mass spectrum of the lowest standard. **No quadratic curves for South Carolina.**

8260B CCCs:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene

1,2-Dichloropropane	Vinyl chloride

8260B SPCCs:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

8260C SPCCs – see **table 6**.

13.5.2 Initial Calibration Verification (ICV) - A second source standard is prepared at or near the CCV concentration and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual ICV integrations must be approved by the section manager or designated peer reviewer.

13.5.3 Continuing Calibration Verification (CCV) - A CCV is analyzed every 12 hour tune and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual CCV integrations must be approved by the section manager or designated peer reviewer.

NOTE: Acceptance criteria for method 624 consists of meeting recovery limits found in table 5 of the method for a QC check sample. This QC check sample is made from a separate source or lot number than the calibration standard at a concentration of 20 µg/L.

QSM5.0: For QSM 5.0, an ending CCV must be analyzed within the 12 hour tune. See **Table 2** of this SOP for acceptance criteria and corrective action.

14. Procedure

14.1 BS (BSD) - A BS (BSD) is analyzed every 12 hour tune. Using standards prepared from an alternate vendor or lot number, blank water (5ml water, 5g glass beads with 5mL water or 100ul of methanol extract prepared with 5g glass beads added to 5mL water) is spiked at the 50 µg/L (5mL/soil) or 10 µg/L (25mL) level. See **Table 2** of this SOP for acceptance criteria and corrective action. **Note: the concentration of the BS will be 20 µg/L when analyzing 624 samples (QC Check Sample).**

14.2 Method Blank - Prior to sample analysis, the system must be shown to be free of contamination through analysis of a method blank. 5ml water, 5g glass beads with 5mL water or 100ul of methanol extract prepared with 5g glass beads added to 5mL water is used for the method blank. See **Table 2** of this SOP for acceptance criteria and corrective action.

14.3 Sample Analysis - Prior to analysis, the samples are prepared for chromatography using the appropriate sample preparation method (5mL water, 25mL water, low soil,

high soil, etc.) See SOP 225 for preparation of a 5035 soil sample. For a 5mL/25mL water sample, use the following procedure:

14.3.1 Load the vial into the Archon/Atomx autosampler in the expected position.

14.3.2 Program the Archon/Atomx for the loaded vial range and necessary dilutions, making sure the programmed method is set for the same volume as the purge vessel on the front of the LSC/Atomx and that the Chemstation sequence matches the Archon/Atomx sequence. Note: TCLP samples are analyzed at a 10x dilution. One TCLP sample is spiked per batch at receipt of leachates.

14.3.3 After analysis of the sample has been completed, check the pH of the sample using pH paper and verify it to be less than a pH of 2 (recorded on the sequence log). If it is not, record the pH on the sequence log and generate a non-conformance report. The sample report will have to be qualified for preservation if the analysis is being performed more than 7 days after sampling. [Note: TCLP samples do not require a pH check.]

14.3.4 Samples being analyzed for EPA 624 must be checked for residual chlorine using the AquaCheck test strips. If the strip turns purple, record the concentration of free chlorine on the sequence log and generate a non-conformance report. The sample report will have to be narrated to indicate the presence of free chlorine.

14.4 **Instrument sequence**

An example of a typical instrument sequence log follows:

1-BFB Tune (12:00 am)
2-CCV
3-BS
4-RL standard
5-Method Blank
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample
15-Sample
16-Sample
17-Sample
18-Sample MS
19-Sample MSD
Ending CCV required within 12 hour window for QSM5.0 (HCV)
20-BFB (12:00pm - 12 hours since last BFB/CCV)
21-CCV
22-BS
23-Method Blank
24-Sample
25-Sample

14.5 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the ChemStation. This run log is signed, dated and

paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through the Chemstation data system. Quantitative measurements are performed using the calculations found in section 15.2 of this SOP. The following must be checked to determine if the sample will need any reanalysis or dilution. See **Table 2** of this SOP for acceptance criteria and corrective action. Formal data evaluation is detailed in SOP QS05. **See SOP QS07 for guidance on manual integrations.**

14.5.1 Internal Standards - Areas counts and retention times.

14.5.2 Surrogates – Recoveries and retention times – Note: ***Federal Register* Method 624 contains no criteria for surrogate recovery.**

14.5.3 Analyte concentration.

14.5.4 Qualitative identification based on spectrum and retention time.

15. Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Calculations:

15.2.1 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

15.2.2 Calibration verification involves the calculation of the percent drift (linear) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\%\ \text{Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\%\ \text{Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±20% for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

15.2.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.]

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

Where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume purged in ug/L.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = For purge-and-trap analysis, V_i is not applicable and is set at 1.

$\overline{RF}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample purged (mL). If units of liters are used for this term, multiply the results by 1000.

15.2.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.]

$$\text{Concentration } (\mu g/kg) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s, A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples.

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

16. Method Performance

Initial Demonstration of Capability (DOC)/Performance (IDP): Each analyst must perform an IDOC/IDP prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples (may be prepared from same source as calibration). The data is calculated for accuracy and precision requirements. The IDOC form is completed by each analyst and then provided to the group leader for further processing and approval. See **Table 2** for acceptance criteria.

17. Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20. Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data or unacceptable data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

21. Waste Management.

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

22. Equipment/Instrument Maintenance

22.1 Purge and Trap

a. Flush out the sample and/or transfer lines with Methanol or replace silcosteel transfer lines.
b. Change the trap or rejuvenate the trap by adding about 250ul of methanol to the bottom side of the trap, letting sit for about 3 hours then placing in GC oven at 150°C for 3-4 hours minimum (overnight preferred). After cooling and capping both ends, trap is ready to be used again.
c. The purge vessel, top of trap fitting or sample pathway may be flushed with methanol and/or sonicated in methanol. Blow-dry with helium prior to reassembly for analysis.

23. Computer hardware and software

23.1 Acquisition Software: HP Chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

23.2 Data Processing Software: HP Chemstation data system. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances of any EICP between specified times or scan-number limits. NIST98.L mass spectral library is installed.

24. Troubleshooting – If routine maintenance in section 22.0 did not bring the instrument back into control, more involved maintenance may be required (i.e. detector maintenance, analytical column maintenance, etc.). Bring to the attention of the Group Leader.

25. References

25.1 40 CFR, Part 136; Appendix A
25.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition and updates
25.3 National Environmental Laboratory Accreditation Conference; CH. 5, 2001
25.4 USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
25.5 The NELAC Institute (TNI) Standards, Volum3 2, 09/2009

25.6 DOD Quality Systems Manual for Environmental Laboratories version 4.2, 10/2010
25.7 DOD Quality Systems Manual for Environmental Laboratories version 5.0, 07/2013

26. Tables, Diagrams, Flowcharts and Validation Data

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Table 1 – LOQ/LOD/DL

S/W	Analyte	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Solid	1,1,1,2-Tetrachloroethane	630-20-6	5	2.5	1.25	ug/Kg	75	125	20	78	125	20
Solid	1,1,1-Trichloroethane (1,1,1-TCA)	71-55-6	5	2.5	1.25	ug/Kg	70	135	30	73	130	20
Solid	1,1,2,2-Tetrachloroethane	79-34-5	5	2.5	1.25	ug/Kg	55	130	30	70	124	20
Solid	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	76-13-1	10	5	2.5	ug/Kg	20	185	30	66	136	20
Solid	1,1,2-Trichloroethane	79-00-5	5	2.5	1.25	ug/Kg	60	125	30	78	121	20
Solid	1,1-Dichloroethane (1,1-DCA)	75-34-3	5	2.5	1.25	ug/Kg	75	125	30	76	125	20
Solid	1,1-Dichloroethene (1,1-DCE)	75-35-4	5	2.5	1.25	ug/Kg	65	135	30	70	131	20
Solid	1,1-Dichloropropene	563-58-6	5	2.5	1.25	ug/Kg	70	135	30	76	125	20
Solid	1,2,3-Trichlorobenzene	87-61-6	5	2.5	1.25	ug/Kg	60	130	30	66	130	20
Solid	1,2,3-Trichloropropane	96-18-4	5	2.5	1.25	ug/Kg	65	130	30	73	125	20
Solid	1,2,4-Trichlorobenzene	120-82-1	5	2.5	1.25	ug/Kg	65	130	30	67	129	20
Solid	1,2,4-Trimethylbenzene	95-63-6	5	2.5	1.25	ug/Kg	65	135	30	75	123	20
Solid	1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	10	5	2.5	ug/Kg	40	135	30	61	132	20
Solid	1,2-Dibromoethane (EDB)	106-93-4	5	2.5	1.25	ug/Kg	70	125	30	78	122	20
Solid	1,2-Dichlorobenzene (12DCB)	95-50-1	5	2.5	1.25	ug/Kg	75	120	30	78	121	20
Solid	1,2-Dichloroethane (EDC)	107-06-2	5	2.5	1.25	ug/Kg	70	135	30	73	128	20
Solid	1,2-Dichloroethane-d4 (Surrogate)	17060-07-0	30	2.5	1.25	ug/Kg	75	140		71	136	
Solid	1,2-Dichloroethene (Total)	540-59-0	10	5	2.5	ug/Kg	65	135	30	78	122	20
Solid	1,2-Dichloropropane	78-87-5	5	2.5	1.25	ug/Kg	70	120	30	76	123	20
Solid	1,3,5-Trichlorobenzene	108-70-3	5	2.5	1.25	ug/Kg	70	130	30	71	128	20
Solid	1,3,5-Trimethylbenzene	108-67-8	5	2.5	1.25	ug/Kg	65	135	30	73	124	20
Solid	1,3-Dichlorobenzene (13DCB)	541-73-1	5	2.5	1.25	ug/Kg	70	125	30	77	121	20
Solid	1,3-Dichloropropane	142-28-9	5	2.5	1.25	ug/Kg	75	125	30	77	121	20
Solid	1,4-Dichlorobenzene (14DCB)	106-46-7	5	2.5	1.25	ug/Kg	70	125	30	75	120	20
Solid	1,4-Dioxane	123-91-1	200	100	40	ug/Kg	50	150	30	55	138	20
Solid	1-Chlorohexane	544-10-5	5	2.5	1.25	ug/Kg	70	130	30	71	130	20
Solid	2,2-Dichloropropane	594-20-7	5	2.5	1.25	ug/Kg	65	135	30	67	133	20
Solid	2-Butanone (Methyl ethyl ketone; MEK)	78-93-3	10	5	2.5	ug/Kg	30	160	30	51	148	20
Solid	2-Chloroethyl vinyl ether	110-75-8	100	50	25	ug/Kg	10	200	30	43	149	20
Solid	2-Chlorotoluene	95-49-8	5	2.5	1.25	ug/Kg	70	130	30	75	122	20
Solid	2-Hexanone (Methyl butyl ketone; MBK)	591-78-6	10	5	2.5	ug/Kg	45	145	30	53	145	20
Solid	4-Chlorotoluene	106-34-4	5	2.5	1.25	ug/Kg	75	125	30	72	124	20
Solid	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	108-10-1	10	5	2.5	ug/Kg	45	145	30	65	135	20
Solid	Acetone	67-64-1	20	10	5	ug/Kg	20	160	30	36	164	20
Solid	Acetonitrile	75-07-0	50	25	10	ug/Kg	50	150	30	54	143	20
Solid	Acrolein	107-02-8	20	10	5	ug/Kg	10	150	30	47	155	20
Solid	Acrylonitrile	107-13-1	20	10	5	ug/Kg	35	180	30	65	134	20

S/W	Analyte	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Solid	Allyl chloride	107-05-1	5	2.5	1.25	ug/Kg	50	150	30	68	135	20
Solid	Benzene	71-43-2	5	2.5	1.25	ug/Kg	75	125	30	77	121	20
Solid	Bromobenzene	108-86-1	5	2.5	1.25	ug/Kg	65	120	30	78	121	20
Solid	Bromochloromethane	74-97-5	5	2.5	1.25	ug/Kg	70	125	30	78	125	20
Solid	Bromodichloromethane (Dichlorobromomethane)	75-27-4	5	2.5	1.25	ug/Kg	70	130	30	75	127	20
Solid	Bromofluorobenzene (Surrogate)	460-00-4	30	2.5	1.25	ug/Kg	85	120		79	119	
Solid	Bromoform	75-25-2	5	2.5	1.25	ug/Kg	55	135	30	67	132	20
Solid	Bromomethane (methyl bromide)	74-83-9	10	5	2.5	ug/Kg	30	160	30	53	143	20
Solid	Carbon Disulfide	75-15-0	5	2.5	1.25	ug/Kg	45	160	30	63	132	20
Solid	Carbon Tetrachloride	56-23-5	5	2.5	1.25	ug/Kg	65	135	30	70	135	20
Solid	Chlorobenzene	108-90-7	5	2.5	1.25	ug/Kg	75	125	30	79	120	20
Solid	Chloroethane	75-00-3	10	5	2.5	ug/Kg	40	155	30	59	139	20
Solid	Chloroform	67-66-3	5	2.5	1.25	ug/Kg	70	125	30	78	123	20
Solid	Chloromethane (methyl chloride)	74-87-3	10	5	2.5	ug/Kg	50	130	30	50	136	20
Solid	Chloroprene (2-chloro-1,3-butadiene)	126-99-8	5	2.5	1.25	ug/Kg	50	150	30	65	133	20
Solid	cis-1,2-Dichloroethene (cis-1,2-DCE)	156-59-2	5	2.5	1.25	ug/Kg	65	125	30	77	123	20
Solid	cis-1,3-Dichloropropene	10061-01-5	5	2.5	1.25	ug/Kg	70	125	30	74	126	20
Solid	cis-1,4-Dichloro-2-butene	1476-11-5	5	2.5	1.25	ug/Kg	50	150	30	69	143	20
Solid	Cyclohexane	110-82-7	5	2.5	1.25	ug/Kg	65	140	30	67	131	20
Solid	Dibromochloromethane (Chlorodibromomethane)	124-48-1	5	2.5	1.25	ug/Kg	65	130	30	74	126	20
Solid	Dibromofluoromethane (Surrogate)	1868-53-7	30	2.5	1.25	ug/Kg	80	125		78	119	
Solid	Dibromomethane	74-95-3	5	2.5	1.25	ug/Kg	75	130	30	78	125	20
Solid	Dichlorodifluoromethane (CFC-12)	75-71-8	10	5	2.5	ug/Kg	35	135	30	29	149	20
Solid	Diethyl ether	60-29-7	5	2.5	1.25	ug/Kg	35	135	30	71	129	20
Solid	Di-isopropyl ether	108-20-3	5	2.5	1.25	ug/Kg	70	130	30	69	127	20
Solid	Ethyl methacrylate	97-63-2	5	2.5	1.25	ug/Kg	45	145	30	69	129	20
Solid	Ethyl tert-Butyl Ether (ETBE)	637-92-3	5	2.5	1.25	ug/Kg	70	130	30	72	126	20
Solid	Ethylbenzene	100-41-4	5	2.5	1.25	ug/Kg	75	125	30	76	122	20
Solid	Hexachlorobutadiene (HCBD)	87-68-3	5	2.5	1.25	ug/Kg	55	140	30	61	135	20
Solid	Hexane	110-54-3	5	2.5	1.25	ug/Kg	60	130	30	45	142	20
Solid	Iodomethane	74-88-4	20	10	5	ug/Kg	55	165	30	71	131	20
Solid	Isobutyl alcohol	78-83-1	100	40	20	ug/Kg	50	150	30	60	135	20
Solid	Isopropylbenzene (Cumene)	98-82-8	5	2.5	1.25	ug/Kg	75	130	30	68	134	20
Solid	m,p-Xylene	108-38-3/106-42-3	10	5	2.5	ug/Kg	80	125	30	77	124	20
Solid	Methacrylonitrile	126-98-7	50	25	10	ug/Kg	50	150	30	66	132	20
Solid	Methyl Acetate	79-20-9	10	5	2.5	ug/Kg	45	165	30	53	144	20
Solid	Methyl methacrylate	80-62-6	5	2.5	1.25	ug/Kg	65	125	30	63	134	20
Solid	Methyl Tertiary Butyl Ether (MTBE)	1634-04-4	5	2.5	1.25	ug/Kg	55	150	30	73	125	20
Solid	Methylcyclohexane	108-87-2	5	2.5	1.25	ug/Kg	65	135	30	66	133	20

S/W	Analyte	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Solid	Methylene Chloride, or Dichloromethane	75-09-2	10	5	2.5	ug/Kg	55	140	30	70	128	20
Solid	Naphthalene	91-20-3	5	2.5	1.25	ug/Kg	40	125	30	62	129	20
Solid	n-Butylbenzene	104-51-8	5	2.5	1.25	ug/Kg	65	140	30	70	128	20
Solid	n-Propylbenzene	103-65-1	5	2.5	1.25	ug/Kg	65	135	30	73	125	20
Solid	o-Xylene	95-47-6	5	2.5	1.25	ug/Kg	75	125	30	77	123	20
Solid	p-Isopropyltoluene	99-87-6	5	2.5	1.25	ug/Kg	75	130	30	73	127	20
Solid	Propionitrile	107-12-0	50	25	10	ug/Kg	50	150	30	68	134	20
Solid	sec-Butylbenzene	135-98-8	5	2.5	1.25	ug/Kg	65	130	30	73	126	20
Solid	Styrene	100-42-5	5	2.5	1.25	ug/Kg	75	125	30	76	124	20
Solid	tert-Amyl Alcohol (2-butanol)	75-85-4	25	12.5	6.25	ug/Kg	65	130	30	52	142	20
Solid	tert-Amyl ethyl ether	919-94-8	50	25	10	ug/Kg	50	150	30	68	113	20
Solid	tert-Amyl methyl ether	994-05-8	10	5	2.5	ug/Kg	50	150	30	73	126	20
Solid	tert-Butyl alcohol	75-65-0	20	10	5	ug/Kg	50	150	30	68	133	20
Solid	tert-Butylbenzene	98-06-6	5	2.5	1.25	ug/Kg	65	130	30	73	125	20
Solid	Tetrachloroethene (PCE; PERC)	127-18-4	5	2.5	1.25	ug/Kg	65	140	30	73	128	20
Solid	Tetrahydrofuran	109-99-9	10	5	2.5	ug/Kg	70	130	30	61	135	20
Solid	Toluene	108-88-3	5	2.5	1.25	ug/Kg	70	125	30	77	121	20
Solid	Toluene-d8 (Surrogate)	2037-26-5	30	2.5	1.25	ug/Kg	85	115		85	116	
Solid	trans-1,2-Dichloroethene (trans-1,2-DCE)	156-60-5	5	2.5	1.25	ug/Kg	65	135	30	74	125	20
Solid	trans-1,3-Dichloropropene	10061-02-6	5	2.5	1.25	ug/Kg	65	125	30	71	130	20
Solid	trans-1,4-Dichloro-2-butene	110-57-6	5	2.5	1.25	ug/Kg	50	150	30	62	136	20
Solid	Trichloroethene (TCE)	79-01-6	5	2.5	1.25	ug/Kg	75	125	30	77	123	20
Solid	Trichlorofluoromethane (CFC-11)	75-69-4	10	5	2.5	ug/Kg	25	185	30	62	140	20
Solid	Vinyl acetate	108-05-4	10	5	2.5	ug/Kg	50	135	30	50	151	20
Solid	Vinyl Chloride (VC)	75-01-4	5	2.5	1.25	ug/Kg	60	125	30	56	135	20
Solid	Xylenes (total)	1330-20-7	15	7.5	3.75	ug/Kg	75	125	30	78	124	20
Water	1,1,1,2-Tetrachloroethane	630-20-6	1.00	0.50	0.25	ug/L	80	130	30	78	124	20
Water	1,1,1-Trichloroethane (1,1,1-TCA)	71-55-6	1.00	0.50	0.25	ug/L	65	130	30	74	131	20
Water	1,1,2,2-Tetrachloroethane	79-34-5	1.00	0.50	0.25	ug/L	65	130	30	71	121	20
Water	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	76-13-1	2.00	1.00	0.50	ug/L	60	130	30	70	136	20
Water	1,1,2-Trichloroethane	79-00-5	1.00	0.50	0.25	ug/L	75	125	30	80	119	20
Water	1,1-Dichloroethane (1,1-DCA)	75-34-3	1.00	0.50	0.25	ug/L	70	135	30	77	125	20
Water	1,1-Dichloroethene (1,1-DCE)	75-35-4	1.00	0.50	0.25	ug/L	70	130	30	71	131	20
Water	1,1-Dichloropropene	563-58-6	1.00	0.50	0.25	ug/L	75	130	30	79	125	20
Water	1,2,3-Trichlorobenzene	87-61-6	2.00	1.00	0.50	ug/L	55	140	30	69	129	20
Water	1,2,3-Trichloropropane	96-18-4	2.00	1.00	0.50	ug/L	75	125	30	73	122	20
Water	1,2,4-Trichlorobenzene	120-82-1	2.00	1.00	0.50	ug/L	65	135	30	69	130	20
Water	1,2,4-Trimethylbenzene	95-63-6	1.00	0.50	0.25	ug/L	75	130	30	76	124	20
Water	1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	2.00	1.00	0.50	ug/L	50	130	30	62	128	20

S/W	Analyte	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Water	1,2-Dibromoethane (EDB)	106-93-4	1.00	0.50	0.25	ug/L	80	120	30	77	121	20
Water	1,2-Dichlorobenzene (12DCB)	95-50-1	1.00	0.50	0.25	ug/L	70	120	30	80	119	20
Water	1,2-Dichloroethane (EDC)	107-06-2	1.00	0.50	0.25	ug/L	70	130	30	73	128	20
Water	1,2-Dichloroethane-d4 (Surrogate)	17060-07-0	30	0.50	0.25	ug/L	70	120		81	118	
Water	1,2-Dichloroethene (Total)	540-59-0	2.00	1.00	0.50	ug/L	60	140	30	79	121	20
Water	1,2-Dichloropropane	78-87-5	1.00	0.50	0.25	ug/L	75	125	30	78	122	20
Water	1,3,5-Trichlorobenzene	108-70-3	2.00	1.00	0.50	ug/L	75	130	30	75	130	20
Water	1,3,5-Trimethylbenzene	108-67-8	1.00	0.50	0.25	ug/L	75	130	30	75	124	20
Water	1,3-Dichlorobenzene (13DCB)	541-73-1	1.00	0.50	0.25	ug/L	75	125	30	80	119	20
Water	1,3-Dichloropropane	142-28-9	1.00	0.50	0.25	ug/L	75	125	30	80	119	20
Water	1,4-Dichlorobenzene (14DCB)	106-46-7	1.00	0.50	0.25	ug/L	75	125	30	79	118	20
Water	1,4-Dioxane	123-91-1	40.0	20.0	10.0	ug/L	50	150	30	59	139	20
Water	1-Chlorohexane	544-10-5	2.00	1.00	0.50	ug/L	75	125	30	76	124	20
Water	2,2-Dichloropropane	594-20-7	1.00	0.50	0.25	ug/L	70	135	30	60	139	20
Water	2-Butanone (Methyl ethyl ketone; MEK)	78-93-3	10.0	5.00	2.50	ug/L	30	150	30	56	143	20
Water	2-Chloroethyl vinyl ether	110-75-8	5.00	2.50	1.25	ug/L	10	165	30	51	139	20
Water	2-Chlorotoluene	95-49-8	1.00	0.50	0.25	ug/L	75	125	30	79	122	20
Water	2-Hexanone (Methyl butyl ketone; MBK)	591-78-6	5.00	2.50	1.25	ug/L	55	130	30	57	139	20
Water	4-Chlorotoluene	106-34-4	1.00	0.50	0.25	ug/L	75	130	30	78	122	20
Water	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	108-10-1	5.00	2.50	1.25	ug/L	60	135	30	67	130	20
Water	Acetone	67-64-1	10.0	5.00	2.50	ug/L	40	140	30	39	160	20
Water	Acetonitrile	75-07-0	10.0	5.00	2.50	ug/L	50	150	30	50	142	20
Water	Acrolein	107-02-8	5.00	2.50	1.25	ug/L	10	200	30	39	155	20
Water	Acrylonitrile	107-13-1	10.0	5.00	2.50	ug/L	35	180	30	63	135	20
Water	Allyl chloride	107-05-1	1.00	0.50	0.25	ug/L	50	150	30	68	130	20
Water	Benzene	71-43-2	1.00	0.50	0.25	ug/L	80	120	30	79	120	20
Water	Bromobenzene	108-86-1	1.00	0.50	0.25	ug/L	75	125	30	80	120	20
Water	Bromochloromethane	74-97-5	1.00	0.50	0.25	ug/L	65	130	30	78	123	20
Water	Bromodichloromethane (Dichlorobromomethane)	75-27-4	1.00	0.50	0.25	ug/L	75	120	30	79	125	20
Water	Bromofluorobenzene (Surrogate)	460-00-4	30	0.50	0.25	ug/L	75	120		85	114	
Water	Bromoform	75-25-2	1.00	0.50	0.25	ug/L	70	130	30	66	130	20
Water	Bromomethane (methyl bromide)	74-83-9	2.00	1.00	0.50	ug/L	30	145	30	53	141	20
Water	Carbon Disulfide	75-15-0	1.00	0.50	0.25	ug/L	35	160	30	64	133	20
Water	Carbon Tetrachloride	56-23-5	1.00	0.50	0.25	ug/L	65	140	30	72	136	20
Water	Chlorobenzene	108-90-7	1.00	0.50	0.25	ug/L	80	120	30	82	118	20
Water	Chloroethane	75-00-3	2.00	1.00	0.50	ug/L	60	135	30	60	138	20
Water	Chloroform	67-66-3	1.00	0.50	0.25	ug/L	65	135	30	79	124	20
Water	Chloromethane (methyl chloride)	74-87-3	1.00	0.50	0.25	ug/L	40	125	30	50	139	20
Water	Chloroprene (2-chloro-1,3-butadiene)	126-99-8	1.00	0.50	0.25	ug/L	50	150	30	65	135	20

S/W	Analyte	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Water	cis-1,2-Dichloroethene (cis-1,2-DCE)	156-59-2	1.00	0.50	0.25	ug/L	70	125	30	78	123	20
Water	cis-1,3-Dichloropropene	10061-01-5	1.00	0.50	0.25	ug/L	70	130	30	75	124	20
Water	cis-1,4-Dichloro-2-butene	1476-11-5	2.00	1.00	0.50	ug/L	50	150	30	57	146	20
Water	Cyclohexane	110-82-7	1.00	0.50	0.25	ug/L	60	130	30	71	130	20
Water	Dibromochloromethane (Chlorodibromomethane)	124-48-1	1.00	0.50	0.25	ug/L	60	135	30	74	126	20
Water	Dibromofluoromethane (Surrogate)	1868-53-7	30	0.50	0.25	ug/L	85	115		80	119	
Water	Dibromomethane	74-95-3	1.00	0.50	0.25	ug/L	75	125	30	79	123	20
Water	Dichlorodifluoromethane (CFC-12)	75-71-8	2.00	1.00	0.50	ug/L	30	155	30	32	152	20
Water	Diethyl ether	60-29-7	2.00	1.00	0.50	ug/L	30	155	30	68	129	20
Water	Di-isopropyl ether	108-20-3	1.00	0.50	0.25	ug/L	50	150	30	67	128	20
Water	Ethyl methacrylate	97-63-2	1.00	0.50	0.25	ug/L	70	135	30	72	126	20
Water	Ethyl tert-Butyl Ether (ETBE)	637-92-3	1.00	0.50	0.25	ug/L	60	130	30	70	127	20
Water	Ethylbenzene	100-41-4	1.00	0.50	0.25	ug/L	75	125	30	79	121	20
Water	Hexachlorobutadiene (HCBD)	87-68-3	2.00	1.00	0.50	ug/L	50	140	30	66	134	20
Water	Hexane	110-54-3	1.00	0.50	0.25	ug/L	50	150	30	48	143	20
Water	Iodomethane	74-88-4	1.00	0.50	0.25	ug/L	50	140	30	69	131	20
Water	Isobutyl alcohol	78-83-1	100.0	50.0	10.0	ug/L	50	150	30	63	133	20
Water	Isopropylbenzene (Cumene)	98-82-8	1.00	0.50	0.25	ug/L	75	125	30	72	131	20
Water	m,p-Xylene	108-38-3/106-42-3	2.00	1.00	0.50	ug/L	75	130	30	80	121	20
Water	Methacrylonitrile	126-98-7	10.0	5.00	2.50	ug/L	50	150	30	63	133	20
Water	Methyl Acetate	79-20-9	2.00	1.00	0.50	ug/L	55	150	30	56	136	20
Water	Methyl methacrylate	80-62-6	1.00	0.50	0.25	ug/L	70	135	30	67	128	20
Water	Methyl Tertiary Butyl Ether (MTBE)	1634-04-4	1.00	0.50	0.25	ug/L	65	125	30	71	124	20
Water	Methylcyclohexane	108-87-2	1.00	0.50	0.25	ug/L	60	125	30	72	132	20
Water	Methylene Chloride, or Dichloromethane	75-09-2	2.00	1.00	0.50	ug/L	55	140	30	74	124	20
Water	Naphthalene	91-20-3	2.00	1.00	0.50	ug/L	55	140	30	61	128	20
Water	n-Butylbenzene	104-51-8	1.00	0.50	0.25	ug/L	70	135	30	75	128	20
Water	n-Propylbenzene	103-65-1	1.00	0.50	0.25	ug/L	70	130	30	76	126	20
Water	o-Xylene	95-47-6	1.00	0.50	0.25	ug/L	80	120	30	78	122	20
Water	p-Isopropyltoluene	99-87-6	1.00	0.50	0.25	ug/L	75	130	30	77	127	20
Water	Propionitrile	107-12-0	10.0	5.00	2.50	ug/L	50	150	30	64	136	20
Water	sec-Butylbenzene	135-98-8	1.00	0.50	0.25	ug/L	70	125	30	77	126	20
Water	Styrene	100-42-5	1.00	0.50	0.25	ug/L	65	135	30	78	123	20
Water	tert-Amyl Alcohol (2-butanol)	75-85-4	5.0	2.5	1.25	ug/L	50	150	30	66	120	20
Water	tert-Amyl ethyl ether	919-94-8	10.0	5.00	2.50	ug/L	50	150	30	64	137	20
Water	tert-Amyl methyl ether	994-05-8	10.0	5.00	2.50	ug/L	75	125	30	68	128	20
Water	tert-Butyl alcohol	75-65-0	5.00	2.50	1.25	ug/L	60	130	30	68	129	20
Water	tert-Butylbenzene	98-06-6	1.00	0.50	0.25	ug/L	70	130	30	78	124	20
Water	Tetrachloroethene (PCE; PERC)	127-18-4	1.00	0.50	0.25	ug/L	45	150	30	74	129	20

S/W	Analyte	CAS	LOQ	LOD	DL	Units	LL	UL	RPD	LL5	UL5	RPD5
Water	Tetrahydrofuran	109-99-9	5.00	2.50	1.25	ug/L	70	130	30	57	133	20
Water	Toluene	108-88-3	1.00	0.50	0.25	ug/L	75	120	30	80	121	20
Water	Toluene-d8 (Surrogate)	2037-26-5	30	1.00	0.50	ug/L	85	120		89	112	
Water	trans-1,2-Dichloroethene (trans-1,2-DCE)	156-60-5	1.00	0.50	0.25	ug/L	60	140	30	75	124	20
Water	trans-1,3-Dichloropropene	10061-02-6	1.00	0.50	0.25	ug/L	55	140	30	73	127	20
Water	trans-1,4-Dichloro-2-butene	110-57-6	1.00	0.50	0.25	ug/L	50	150	30	43	140	20
Water	Trichloroethene (TCE)	79-01-6	1.00	0.50	0.25	ug/L	70	125	30	79	123	20
Water	Trichlorofluoromethane (CFC-11)	75-69-4	2.00	1.00	0.50	ug/L	60	145	30	65	141	20
Water	Vinyl acetate	108-05-4	5.00	2.50	1.25	ug/L	60	150	30	54	146	20
Water	Vinyl Chloride (VC)	75-01-4	1.00	0.50	0.25	ug/L	50	145	30	58	137	20
Water	Xylenes (total)	1330-20-7	3.00	1.50	0.75	ug/L	75	130	30	79	121	20

LL= Low Limit, UL = Upper Limit, RPD = relative percent difference, LL5/UL5/RPD5 are limits from QSM5, highlighted limits are in-house limits.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Method 8260B/8260C)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial demonstration with quarterly verification from LOD, LOQ. May be required annually for specific certifications.	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis with quarterly verification.	Refer to SOP QS08.			
LOQ establishment and verification	Prior to initial analysis with quarterly verification.	Refer to SOP QS08.			
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to table 3 of this SOP.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs – See table 6 2. RSD for RFs 8260B CCCs ≤ 30% and one option below: **Option 1:** RSD for each analyte ≤ 15%; **Option 2:** linear least squares regression r2 ≥0.99; **Option 3**: non-linear least squares regression (quadratic) r2 ≥ 0.99 (6 points shall be used for second order). 3. Linear curve fits should have LOQ standard level within 70%-130% when back calculated against curve.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Method specific RF criteria shall be met. **Curves forced through the origin are an acceptable option now that QSM 5.0 has been finalized.**

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B/8260C) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value. [± 25% for non-DoD 8260B; ± 30% for non-DoD 8260C]	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the sequence CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units. Note - retention times may be updated based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping).	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time. For DoD QSM 5.0, ending CCV required within 12 hour tune time.	1. Average RF for SPCCs: see table 6 2. %Difference/Drift for all target compounds and surrogates: VOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). **Ending CCV for QSM 5.0 ± 50%** [± 20% for CCCs only non-DoD 8260B ± 20% for all parameters non-DoD 8260C with up to 20% exceeding]	DoD project level approval must be obtained for each of the failed analytes or corrective action must be discussed with Group Leader/quality manager. For QSM, immediate analysis of 2 passing CCVs can be used to report unqualified data.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed, holding time has been exceeded or client has approved reporting.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B/8260C) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or daily CCV **(± 10 seconds QSM 5.0)**; EICP area within -50% to +100% of ICAL midpoint standard (non-DoD allows daily CCV).	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply qualifier to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Common laboratory contaminants, no analytes detected >LOQ	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
BS containing all analytes to be reported, including surrogates (BSD if no MS/MSD)	One per preparatory batch.	QC acceptance criteria specified by client or DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean BS recovery**. See table 1 (above) for limits used by Empirical.** Discuss marginal exceedences with Group Leader or quality manager.	Correct problem, then reprep and reanalyze the BS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid BS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix	Use BS criteria, above.	Examine the project-specific DQOs. Discuss marginal exceedences with Group Leader or quality manager.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the BS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix	MSD: For matrix evaluation, use BS acceptance criteria above. MSD or sample duplicate: RPD ≤ 30% or client specified limit (between MS and MSD or sample and sample duplicate**, ≤ 20% QSM 5.0**).	Examine the project-specific DQOs. Discuss marginal exceedences with Group Leader or quality manager.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B/8260C) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD or Client. Otherwise, in-house control limits may be used. No limits specified for Method 624. **See table 1 (above) for limits used by Empirical.**	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Surrogate recoveries qualified on report forms.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 3, Tuning 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 4, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):		
Batch Number(s)		
Sequence Number:	**Run Date:**	**Instrument ID:**
Method: 8260B/8260C/624	**Calibration Number:**	**NCR#**

QA/QC Item	Yes	No	NA	2nd Check
1. Was the autosampler tray verified against the sequence file?				
2. Is the BFB tune performed every 12 hours and are the tuning criteria met?				
3. Are the % RSDs within 15% or ≥0.99 r2 linear corr or ≥0.99 quadratic COD for all analytes in the initial calibration? Are SPCC response factor criteria met? Were retention times checked for compounds with the same spectra? Were concentrations checked for compounds with different concentrations? ≤50% resolution verified for structural isomers?				
4. Is recalculation of low standard for linear curve fits within 70%-130% (should)? List exceptions and discuss with management prior to use.				
5. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met (+/-20% DoD, +/-25% 8260B, +/-30% 8260C)? Are SPCC response factor criteria met?				
6. Does the Continuing Calibration Standard (CCV) meet the ± 20% difference criteria and IS within 50% to +100% of calibration curve midpoint? SPCC criteria met? **QSM5.0 ending CCV within 12 hours and analyte recoveries within ± 50%?**				
7. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the RL (LOD for DoD except phthalates)?				
8. Are the BS, BSD, MS, MSD within control limits and run at the desired frequency?				
9. Are all sample holding times met, analytes within calibration range, IS areas within 50% to +100% of ICAL midpoint response and surrogate recoveries within limits?				
10. Sample_____shows calculation verified from raw areas to final LIMS concentration.				
11. Reagents/Standards verified accurate on bench sheets and in LIMS?				
12. Data uploaded to LIMS with correct analysts/instrument ID reflected?				
13. Water pH values recorded on instrument sequence and entered in LIMS bench sheet?				
14. Were RE's checked against original analyses for consistency?				

Comments on any "No" response:

_____

_____

_____

_____

_____

_____

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 5, Internal Standard Association

ISRef	Internal Standard (IS)	Analyte	ISRef	Internal Standard (IS)	Analyte
900	Fluorobenzene	1,1,1-Trichloroethane	900	Fluorobenzene	Tert-Amyl Methyl Ether
900	Fluorobenzene	1,1,2-Trichloro-1,2,2-trifluoroethane	900	Fluorobenzene	tert-Butyl alcohol
900	Fluorobenzene	1,1-Dichloroethane	900	Fluorobenzene	Tetrahydrofuran
900	Fluorobenzene	1,1-Dichloroethene	900	Fluorobenzene	trans-1,2-Dichloroethene
900	Fluorobenzene	1,1-Dichloropropene	900	Fluorobenzene	Trichloroethene
900	Fluorobenzene	1,2-Dichloroethane	900	Fluorobenzene	Trichlorofluoromethane
900	Fluorobenzene	1,2-Dichloroethane-d4	900	Fluorobenzene	Vinyl acetate
900	Fluorobenzene	1,2-Dichloroethene (total)	900	Fluorobenzene	Vinyl chloride
900	Fluorobenzene	1,2-Dichloropropane	901	Chlorobenzene-d5	1,1,1,2-Tetrachloroethane
900	Fluorobenzene	1,3-Dichloropropene (total)	901	Chlorobenzene-d5	1,1,2-Trichloroethane
900	Fluorobenzene	1,4-Dioxane	901	Chlorobenzene-d5	1,2,3-Trichloropropane
900	Fluorobenzene	2,2-Dichloropropane	901	Chlorobenzene-d5	1,2-Dibromoethane (EDB)
900	Fluorobenzene	2-Butanone	901	Chlorobenzene-d5	1,3-Dichloropropane
900	Fluorobenzene	2-Chloroethyl vinyl ether	901	Chlorobenzene-d5	1-Chlorohexane
900	Fluorobenzene	4-Methyl-2-pentanone	901	Chlorobenzene-d5	2-Hexanone
900	Fluorobenzene	Acetaldehyde	901	Chlorobenzene-d5	Bromofluorobenzene
900	Fluorobenzene	Acetone	901	Chlorobenzene-d5	Bromoform
900	Fluorobenzene	Acetonitrile	901	Chlorobenzene-d5	Chlorobenzene
900	Fluorobenzene	Acrolein	901	Chlorobenzene-d5	Dibromochloromethane
900	Fluorobenzene	Acrylonitrile	901	Chlorobenzene-d5	Ethyl Methacrylate
900	Fluorobenzene	Allyl chloride	901	Chlorobenzene-d5	Ethylbenzene
900	Fluorobenzene	Benzene	901	Chlorobenzene-d5	Isopropylbenzene
900	Fluorobenzene	Bromochloromethane	901	Chlorobenzene-d5	m,p-Xylene
900	Fluorobenzene	Bromodichloromethane	901	Chlorobenzene-d5	o-Xylene
900	Fluorobenzene	Bromomethane	901	Chlorobenzene-d5	Styrene
900	Fluorobenzene	Carbon disulfide	901	Chlorobenzene-d5	Tetrachloroethene
900	Fluorobenzene	Carbon tetrachloride	901	Chlorobenzene-d5	Toluene
900	Fluorobenzene	Chloroethane	901	Chlorobenzene-d5	Toluene-d8
900	Fluorobenzene	Chloroform	901	Chlorobenzene-d5	trans-1,3-Dichloropropene
900	Fluorobenzene	Chloromethane	901	Chlorobenzene-d5	Xylenes (total)
900	Fluorobenzene	Chloroprene	902	1,4-Dichlorobenzene-d4	1,1,2,2-Tetrachloroethane
900	Fluorobenzene	cis-1,2-Dichloroethene	902	1,4-Dichlorobenzene-d4	1,2,3-Trichlorobenzene
900	Fluorobenzene	cis-1,3-Dichloropropene	902	1,4-Dichlorobenzene-d4	1,2,4-Trichlorobenzene
900	Fluorobenzene	Cyclohexane	902	1,4-Dichlorobenzene-d4	1,2,4-Trimethylbenzene
900	Fluorobenzene	Dibromofluoromethane	902	1,4-Dichlorobenzene-d4	1,2-Dibromo-3-chloropropane
900	Fluorobenzene	Dibromomethane	902	1,4-Dichlorobenzene-d4	1,2-Dichlorobenzene
900	Fluorobenzene	Dichlorodifluoromethane	902	1,4-Dichlorobenzene-d4	1,3,5-Trichlorobenzene
900	Fluorobenzene	Diethyl ether	902	1,4-Dichlorobenzene-d4	1,3,5-Trimethylbenzene
900	Fluorobenzene	Diisopropyl Ether	902	1,4-Dichlorobenzene-d4	1,3-Dichlorobenzene
900	Fluorobenzene	Ethyl tert-Butyl Ether	902	1,4-Dichlorobenzene-d4	1,4-Dichloro-2-butene (total)
900	Fluorobenzene	Hexane	902	1,4-Dichlorobenzene-d4	1,4-Dichlorobenzene
900	Fluorobenzene	Iodomethane	902	1,4-Dichlorobenzene-d4	2-Chlorotoluene
900	Fluorobenzene	Isobutyl alcohol	902	1,4-Dichlorobenzene-d4	4-Chlorotoluene
900	Fluorobenzene	Isopropyl Ether	902	1,4-Dichlorobenzene-d4	Bromobenzene
900	Fluorobenzene	Methacrylonitrile	902	1,4-Dichlorobenzene-d4	cis-1,4-Dichloro-2-butene
900	Fluorobenzene	Methyl Acetate	902	1,4-Dichlorobenzene-d4	Hexachlorobutadiene
900	Fluorobenzene	Methyl Methacrylate	902	1,4-Dichlorobenzene-d4	Naphthalene
900	Fluorobenzene	Methyl t-Butyl Ether	902	1,4-Dichlorobenzene-d4	n-Butylbenzene
900	Fluorobenzene	Methylcyclohexane	902	1,4-Dichlorobenzene-d4	n-Propylbenzene
900	Fluorobenzene	Methylene chloride	902	1,4-Dichlorobenzene-d4	p-Isopropyltoluene
900	Fluorobenzene	Propionitrile	902	1,4-Dichlorobenzene-d4	sec-Butylbenzene
900	Fluorobenzene	tert-Amyl alcohol	902	1,4-Dichlorobenzene-d4	tert-Butylbenzene
900	Fluorobenzene	tert-Amyl ethyl ether	902	1,4-Dichlorobenzene-d4	trans-1,4-Dichloro-2-butene

Table 6, 8260 SPCC criteria (Minimum Response Factors)

Volatile Compounds	8260B	8260C
1,1,1-Trichloroethane		0.1
1,1,2,2-Tetrachloroethane	0.3	0.3
1,1,2-Trichloro-1,2,2-trifluoroethane		0.1
1,1,2-Trichloroethane		0.1
1,1-Dichloroethane	0.1	0.2
1,1-Dichloroethene		0.1
1,2,4-Trichlorobenzene		0.2
1,2-Dibromo-3-chloropropane		0.05
1,2-Dibromoethane		0.1
1,2-Dichlorobenzene		0.4
1,2-Dichloroethane		0.1
1,2-Dichloropropane		0.1
1,3-Dichlorobenzene		0.6
1,4-Dichlorobenzene		0.5
2-Butanone		0.025*
2-Hexanone		0.1
4-Methyl-2-pentanone		0.1
Acetone		0.025*
Benzene		0.5
Bromodichloromethane		0.2
Bromoform	0.1	0.1
Bromomethane		0.1
Carbon disulfide		0.1
Carbon tetrachloride		0.1
Chlorobenzene	0.3	0.5
Chloroethane		0.1
Chloroform		0.2
Chloromethane	0.1	0.1
cis-1,2-Dichloroethene		0.1
cis-1,3-Dichloropropene		0.2
Cyclohexane		0.1
Dibromochloromethane		0.1
Dichlorodifluoromethane		0.1
Ethylbenzene		0.1
Isopropylbenzene		0.1
meta-/para-Xylene		0.1
Methyl Acetate		0.1
Methyl tert-Butyl Ether		0.1
Methylcyclohexane		0.1
Methylene chloride		0.1
ortho-Xylene		0.3
Styrene		0.3
Tetrachloroethene		0.2
Toluene		0.4
trans-1,2-Dichloroethene		0.1
trans-1,3-Dichloropropene		0.1
Trichloroethene		0.2
Trichlorofluoromethane		0.1
Vinyl chloride		0.1

* As the SW-846 Methods Information Communication Exchange Service (MICE Service) has confirmed the minimum response factor criteria listed in Table 4 of 8260C are recommendations and not mandatory, Empirical Laboratories has assigned Acetone and 2-Butanone alternate minimum response factor criteria of 0.025 in place of the 0.1 indicated in Table 4 of 8260C. Documentation that Acetone/2-Butanone sensitivity is adequate for the needs of the project can be provided upon client request. This exceeds National Functional Guidelines 2013 criteria of 0.01.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 211 **REVISION #: 31** **EFFECTIVE DATE: 20160812**

GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD) ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS (PCB) BY EPA METHOD 608/608.2 or SW846 METHOD 8081A/8082 or 8081B/8082A

APPROVALS:

Lab Director: _____ Date: **20160812**

Data Quality Manager: _____ Date: **20160812**

Group Leader: _____ Date: **20160812**
Jade Holliman

SOP211_R31_20160812_PP

- Added reference to 8000D and removed 8000B.
- Table 1 updated for in-house limits with headers defined
- LLOQ and IDP terminology added from SW-846 Update V.
- MDL/LOD/LOQ references simplified in table 2.
- Revised checklist to include linear calibration read-back with list/discuss.

SOP211_R30_20151221_PP

- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.

SOP211_R29_20150921_PP

- All references to supervisor updated to reflect group leader.
- Section 12 updated to reference QS04.
- Table 1 updated with in-house limits, where appropriate.
- Table 2 updated with LOQ marginal exceedences and 2 passing CCV allowance.
- Column and software references updated throughout.
- BS standard source updated.

SOP211_R28_20140730_PP

- Watermark update to include proprietary reference.
- MSDS sheet reference updated to reflect vendor website access.
- Identification references updated from 8081A/8082 to 8081B/8082A.
- Tables 1 and 2 updated to reflect QSM4.2 and QSM5.0 specifications.

Revision 27, 20140207

- Sections were updated to accommodate QSM5.0 additions for 13.0 Equipment/Instrument Maintenance, 14.0 Computer Hardware and Software plus 17.0 Troubleshooting. Also added section 22.0, Corrective actions and out-of-control data.
- Section 12 references to pesticide spiking solution were clarified to reference single component pesticides.
- Section 16 reference to Mirex removed as it is no longer used. Other standard makeup details were updated and the reference to RT update to the midpoint of the curve removed as a CCV is always analyzed prior to sample analysis. Multi-component quantitation clarified to require a minimum of 3 peaks.
- Analyst Data Review Checklist replaced.

Revision 26, 20121217

- Updated references to Chlordane (technical) to reflect Chlordane (n.o.s.) – not otherwise specified.
- Removed references to Toxaphene and Chlordane (single point) in table 14.5.1.
- Clarified BS and MS/MSD requirements in section 12.
- Updated analyst review checklist.
- Update table 2 to clarify reporting results with correction of "M" qualifier for 100% difference rather than the incorrect 10% originally reflected.

Revision 25, 20120820

- Retention time window determination updated to reflect default windows used.

- LIMS standard documentation updated to include scanned COA process.
- Toxaphene/Chlordane updated to reflect full calibration rather than single-point.
- Example sequence run log updated to reflect CCVs after 10 field samples. Minimum frequency updated to reflect every 10 field samples or 12 hours maximum.
- Table 1 limits updated.
- Table 2 updated for LOD/LOQ and "M" qualifier.
- Table 3 and 4 removed with subsequent table re-numbering and updated references in body of SOP.
- Table 3 updated for LIMS upload with correct primary analyst and sample reflecting full re-calculation.
- Table 4 updated for standard concentrations on toxaphene and chlordane.

Revision 24, 20110515
- Added reference to QS04 in section 14.10.1.
- Added reference to Table 6 in section 14.10.2.
- Added Table 6 to end of this SOP.

Revision 23, 20100920
- All temperature references of 1°C-4.4°C were revised to reflect 0°C-6°C.

Revision 22, 07/07/10
- The SOP has been updated to move specific requirements to tables at the back of the SOP and add Mirex, PCB-1262, PCB-1268 as analytes.

Revision 21, 04/11/10
- The SOP is an update from Revision 20 dated 04/27/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD) ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS (PCB) BY EPA METHOD 608/608.2 or SW846 METHOD 8081A/8082 or 8081B/8082A

1.0 Identification of the Test Method

This SOP is compliant with SW-846 Methods 8081A/8082 and 8081B/8082A. *Federal Register* Method 608/608.2 and CLP Method for Pesticides have also been used in the development of this SOP.

2.0 Applicable Matrix or Matrices

This Standard Operating Procedure, SOP, is used for the analysis of Pesticide/PCB organic compounds in a variety of matrices (soils, sediments, waters, etc.).

3.0 Limits of Detection and Quantitation

Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Detection Limit/Method Detection Limit, Limit of Detection and Reporting Limit/Limit of Quantitation (Lower Limit of Quantitation or LLOQ) for each analyte.

4.0 Scope of Application, Including Parameters to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Detection Limit/Method Detection Limit, Limit of Detection and Reporting Limit/Limit of Quantitation for each analyte.

4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the ECD. Pesticide analytes are identified and confirmed based on the retention time of known standards. PCB and multi-component pesticide analytes are identified based on pattern recognition and retention time. Analytes are quantitated relative to known standards using the external standard method.

6.0 Definitions

Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

Section 3.0 of SW-846 Methods 8081A/8082 and Section 4.0 of Methods 8081B/8082A details interferences and potential problems which may be encountered when dealing with pesticide/PCB analyses. Please see sample clean-up SOPs (307, 308 and 309) to evaluate possible clean-up options for any encountered interferences.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

8.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.

8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes in the LIMS.

8.4 MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your section group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9.0 Equipment & Supplies

9.1 GC's:

9.1.1 Agilent 6890N- complete with temperature programmable gas chromatograph suitable for split/splitless injection.

9.2 Columns:

9.2.1 Restek Siltek Guard Column (or equivalent): 10 meter x 0.32 mm ID

9.2.2 DB-CLP1 (or equivalent): 30 meter x 0.32 mm ID x 0.25 µm film thickness fused silica column.

9.2.3 DB-CLP2 (or equivalent): 30 meters x 0.32 mm ID x 0.5 µm film thickness fused silica column.

9.3 Autosamplers:

9.3.1 Agilent 7683 autosamplers capable of reproducibility from one injection to another, proven by meeting QC and calibration criteria.

10.0 Equipment/Instrument Maintenance

10.1 Cool oven & injection port to 40°C and remove the injection tower.

10.2 Remove the septum nut and replace septum.

10.3 Remove upper weldment and replace glass liner and o-ring.

10.4 Remove the column nut & injection port nut and replace the inlet seal (gold).

10.5 Clip/discard about a loop (40-55cm) off the inlet of the guard column. Measure 28mm from the back of the column nut to the tip of the guard column inlet and place back into the bottom of the injection port.

10.6 Close the oven door, replace injection tower, fill solvent vials, and load the appropriate ChemStation method on the acquisition PC.

10.7 Bake injection port @ 300°C and oven @ 320°C ~30min. (or longer depending on detector signal).

11.0 Computer Hardware and Software

11.1 Computers:

ECD3: Compaq EVO D310 running Windows 2000 SP-4.

ECD4: Dell Optiplex GX620 running Windows 2000 SP-4.

11.2 Acquisition Software: HP Chemstation system Rev.A.09.01 [1206] is interfaced to both ECD3 and ECD4. The system acquires and stores data throughout the chromatographic program.

11.3 Data Processing Software: ChemStation/Enviroquant data system is interfaced to the HP Chemstation. The system accepts, processes and stores acquired data.

12.0 Reagents and Standards

Quality Systems SOP QS04 " TRACEABILITY AND EXPIRATION DATES OF TEST - RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

12.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the reagents and standards used for the performance of the method. **All reagents shall be made from ACS reagent grade chemicals. All reagents used for preparation and analysis are entered into LIMS. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the associated samples.**

12.2 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the COA and recorded in the LIMS. The date they are opened is recorded in the LIMS along with their lot number and vendor and given a sequential number. Each standard that is prepared is recorded in the LIMS and given a sequential number. All certificates received with standards must be marked with the LIMS ID and forwarded to the administration department for scanning/saving in order to be available for review within LIMS. The following are noted in the LIMS: standard makeup, solvent used, date received, date opened, date prepared, expiration date and analyst. Each standard label is completed with the standard number, name, concentration, expiration date, and analyst initials. All stocks and standards are stored in the refrigerator at a temperature of 0°C-6°C from the date they are received/prepared. The refrigerator and freezer temperature is monitored daily with a calibrated thermometer and recorded with calibration correction in the GC refrigerator temperature logbook. See **Table 4** for information on standard sources/calibration concentrations.

12.3 Reagent: Hexane - pesticide quality or equivalent.

13.0 Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 0°C-6°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 0°C-6°C. Water samples have a holding time of 7 days from date of sampling while soil samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed.

14.0 Quality Control

14.3 Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

14.4 Surrogates - All samples and QC are spiked with surrogates prior to extraction. See **Table 2** for criteria and corrective action.

14.5 BS Sample - The BS is extracted 1 pesticide single-component spike per pesticide extraction batch of up to 20 samples and/or 1 PCB per PCB extraction batch of up to 20 samples to provide accuracy results. It is spiked using an intermediate standard from vendor stock. See **Table 2** for criteria and corrective action. When Pesticides and PCBs are extracted together, the pesticide BS is generally BS1 and the PCB BS is generally BS2.

14.6 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See **Table 2** for criteria and corrective action.

14.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, (1 pesticide single-component spike per pesticide extraction batch of up to 20 samples and/or 1 PCB per PCB extraction batch of up to 20 samples) if sample is available. If no sample is available, a BSD must be extracted to provide precision results. See **Table 2** for criteria and corrective action. Some factors that may affect MS/MSD results are indicated in section 17.0.

15.0 Calibration and Standardization

15.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

15.2 See Section 16.4 for Calibration details.

16.0 Procedure

16.1 The GC/ECD should be primed by injecting a single-component pesticide standard at 200-500 µg/L and/or PCB standard at 2,500 µg/L, 2-5 times more concentrated than the mid-level standard. Inject this prior to beginning initial or daily calibration.

16.2 Chromatographic conditions:

16.2.1 DB-CLP1/CLP2 columns:

GC	ECD3
Purge on	60ml/min at 0.50 min.
Injector/Detector temperature	250/340°C
Column flow	3.0 mL/min
Initial column temperature	100°C for 1.0 minutes
Temperature ramp	35°C/min
Intermediate column temperature	220°C for 0.0 minutes
Second Temperature Ramp	15°C/min
Final Column Temperature	340°C for 2.0 minutes

16.2.2 DB-CLP1/CLP2columns:

GC	ECD4
Purge on	60ml/min at 0.50 min.
Injector/Detector temperature	250/350°C
Column flow	3.0 mL/min
Initial column temperature	100°C for 1.0 minutes
Temperature ramp	35°C/min
Intermediate column temperature	220°C for 0.0 minutes
Second Temperature Ramp	15°C/min
Final Column Temperature	340°C for 2.0 minutes

Note: Current gas chromatograph conditions can be confirmed in the corresponding maintenance log.

16.3 Eval Mix – Before pesticide calibration and/or sample analysis (every 12 hours thereafter for DoD work), a degradation check standard (evaluation mix) of endrin and 4,4'-DDT must be injected. Degradation of either compound must not exceed 15 percent. See **Table 2** for criteria and corrective action.

16.4 Calibration

16.4.1 Initial Calibration - An initial multi-point calibration curve must be prepared in hexane, analyzed and shown to meet the initial calibration criteria before any sample analyses may be performed. Check acceptability of linear and quadratic curve fits by re-calculating each calibration point back to the curve. See **Table 2** for criteria and corrective action. See **Table 4** for standard concentrations/sources and below for makeup of the intermediates. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not

exceed the linear range of the detector. For single component pesticides and surrogates, a seven point calibration is injected and analyzed for each analyte of interest. For Toxaphene and Chlordane (n.o.s.), a six-point calibration is injected and analyzed. Initial calibration for Aroclors may be accomplished by using a six-point curve that contains Aroclors 1016 and 1260. The mixture of these two Aroclors contains many of the peaks represented in the other Aroclor mixtures (1221, 1232, 1242, 1248, 1254, 1262 & 1268). Full calibration is required if they are expected/detected. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

Mix A/B (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 500µL of A/B Mix and 500µL Surrogate are injected into a 10mL volumetric flask containing approximately 8mL hexane and diluted to volume with same to make a 10 µg/mL standard.*

Chlordane (n.o.s.) Calibration Intermediate Solution: Using a 100µL syringe, 100µL of Chlordane (n.o.s.) is injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 1.0 µg/mL standard.*

Toxaphene Calibration Intermediate Solution: Using a 1000µL syringe, 1000µL of Toxaphene is injected into a 10mL volumetric flask containing approximately 8.5mL hexane and diluted to volume with same to make a 100 µg/mL standard.*

Aroclor 1016/1260 (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 500µL of Aroclor 1016/1260 and 250µL Surrogate are injected into a 10mL volumetric flask containing approximately 8.0mL hexane and diluted to volume with same to make a 50 µg/mL and 5ug/ml standard.*

*After capping and inverting several times, all solutions are transferred into labeled, 12ml, teflon-lined, screw-capped vials and stored in the refrigerator at 0°C-6°C or less for up to 6 months. These standards are used to make the calibration curve standards in hexane at the concentrations found in Table 4.

16.4.2 Initial Calibration Verification - A second source standard must be prepared in hexane, analyzed and calculated against the initial calibration curve, then shown to meet the ICV criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 4** for standard concentrations/sources. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

16.4.3 Continuing Calibration Verification (CCV) - Every 10 field samples – maximum 12 hours - (and at the end of the analysis sequence), a CCV must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 4** for standard concentrations/sources. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All

integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

16.4.4 RT Windows - Retention time criteria set forth in SW-846 method 8000D Section 11.6 are used to set retention time windows. As generated in-house retention time windows are less than +/-0.03 minutes, the windows default to +/-0.03 minutes. Retention times are updated with the first CCV of the day.

16.5 Samples - Prior to using Method 608, SW-846 8081A, 8081B, 8082, 8082A or CLP (pesticide method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3541, 3546, 3640, 3550, 3580, EPA method 608 or CLP).

16.5.1 Example of a sequence run log:

1-Primer A/B Mix or Primer PCB (1-2x high point conc.)
2- EVAL Mix (Pest only)
3- CCV A/B Mix
4- CCV Toxaphene
5-CCV Chlordane (n.o.s.)
6- CCV PCB 1660
7- Method Blank
8-BS A/B Mix
9-BS PCB
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample
15-Sample
16-Sample
17-Sample
18-Sample
19-Sample
20- CCV A/B Mix
21- CCV Toxaphene
22-CCV Chlordane (n.o.s.)
23-CCV PCB
24-Sample
25-Sample-MS
26-Sample-MSD
27-Sample
28-Sample
29-Sample
30-Sample
31-Sample
32-Sample
33- CCV A/B Mix
34- CCV Toxaphene
35-CCV Chlordane (n.o.s.)
36-CCV PCB

16.6 Data Reduction/Evaluation - Each sample analysis sequence is documented in the run logbook for the instrument. After the sample has been analyzed, the data is processed through the ChemStation/Enviroquant data system. Quantitative measurements are performed as described in SW-846 8081A Section 7.5.6, and SW-846 8081B Section 11.5.6.1. The following must be checked to determine if the sample will need any reanalysis, cleaning or dilution. Criteria and corrective action are found in **Table 2**. Formal data evaluation is detailed in SOP QS05 and documented using the Analyst Data Review Checklist (see Table 3). Manual integration guidance is found in SOP QS07.

16.6.1 Analyte concentration after rounding to 3 significant figures must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the mid-range to the top half of the curve.

16.6.2 If the sample shows signs of sulfur contamination in the time range where sulfur compounds elute a sulfur cleanup is required [see SOP-307].

16.6.3 If the sample has extraneous peaks eluting in the chromatogram an acid cleanup is required for PCB samples and may be applicable for certain pesticides, (acid clean-up may be required for all PCB samples, check with your group leader), [see SOP-308].

16.6.4 Analyte quantitation verification.

16.7 Identification/Quantitation [See SW-846 method 8081B Sections 11.5/11.6 or method 8082A Section 11.7/11.8].

16.7.1 Single peak components are identified by retention time on a primary column with confirmation by retention time on a secondary or confirmation column. Which column is used for primary/confirmation is determined by the chromatography in the region of the compound and the logic outlined in SOP QS03.

16.7.1.1 Due to coelution of certain compounds confirmation for all analytes may not be achieved. The analyst must use experience and judgment to decide if the compound is there. If a call is made, the data should be qualified appropriately.

16.7.1.2 If a compound is outside of its window on one column but in the window on the other column, the analyst will need to use their judgment or seek guidance from the organic section group leader or another experienced analyst to determine if the analyte is present.

16.7.2 Multi-peak components (PCB's, Toxaphene and Chlordane (n.o.s.)) are identified by pattern recognition using an on scale standard chromatogram to compare to an on scale sample chromatogram enabling the analyst to judge whether the sample pattern matches a standard pattern. See **Table 5** for example chromatograms of multi-component analytes. Confirmation of multi-peak components is required by the method and may be accomplished in several ways. If the sample is from a source known to contain specific Aroclors then this information may be used as a confirmation. Documentation of this approach must meet the requirements outlined in Sec. 11.7.3 of SW-846 Method 8082A. Another approach is to use a column of dissimilar stationary phase and compare the pattern to a known Aroclor standard. Finally if the concentration is high enough GC/MS may be used as confirmation.

A. Generally, five unique peaks representing the full range of the multi-peak component are used in the quantitation of the multi-peak components.

B. Multi-peak components that still have matrix interference after appropriate sample cleanup steps have been taken may need to be hand calculated using peaks that do not have interference. This should be brought to the organic section group leader's attention. A minimum of 3 peaks is needed for quantitation.

C. Multi-peak components that exhibit a weathered pattern may need to be hand calculated by the analyst. The analyst will need to use peaks that exhibit the full range of weathering. The number of peaks used to quantitate the multi-peak component will depend on the analyst's judgment of what it will take to achieve the truest concentration of the component. This should be brought to the organic section group leader's attention. A minimum of 3 peaks is needed for quantitation.

16.7.3 Quantitation – Once a compound has been identified qualitatively, the concentration must then be quantitated. Calculations follow in Section 18.0.

17.0 Troubleshooting – If routine maintenance in section 10.0 did not bring the instrument back into control, more involved maintenance may be required (i.e. detector maintenance, analytical column maintenance, column splitter, etc.). Bring to the attention of the Section Group leader.

18.0 Data Analysis and Calculations

18.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

18.2 Calculate the calibration factor (CF) for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

18.3 The mean CF is calculated as follows:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CFi}{n}$$

$$\text{AvgCF} = \frac{\square\square \text{ CF for each standard)}}{\text{N}}$$

18.4 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i-1}^{n} (CFi - \overline{CF})^2}{n-1}}$$

$$RSD = \frac{SD}{CF} \times 100$$

18.5 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\%\ \text{Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\%\ \text{Difference} = \frac{(\text{CCV CF} - \text{Average CF}) * 100}{\text{Average CF}}$$

where CCV CF is the calibration factor from the analysis of the verification standard and mean CF is the average calibration factor from the initial calibration.

18.6 Concentration in water samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration}\ (\mu\text{g/L}) = \frac{(A_x)(V_t)(D)}{(\overline{CF})(V_i)(V_s)}$$

where:

A_x = Area (or height) of the peak for the analyte in the sample.
V_t = Total volume of the concentrated extract (µL).
D= Dilution factor, if the sample was diluted prior to analysis.
If no dilution was made, D = 1. The dilution factor is always dimensionless.
V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.
CF = Mean response factor from the initial calibration.
V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

18.7 Concentration in non-aqueous samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.]

$$\text{Concentration}\ (\mu\text{g/kg}) = \frac{(A_x)(V_t)(D)}{(\overline{CF})(V_i)(W_s)}$$

where:

A_x, V_t, D, and CF are the same as for aqueous samples, and
W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

19.0 Method Performance

Initial Demonstration of Capability (DOC)/Performance (IDP): Each analyst must perform an IDOC/IDP prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples (may be prepared from same source as calibration). The data is calculated for accuracy and precision requirements. The IDOC form is completed by each analyst and then provided to the group leader for further processing and approval. See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

19.1 Method Detection Limit Study or Detection Limit Determination
19.2 Limit of Detection Verification
19.3 Limit of Quantitation or Reporting Limit Verification
19.4 Initial Demonstration of Capability (IDOC)/Performance (IDP)
19.5 PT Studies

20.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

21.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

22.0 Corrective actions and out-of-control data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

23.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

23.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

23.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a non-conformance report to document the problem.

23.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a non-conformance report to document the problem.

23.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your group leader who should discuss the problem with the client/project manager to determine the action to be taken.

24.0 Waste Management

Please see Waste Disposal, SOP QS14 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

25.0 References

25.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Method 8081A, 8081B, 8082, 8082A

25.2 USEPA Code of Federal Regulations, 40, CH 1, PT 136; Method 608, 608.2; APX-B

25.3 USEPA Contract Laboratory Program (CLP) for Organics ILM04.2; ILM04.3

25.4 DOD Quality Systems Manual, Ver. 3/4.1

25.5 DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003.) Dated 10/25/2010

25.6 DoD Quality Systems Manual for Environmental Laboratories version 5.0, 7/2013 [Based on ISO/IEC 17025:2005(E) and the NELAC Institute (TNI) Standards, Volume 1, (September 2009)].

26.0 Tables, Diagrams, Flowcharts and Validation Data

26.1 Table 1, DL, LOD, LOQ and recovery limits

26.2 Table 2, QA/QC summary table

26.3 Table 3, Data Review Checklist

26.4 Table 4, Calibration Standards

26.5 Table 5, Example Chromatograms

S/W	Method	Analyte	CAS	LOQ	LOD	DL	Units	LL	UL	LL5	UL5	SOP	Notes
Solid	8081B	4,4'-DDD	72-54-8	0.670	0.340	0.170	ug/Kg	30	135	56	139	343/211	
Solid	8081B	4,4'-DDE	72-55-9	0.670	0.340	0.170	ug/Kg	70	125	56	134	343/211	
Solid	8081B	4,4'-DDT	50-29-3	0.670	0.340	0.170	ug/Kg	45	140	50	141	343/211	
Solid	8081B	Aldrin	309-00-2	0.67	0.340	0.110	ug/Kg	45	140	45	136	343/211	
Solid	8081B	alpha-BHC (alpha-HCH)	319-84-6	0.670	0.340	0.110	ug/Kg	60	125	45	137	343/211	
Solid	8081B	alpha-Chlordane	5103-71-9	0.670	0.340	0.110	ug/Kg	65	120	54	133	343/211	
Solid	8081B	beta-BHC (beta-HCH)	319-85-7	0.670	0.340	0.110	ug/Kg	60	125	50	136	343/211	
Solid	8081B	Chlordane (n.o.s.)	57-74-9	3.4	1.7	1.14	ug/Kg	50	150	43	149	343/211	
Solid	8081B	Decachlorobiphenyl (Surrogate)	2051-24-3	0.670	0.340	0.110	ug/Kg	60	125	52	129	343/211	LAB
Solid	8081B	delta-BHC (delta-HCH)	319-86-8	0.670	0.340	0.110	ug/Kg	55	130	47	139	343/211	
Solid	8081B	Dieldrin	60-57-1	0.670	0.340	0.170	ug/Kg	65	125	56	136	343/211	
Solid	8081B	Endosulfan I	959-98-8	0.670	0.340	0.110	ug/Kg	15	135	53	132	343/211	
Solid	8081B	Endosulfan II	33213-65-9	0.670	0.340	0.170	ug/Kg	35	140	53	134	343/211	
Solid	8081B	Endosulfan sulfate	1031-07-8	0.670	0.340	0.170	ug/Kg	60	135	55	136	343/211	
Solid	8081B	Endrin	72-20-8	0.670	0.340	0.170	ug/Kg	60	135	57	140	343/211	
Solid	8081B	Endrin aldehyde	7421-93-4	0.670	0.340	0.170	ug/Kg	35	145	35	137	343/211	
Solid	8081B	Endrin ketone	53494-70-5	0.670	0.340	0.170	ug/Kg	65	135	55	136	343/211	
Solid	8081B	gamma-BHC (Lindane; gamma-HCH)	58-89-9	0.670	0.340	0.110	ug/Kg	60	125	49	135	343/211	
Solid	8081B	gamma-Chlordane	5103-74-2	0.670	0.340	0.110	ug/Kg	65	125	53	135	343/211	
Solid	8081B	Heptachlor	76-44-8	0.670	0.340	0.110	ug/Kg	50	140	47	136	343/211	
Solid	8081B	Heptachlor epoxide	1024-57-3	0.670	0.340	0.110	ug/Kg	65	130	52	136	343/211	
Solid	8081B	Methoxychlor	72-43-5	0.670	0.340	0.110	ug/Kg	55	145	52	143	343/211	
Solid	8081B	Tetrachloro-m-xylene (Surrogate)	877-09-8	0.670	0.340	0.110	ug/Kg	70	125	42	129	343/211	
Solid	8081B	Toxaphene	8001-35-2	33.0	22.0	11.0	ug/Kg	50	150	33	141	343/211	
Water	8081B	4,4'-DDD	72-54-8	0.0200	0.0100	0.00500	ug/L	25	150	56	143	302/211	
Water	8081B	4,4'-DDE	72-55-9	0.0200	0.0100	0.00500	ug/L	35	140	57	135	302/211	
Water	8081B	4,4'-DDT	50-29-3	0.0200	0.0100	0.00500	ug/L	45	140	51	143	302/211	
Water	8081B	Aldrin	309-00-2	0.0200	0.0100	0.00330	ug/L	25	140	45	134	302/211	
Water	8081B	alpha-BHC (alpha-HCH)	319-84-6	0.0200	0.0100	0.00330	ug/L	60	130	54	138	302/211	
Water	8081B	alpha-Chlordane	5103-71-9	0.0200	0.0100	0.00330	ug/L	65	125	60	129	302/211	
Water	8081B	beta-BHC (beta-HCH)	319-85-7	0.0200	0.0100	0.00330	ug/L	65	125	56	136	302/211	
Water	8081B	Chlordane (n.o.s.)	57-74-9	0.100	0.0500	0.0170	ug/L	50	150	62	140	302/211	
Water	8081B	Decachlorobiphenyl (Surrogate)	2051-24-3	0.0200	0.0100	0.00330	ug/L	40	135	26	131	302/211	LAB
Water	8081B	delta-BHC (delta-HCH)	319-86-8	0.0200	0.0100	0.00330	ug/L	45	135	52	142	302/211	
Water	8081B	Dieldrin	60-57-1	0.0200	0.0100	0.00500	ug/L	60	130	60	136	302/211	
Water	8081B	Endosulfan I	959-98-8	0.0200	0.0100	0.00330	ug/L	50	110	62	126	302/211	
Water	8081B	Endosulfan II	33213-65-9	0.0200	0.0100	0.00500	ug/L	30	130	52	135	302/211	

S/W	Method	Analyte	CAS	LOQ	LOD	DL	Units	LL	UL	LL5	UL5	SOP	Notes
Water	8081B	Endosulfan sulfate	1031-07-8	0.0200	0.0100	0.00500	ug/L	55	155	62	133	302/211	
Water	8081B	Endrin	72-20-8	0.0200	0.0100	0.00500	ug/L	55	135	60	138	302/211	
Water	8081B	Endrin aldehyde	7421-93-4	0.0200	0.0100	0.00500	ug/L	55	135	51	132	302/211	
Water	8081B	Endrin ketone	53494-70-5	0.0200	0.0100	0.00500	ug/L	75	125	58	134	302/211	
Water	8081B	gamma-BHC (Lindane; gamma-HCH)	58-89-9	0.0200	0.0100	0.00330	ug/L	25	135	59	134	302/211	
Water	8081B	gamma-Chlordane	5103-74-2	0.0200	0.0100	0.00330	ug/L	60	125	56	136	302/211	
Water	8081B	Heptachlor	76-44-8	0.0200	0.0100	0.00330	ug/L	40	130	54	130	302/211	
Water	8081B	Heptachlor epoxide	1024-57-3	0.0200	0.0100	0.00330	ug/L	60	130	61	133	302/211	
Water	8081B	Methoxychlor	72-43-5	0.0200	0.0100	0.00330	ug/L	55	150	54	145	302/211	
Water	8081B	Tetrachloro-m-xylene (Surrogate)	877-09-8	0.0200	0.0100	0.00330	ug/L	25	140	44	124	302/211	
Water	8081B	Toxaphene	8001-35-2	1.00	0.667	0.330	ug/L	50	150	33	134	302/211	
Solid	8082A	Aroclor-1016	12674-11-2	16.7	8.33	4.17	ug/Kg	40	140	47	134	343/211	Routine 1016/1260 spike
Solid	8082A	Aroclor-1221	11104-28-2	16.7	8.33	4.17	ug/Kg	*40*	*140*	*47**	*134**	343/211	**1016 limits used if this aroclor spiked.*
Solid	8082A	Aroclor-1232	11141-16-5	16.7	8.33	4.17	ug/Kg	*40*	*140*	*47**	*134**	343/211	**1016 limits used if this aroclor spiked.*
Solid	8082A	Aroclor-1242	53469-21-9	16.7	8.33	4.17	ug/Kg	*40*	*140*	*47**	*134**	343/211	**1016 limits used if this aroclor spiked.*
Solid	8082A	Aroclor-1248	12672-29-6	16.7	8.33	4.17	ug/Kg	*40*	*140*	*53**	*140**	343/211	**1260 limits used if this aroclor spiked.*
Solid	8082A	Aroclor-1254	11097-69-1	16.7	8.33	4.17	ug/Kg	*40*	*140*	67	135	343/211	Limits if 1254 aroclor spiked
Solid	8082A	Aroclor-1260	11096-82-5	16.7	8.33	4.17	ug/Kg	60	130	53	140	343/211	Routine 1016/1260 spike
Solid	8082A	Aroclor-1262	37324-23-5	16.7	8.33	4.17	ug/Kg	*40*	*140*	*53**	*140**	343/211	**1260 limits used if this aroclor spiked.*
Solid	8082A	Aroclor-1268	11100-14-4	16.7	8.33	4.17	ug/Kg	*40*	*140*	*53**	*140**	343/211	**1260 limits used if this aroclor spiked.*
Solid	8082A	Decachlorobiphenyl (Surrogate)	2051-24-3	0.670	0.340	0.110	ug/Kg	60	125	52	129	343/211	LAB
Solid	8082A	Tetrachloro-m-xylene (Surrogate)	877-09-8	0.670	0.340	0.110	ug/Kg	70	125	44	130	343/211	
Water	8082A	Aroclor-1016	12674-11-2	0.500	0.250	0.125	ug/L	25	145	46	129	302/211	Routine 1016/1260 spike
Water	8082A	Aroclor-1221	11104-28-2	0.500	0.250	0.125	ug/L	*30*	*145*	*46**	*129**	302/211	**1016 limits used if this aroclor spiked.*
Water	8082A	Aroclor-1232	11141-16-5	0.500	0.250	0.125	ug/L	*30*	*145*	*46**	*129**	302/211	**1016 limits used if this aroclor spiked.*
Water	8082A	Aroclor-1242	53469-21-9	0.500	0.250	0.125	ug/L	*30*	*145*	*46**	*129**	302/211	**1016 limits used if this aroclor spiked.*
Water	8082A	Aroclor-1248	12672-29-6	0.500	0.250	0.125	ug/L	*30*	*145*	*45**	*134**	302/211	**1260 limits used if this aroclor spiked.*
Water	8082A	Aroclor-1254	11097-69-1	0.500	0.250	0.125	ug/L	*30*	*145*	34	127	302/211	Limits if 1254 aroclor spiked
Water	8082A	Aroclor-1260	11096-82-5	0.500	0.250	0.125	ug/L	30	145	45	134	302/211	Routine 1016/1260 spike
Water	8082A	Aroclor-1262	37324-23-5	0.500	0.250	0.125	ug/L	*30*	*145*	*45**	*134**	302/211	**1260 limits used if this aroclor spiked.*
Water	8082A	Aroclor-1268	11100-14-4	0.500	0.250	0.125	ug/L	*30*	*145*	*45**	*134**	302/211	**1260 limits used if this aroclor spiked.*
Water	8082A	Decachlorobiphenyl (Surrogate)	2051-24-3	0.0200	0.0100	0.00330	ug/L	40	135	26	131	302/211	LAB
Water	8082A	Tetrachloro-m-xylene (Surrogate)	877-09-8	0.0200	0.0100	0.00330	ug/L	25	140	44	124	302/211	From 8081B

LL= Low Limit, UL = Upper Limit, LL5/UL5 are limits from QSM5, highlighted limits are in-house limits.

Table 2. Organic Analysis by Gas Chromatography (Methods 8081, 8082)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial demonstration with quarterly verification from LOD, LOQ. May be required annually for specific certifications.	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis with quarterly verification.	Refer to SOP QS08.			
LOQ establishment and verification	Prior to initial analysis with quarterly verification.	Refer to SOP QS08.			
Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 15% for both DDT and Endrin.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 15% for both DDT and Endrin.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: **Option 1**: RSD for each analyte ≤ 20%; **Option 2:** linear least squares regression: $r \geq 0.995$; ($r^2 \geq 0.990$) **Option 3:** non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order). Note: Linear/quadratic curve fits should have calibration standards 70%-130% when back calculated against curve.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration, if detected. Results may not be quantitated using a single point.

Table 2. Organic Analysis by Gas Chromatography (Methods 8081, 8082) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL;	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate for DoD analyses.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence. Multi-component pesticides required only before sample analysis.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL;	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification. 2 passing CCVs immediately following failing CCV allows reporting of unqualified data	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem, then see SOP QS05. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (BS) containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean BS recovery.	Correct problem, then reprep and reanalyze the BS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid BS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table 2. Organic Analysis by Gas Chromatography (Methods 8081, 8082) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix.	For matrix evaluation, use BS acceptance criteria specified by DoD, if available. Otherwise, use in-house BS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the BS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation, use BS acceptance criteria specified by DoD, if available. Otherwise, use in-house BS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Qualify surrogate results on form I.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply "P" qualifier if RPD > 40% (define in the case narrative. Apply "M" qualifier if RPD > 100% (defined in case narrative). Report lower result.	Use project-specific reporting requirements if available; otherwise, report the result from the higher column.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 3, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):		
Sequence Number:	**Run Date:**	**Instrument ID:**
Method: **8081/8082**	**Calibration:**	**NCR:**

QA/QC Item	Yes	No	NA	2nd Review
A. Initial Calibration				
1. Did the evaluation mix pass criteria?				
2. Does the curve consist of at least five Calibration Standards (six for quadratic curve)?				
3. Is the low standard less than or equal to the MRL/LOQ?				
4. Are the %RSD or fit criteria within QC limits for all analytes?				
5. Is recalculation of calibration points for linear/quadratic curve fits within 70%-130% (should)? List exceedences and discuss with management prior to use.				
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 10 samples or every 12 hours and at the end of the sequence?				
2. Are the % differences within QC limits for all analytes?				
D. Sample Analysis				
1. Did the evaluation mix pass criteria?				
2. Are all sample holding times met?				
3. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?				
4. For single peak analytes - are all compounds identified on the primary column confirmed on the secondary column?				
5. For multi-peak analytes - does the pattern of the analyte in the sample match the pattern of the standard?				
6. Are surrogate recoveries within QC limits? (one surrogate both columns)				
E. QC Samples				
1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than the DLs?				
2. Is the Blank Spike and its percent recovery within QC limits?				
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and is the percent recovery/RPD within QC limits?				
F. Others				
1. Are all nonconformances included and noted?				
2. Did analyst initial/date the appropriate printouts and report sheets?				
3. Are all manual integrations checked by a second reviewer to verify they were performed correctly?				
4. Data uploaded to LIMS correctly with associated primary analyst correct?				
5. Samples _____ showing full re-calculation from raw data to final concentration in LIMS.				
6. Reagents/Standards verified accurate on bench sheets and in LIMS.				

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 4 – Standard concentrations/sources
NOTE: All standards are fully documented within the LIMS

	Level 1 (ppb)	Level 2 (ppb)	Level 3 (ppb)	Level 4 (ppb)	Level 5 (ppb)	Level 6 (ppb) MIDPOINT	Level 7 (ppb)	Primary Source (Concentration-ppm)	Secondary Source** (Concentration-ppm)
Single Component Pesticides	1	5	10	25	50	100	200	Restek (200)	Accustandard (1000)
DCB/TCMX	1	5	10	25	50	100	200	Restek (200)	NA
Chlordane (n.o.s.)	-	5	10	20	50	100	200	Restek (1000)	Ultra Scientific (5000)
Toxaphene	-	100	250	500	750	1000	2000	Restek (1000)	Accustandard (100)
PCB-1016/PCB-1260	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (1000)
PCB-1221*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1242*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1248*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1254*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1262*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (100)
PCB-1268*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (500)

* - PCB calibration 1016/1260 unless other pattern detected.

** - Secondary Source may be from any vendor other than the primary source.

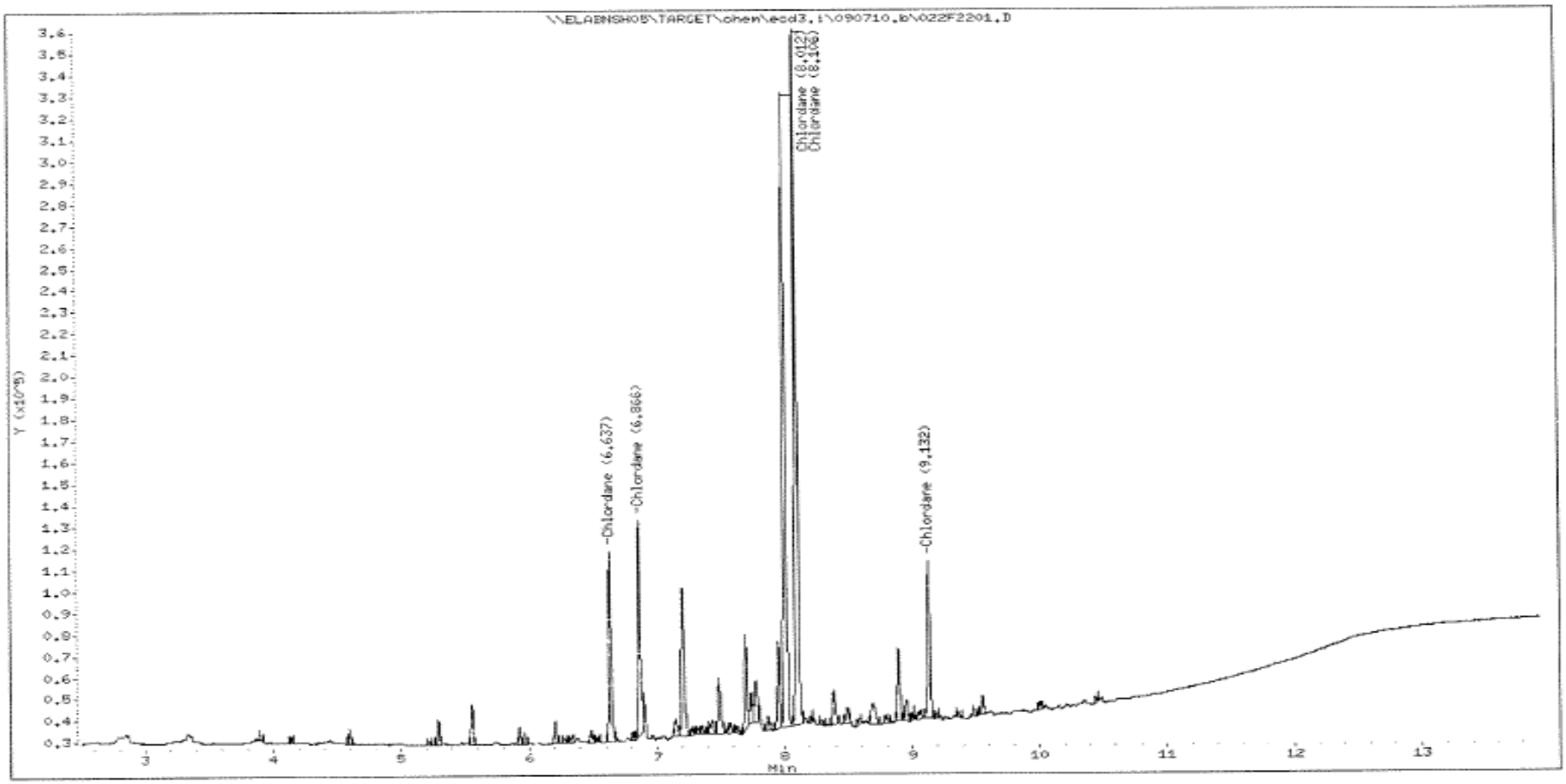
Table 5 – Example Chromatographs

Data File: \\ELABNSH05\TARGET\chem\ecd3.i\090710.b\022F2201.D
Date : 07-SEP-2010 22:08
Client ID:
Sample Info: SEQ-CCV2

Column phase: ZB MR-1

Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32



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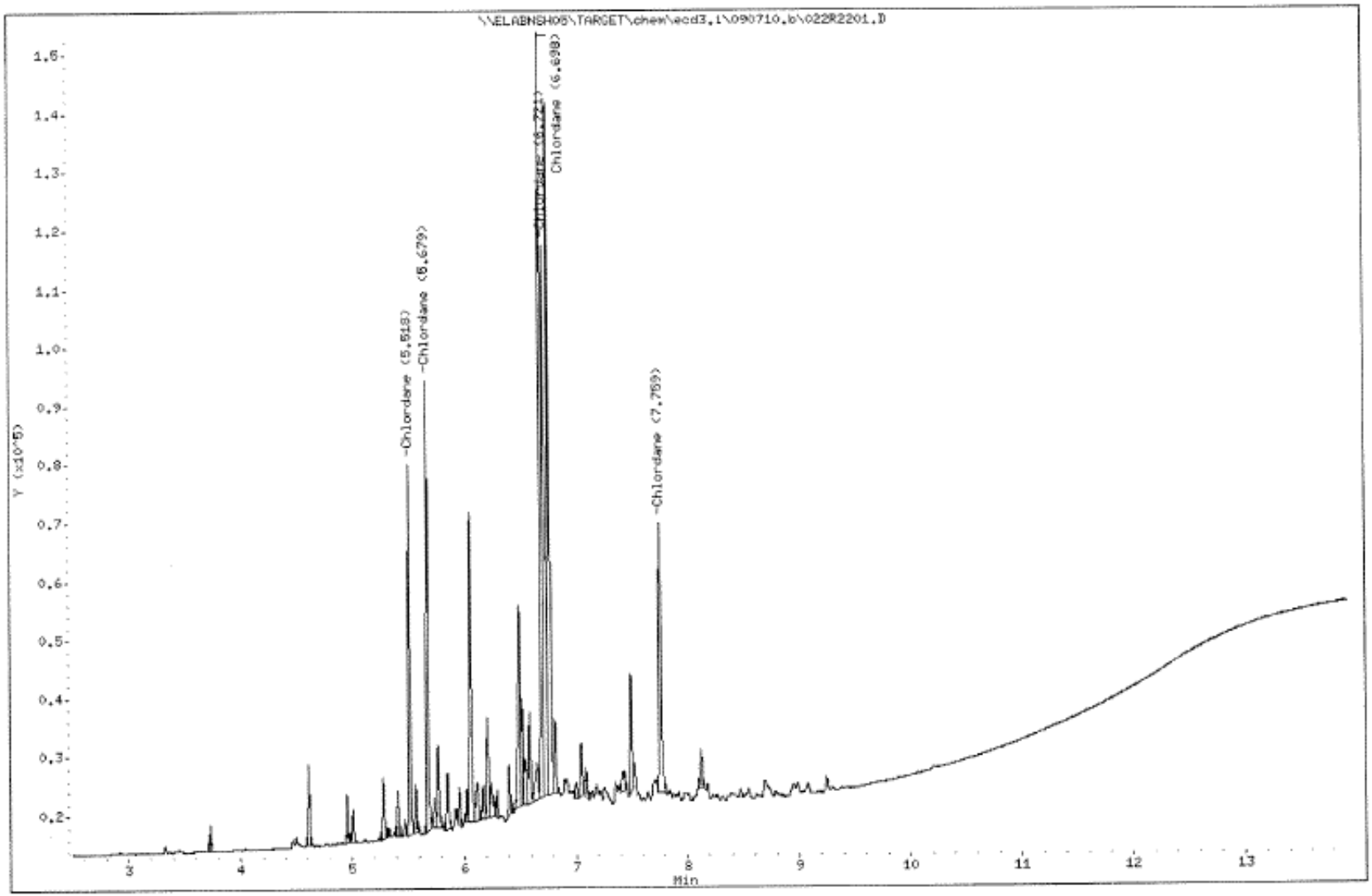
Date : 07-SEP-2010 22:08
Client ID:
Sample Info: SEQ-CCV2

Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32

Column phase: ZB MR-2

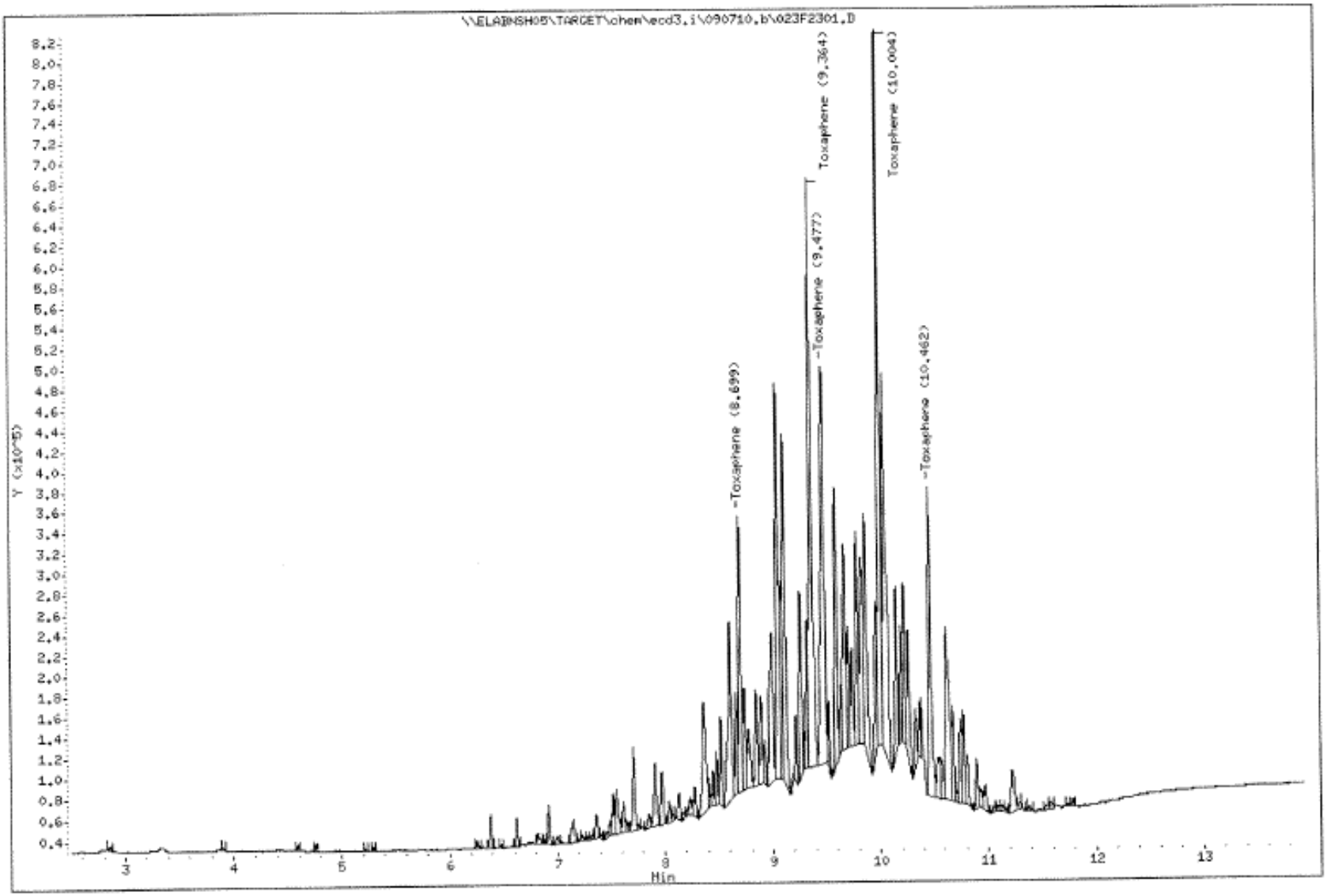


Data File: \\ELABNSH05\TARGET\chem\ecd3.i\090710.b\023F2301.D
Date : 07-SEP-2010 22:26
Client ID:
Sample Info: SEQ-CCV3

Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32

Column phase: ZB HR-1



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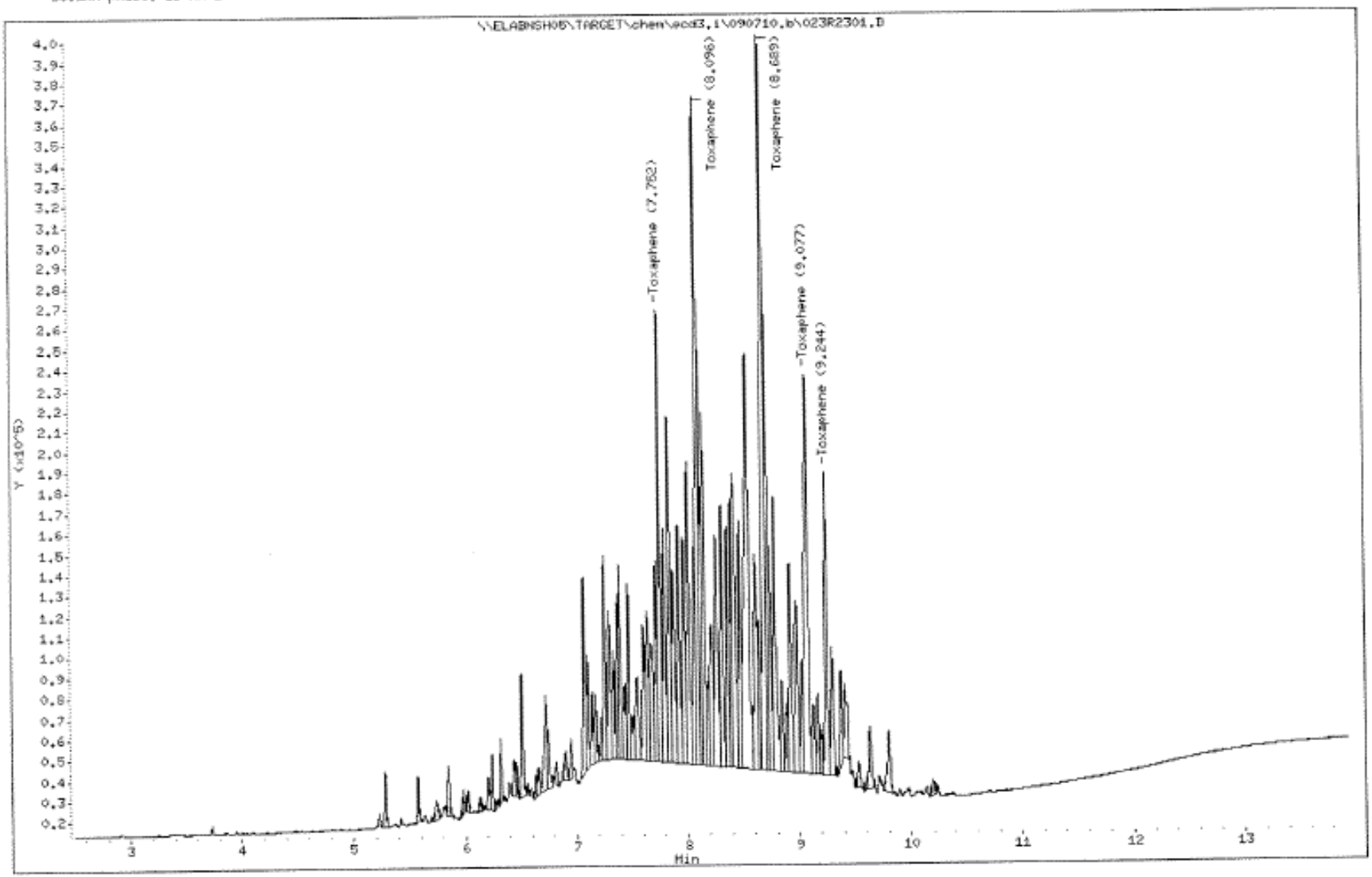
Date : 07-SEP-2010 22:26
Client ID:
Sample Info: SEQ-CCV3

Column phase: ZB HR-2

Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32

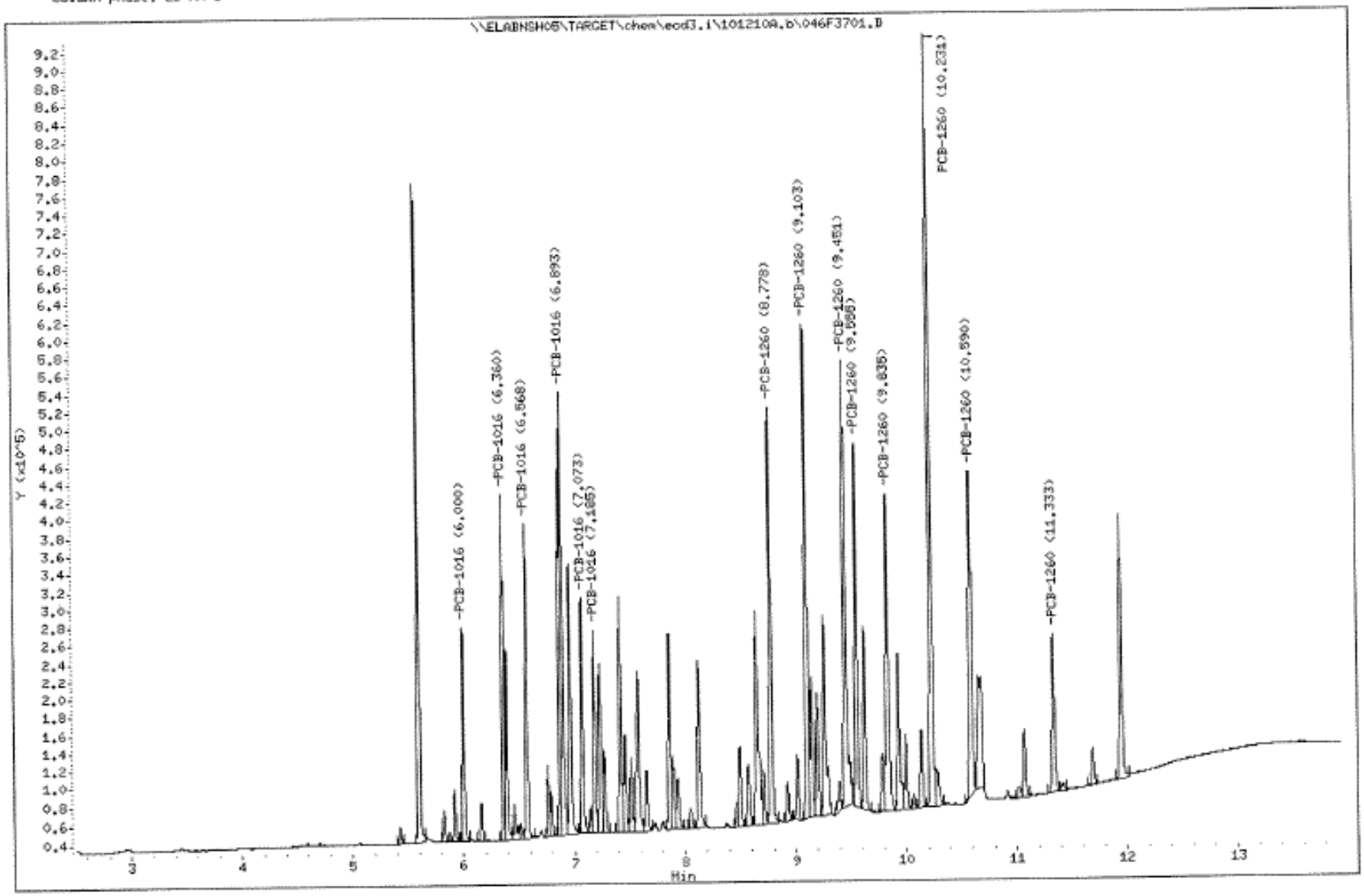


Data File: \\ELABNSH05\TARGET\chem\ecd3.i\101210A.b\046F3701.D
Date : 13-OCT-2010 14:29
Client ID:
Sample Info: SEQ-CAL2

Column phase: ZB MR-1

Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32



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Instrument: ecd3.i

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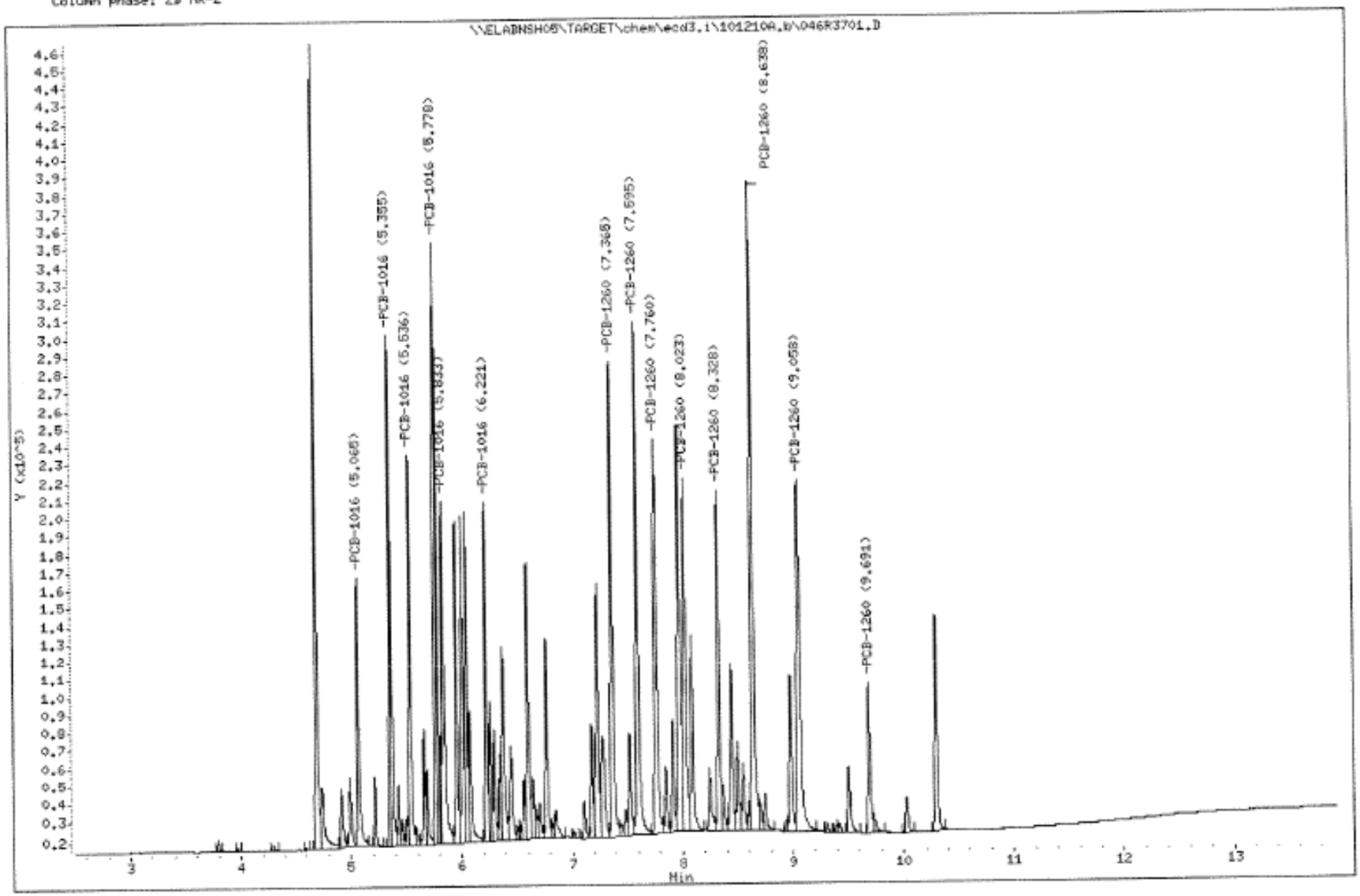
Column phase: ZB MR-2



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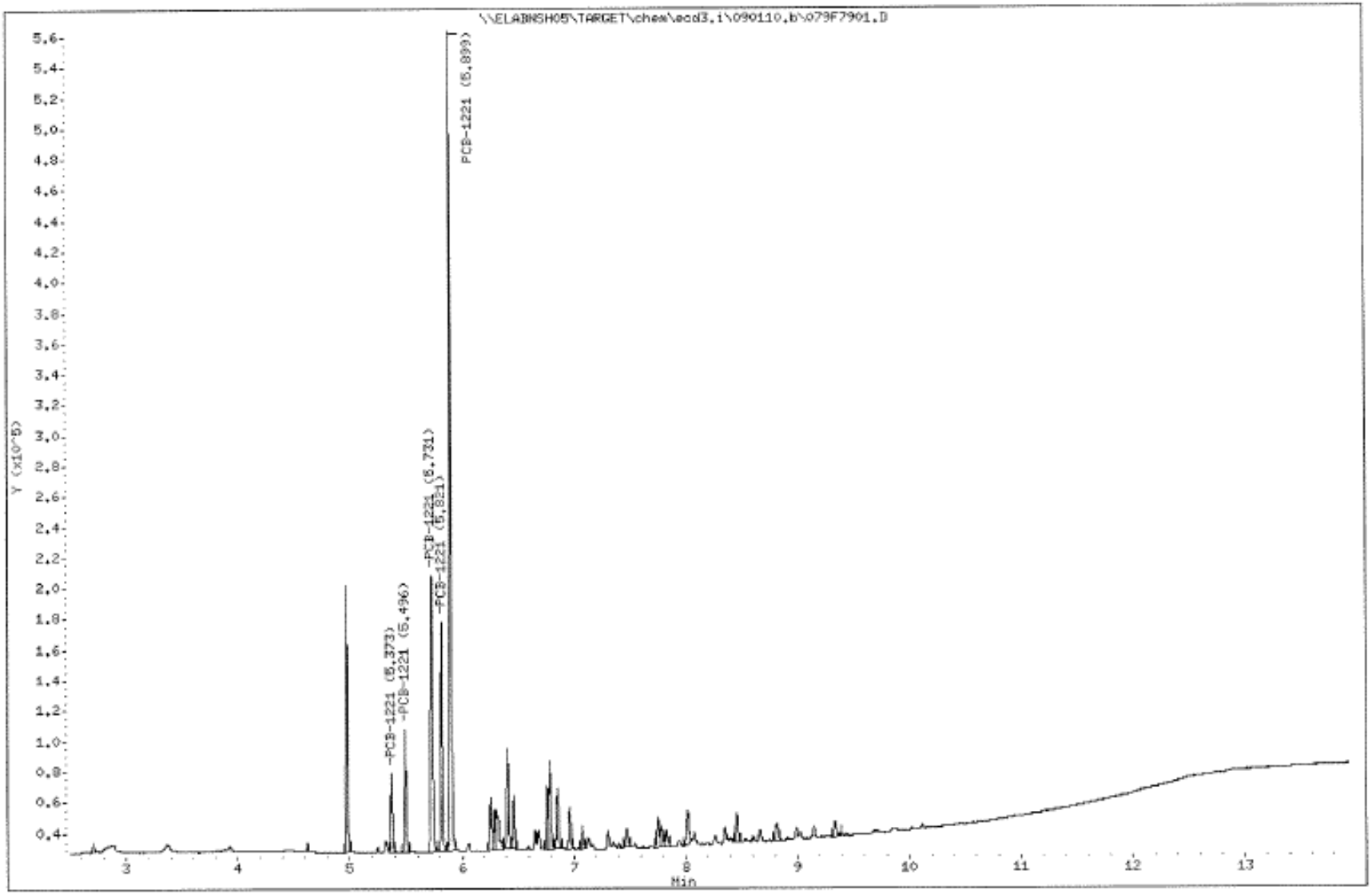

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Column phase: ZB MR-2

Instrument: ecd3.i

Operator: WAL
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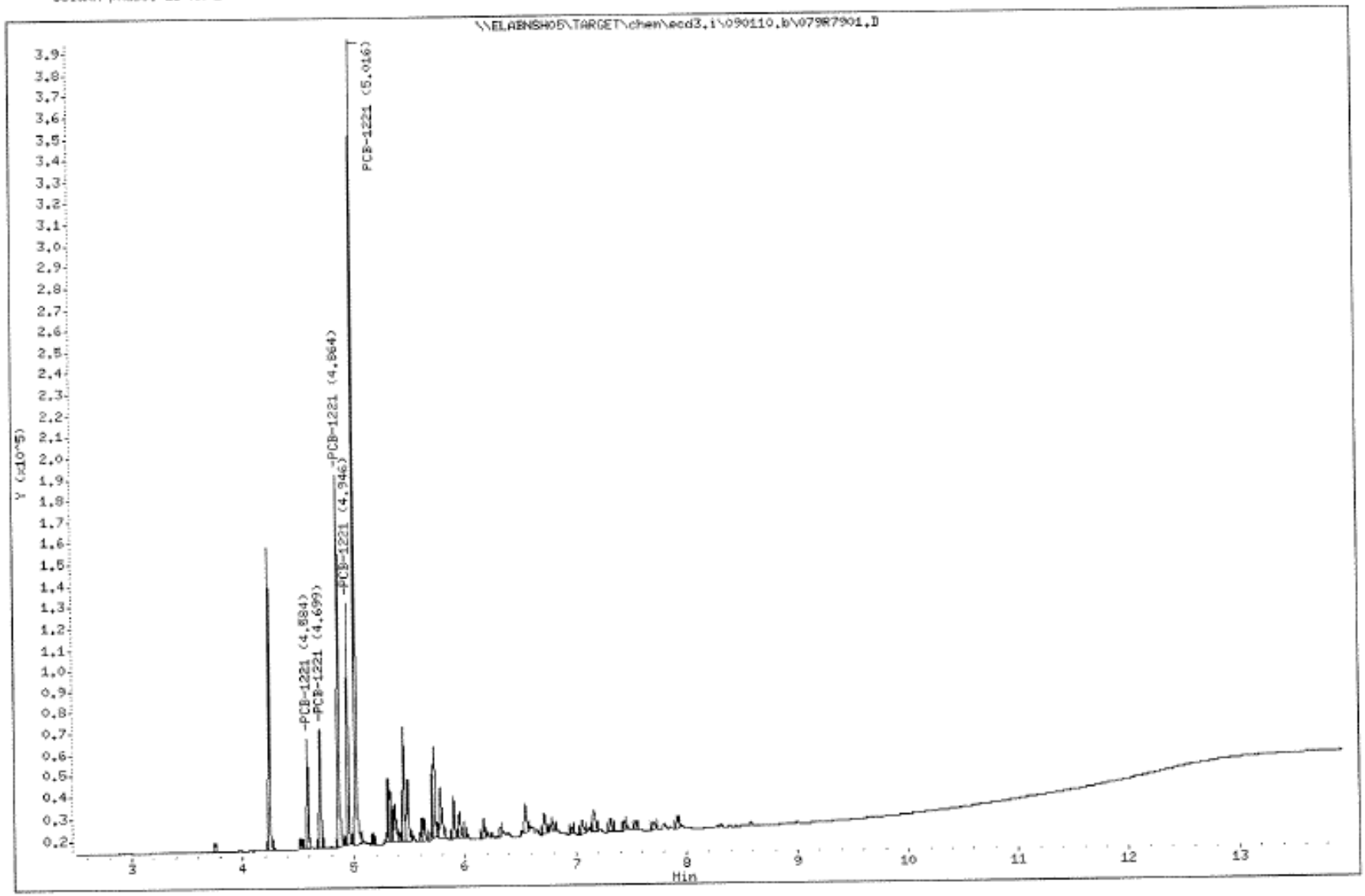

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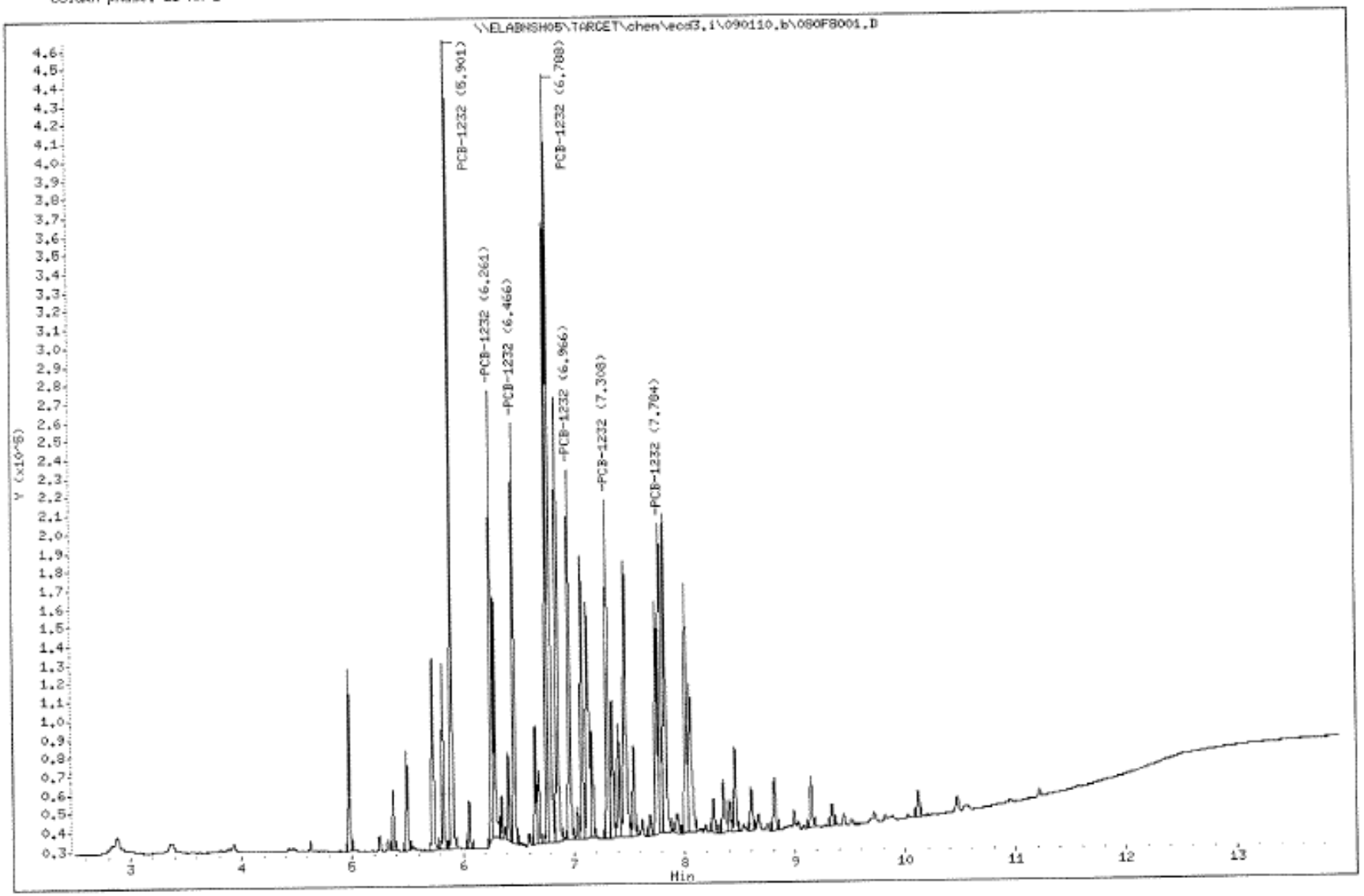
Sample Info: AR-1232

Column phase: ZB MR-1

Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32



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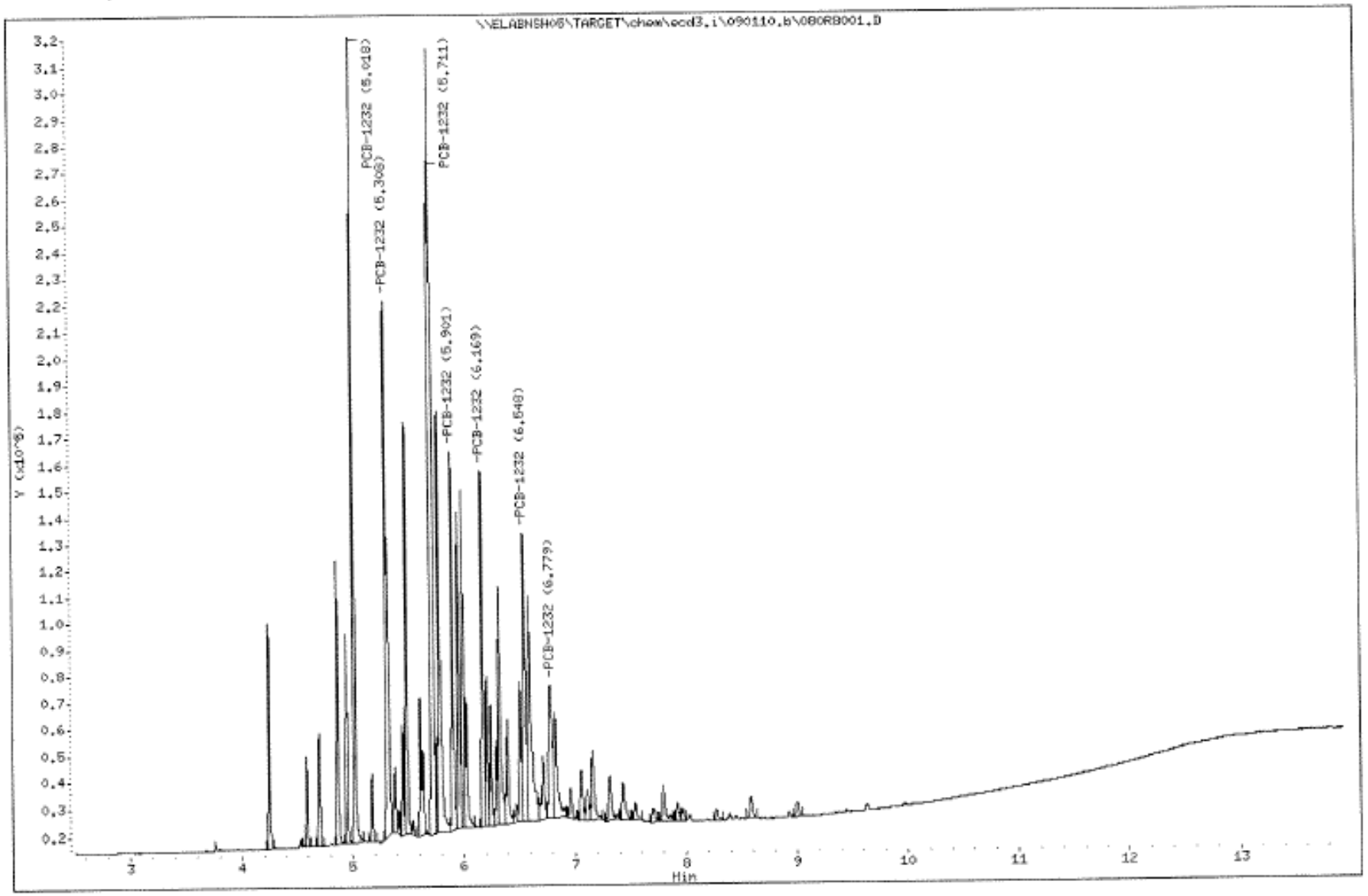
Sample Info: AR-1232

Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32

Column phase: ZB MR-2



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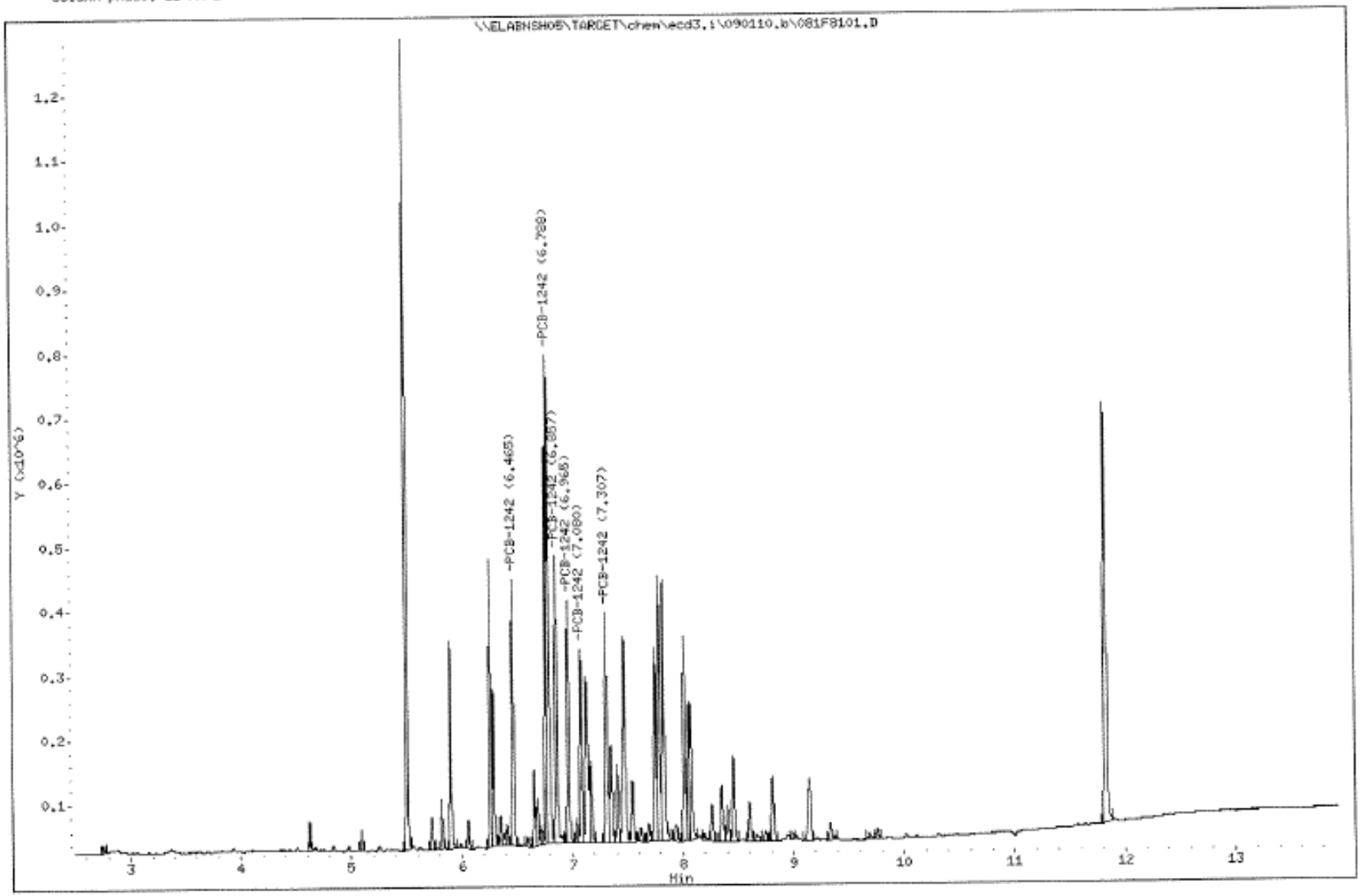
Column phase: ZB MR-1

Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32



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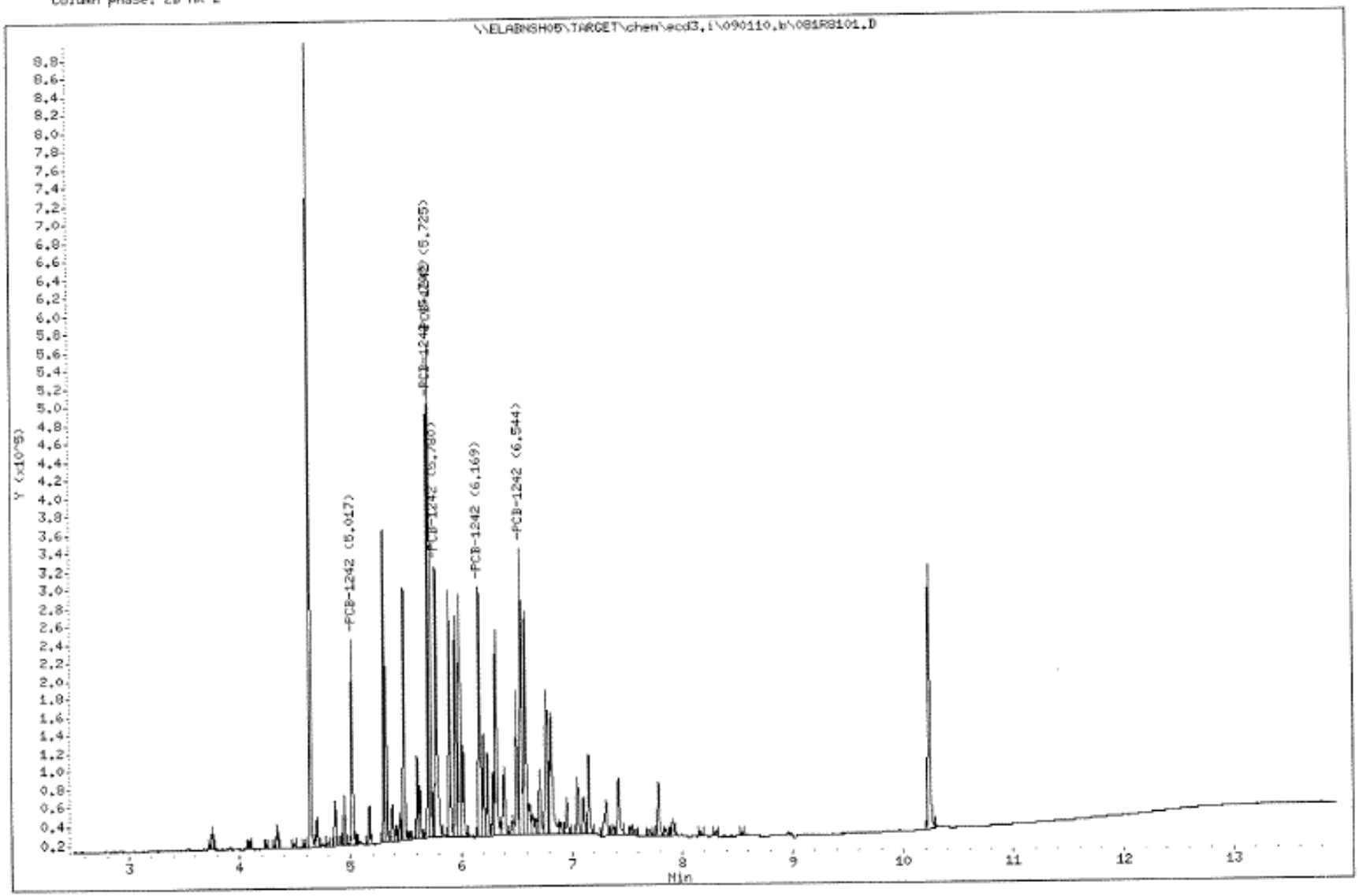

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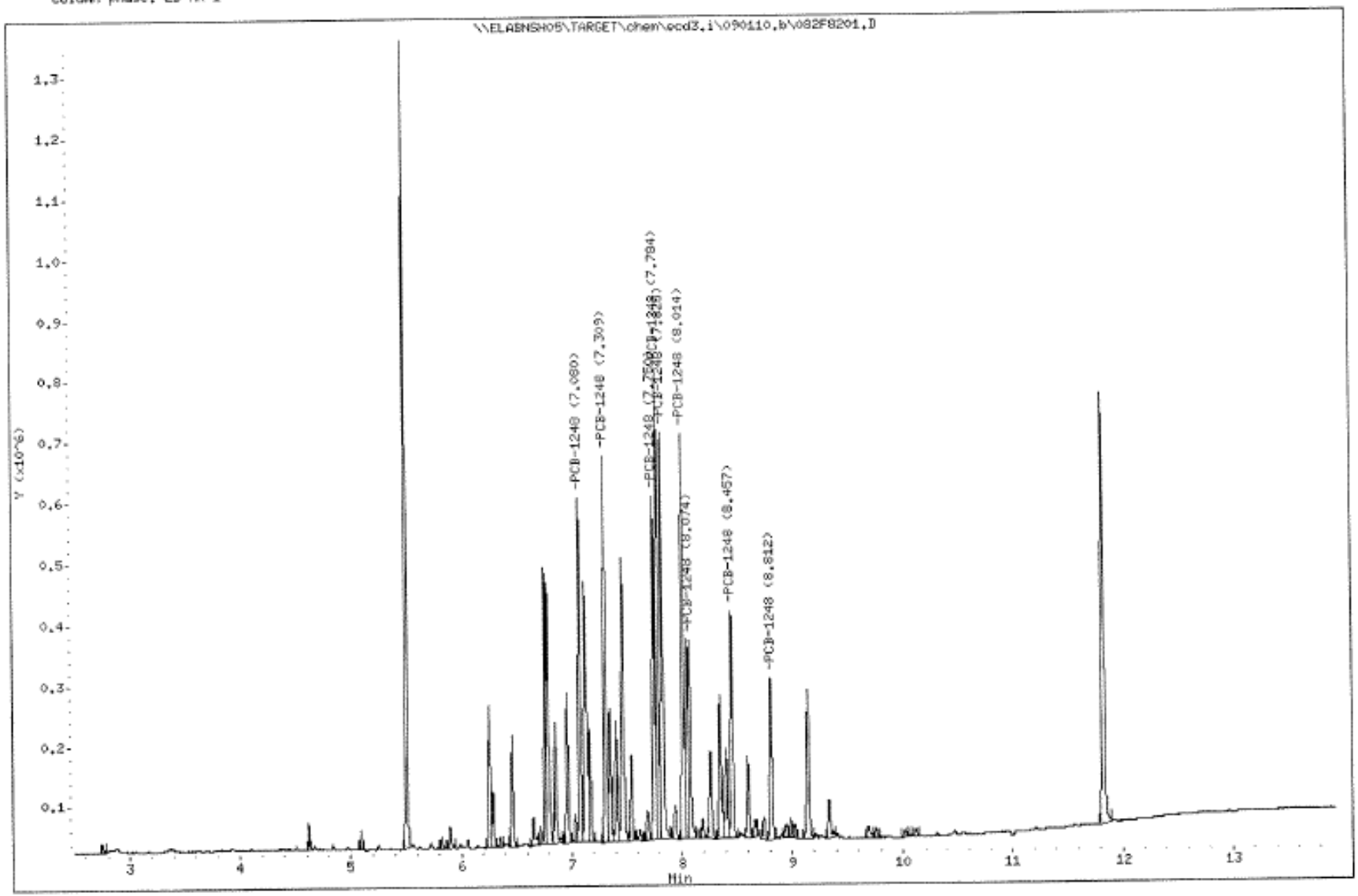
Column phase: ZB HR-1

Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32



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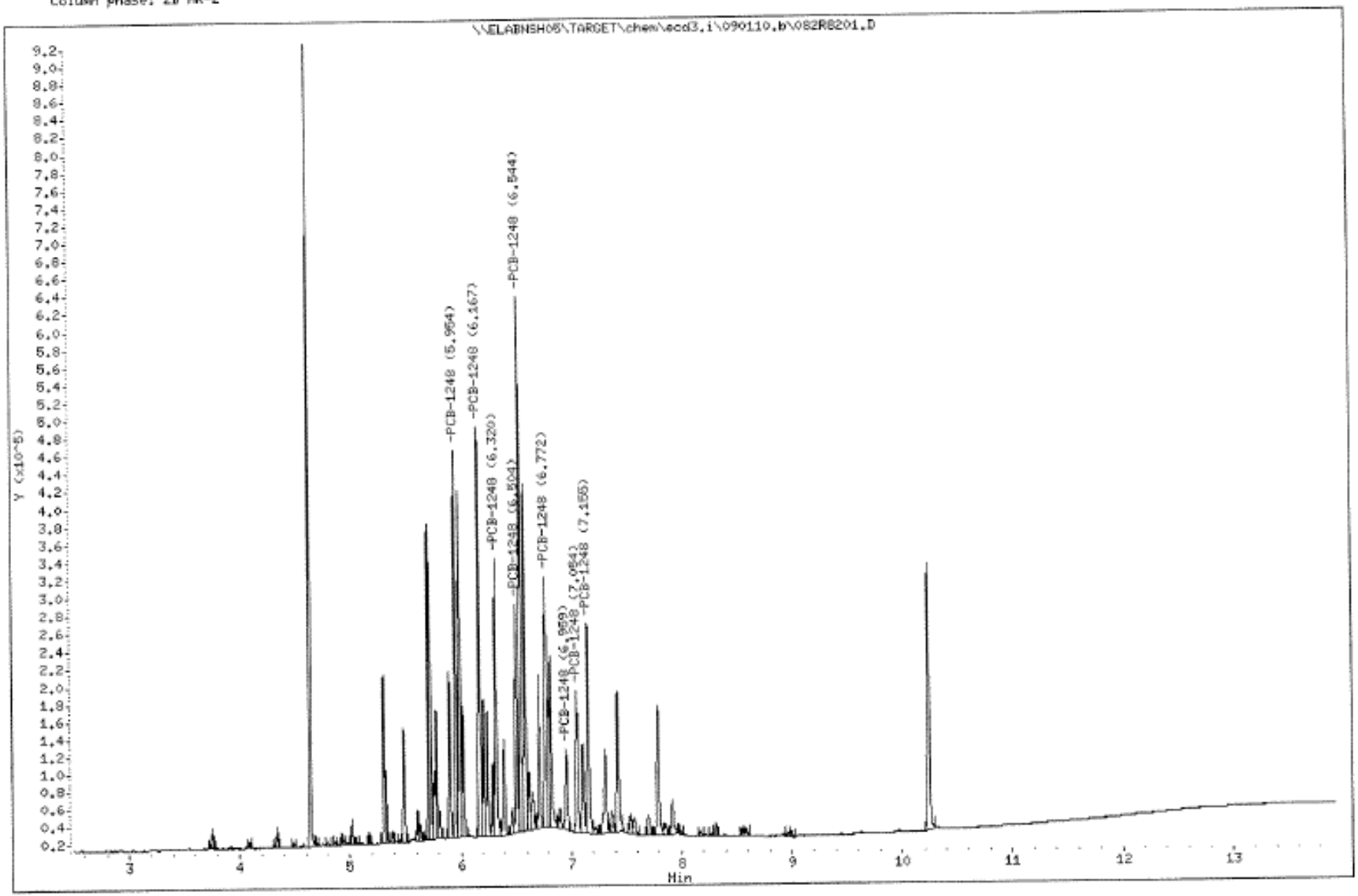
Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32

Column phase: ZB MR-2



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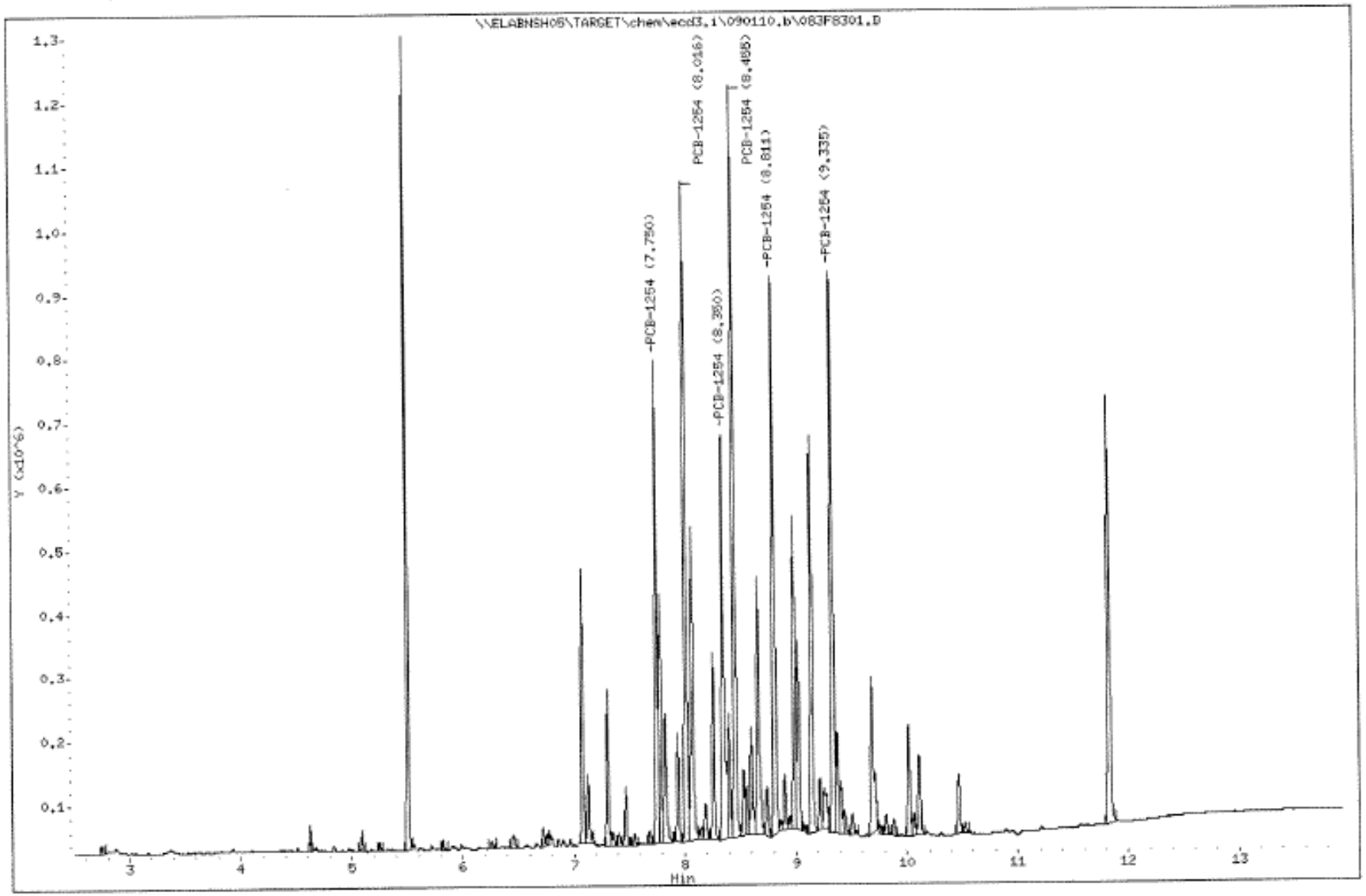
Sample Info: AR-1254

Column phase: ZB MR-1

Instrument: ecd3.i

Operator: WAL
Column diameter: 0.32



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Instrument: ecd3.i

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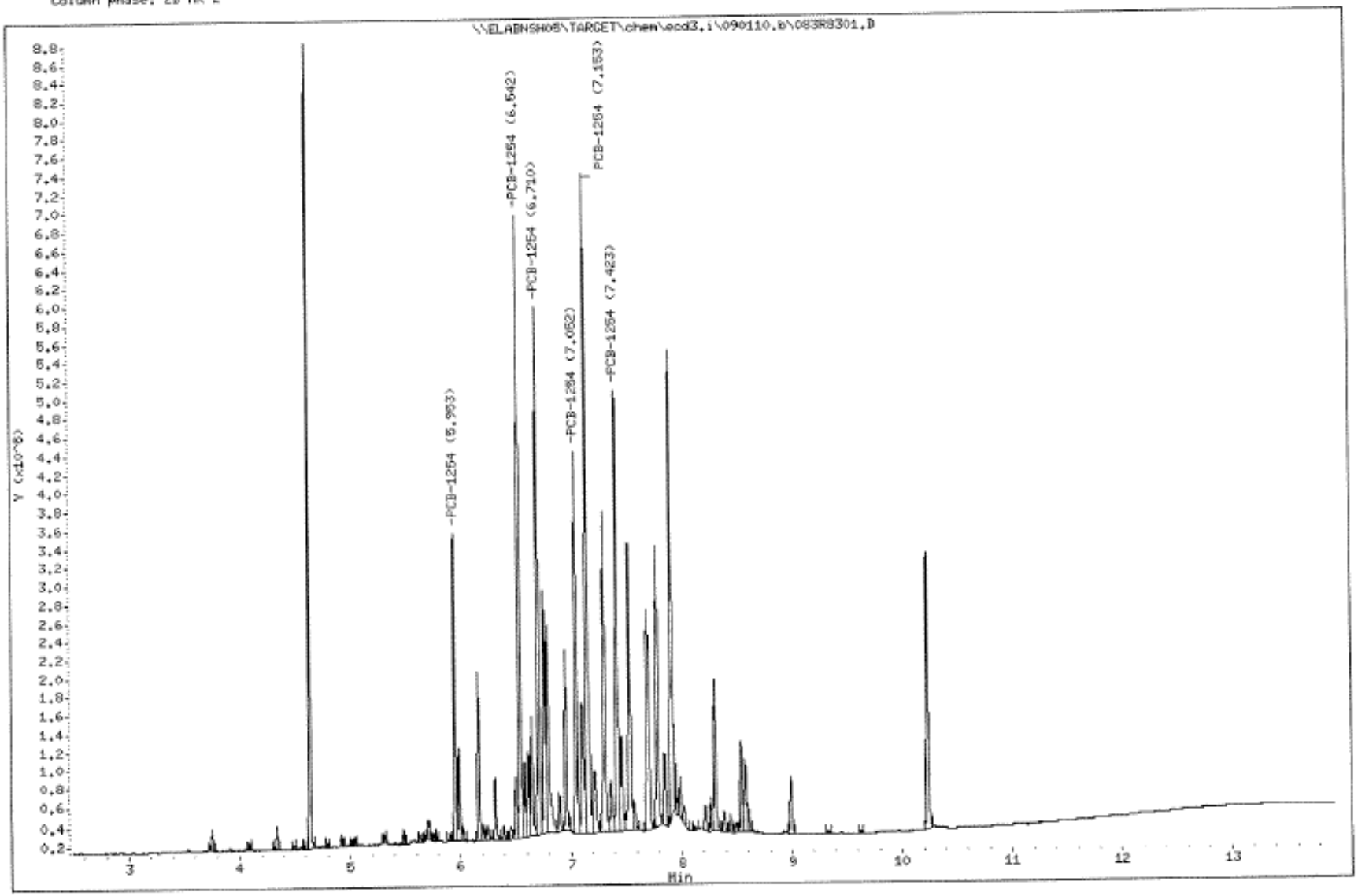
Column diameter: 0.32



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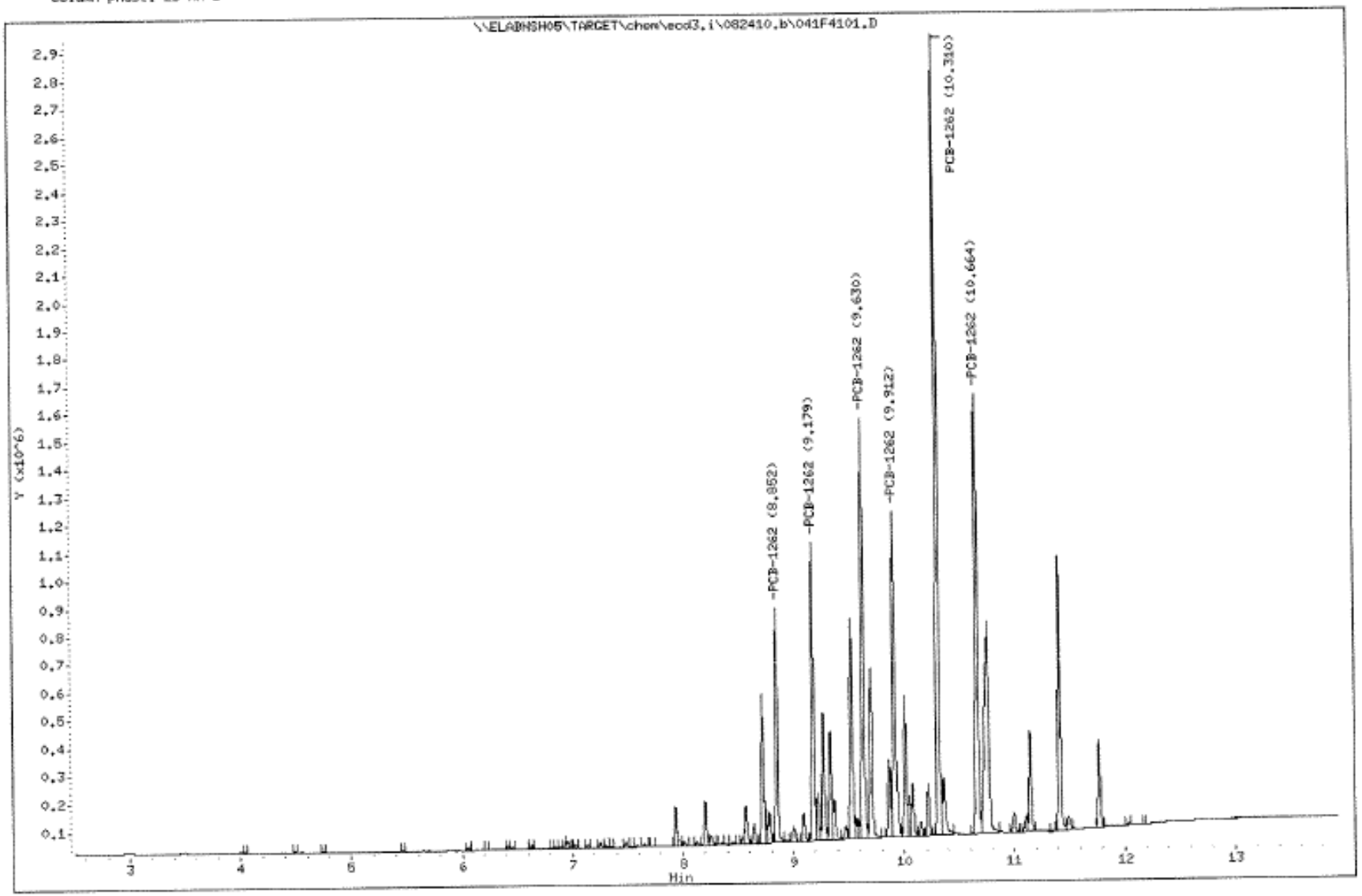
Column phase: ZB MR-1



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Instrument: ecd3.i

Operator: WAL
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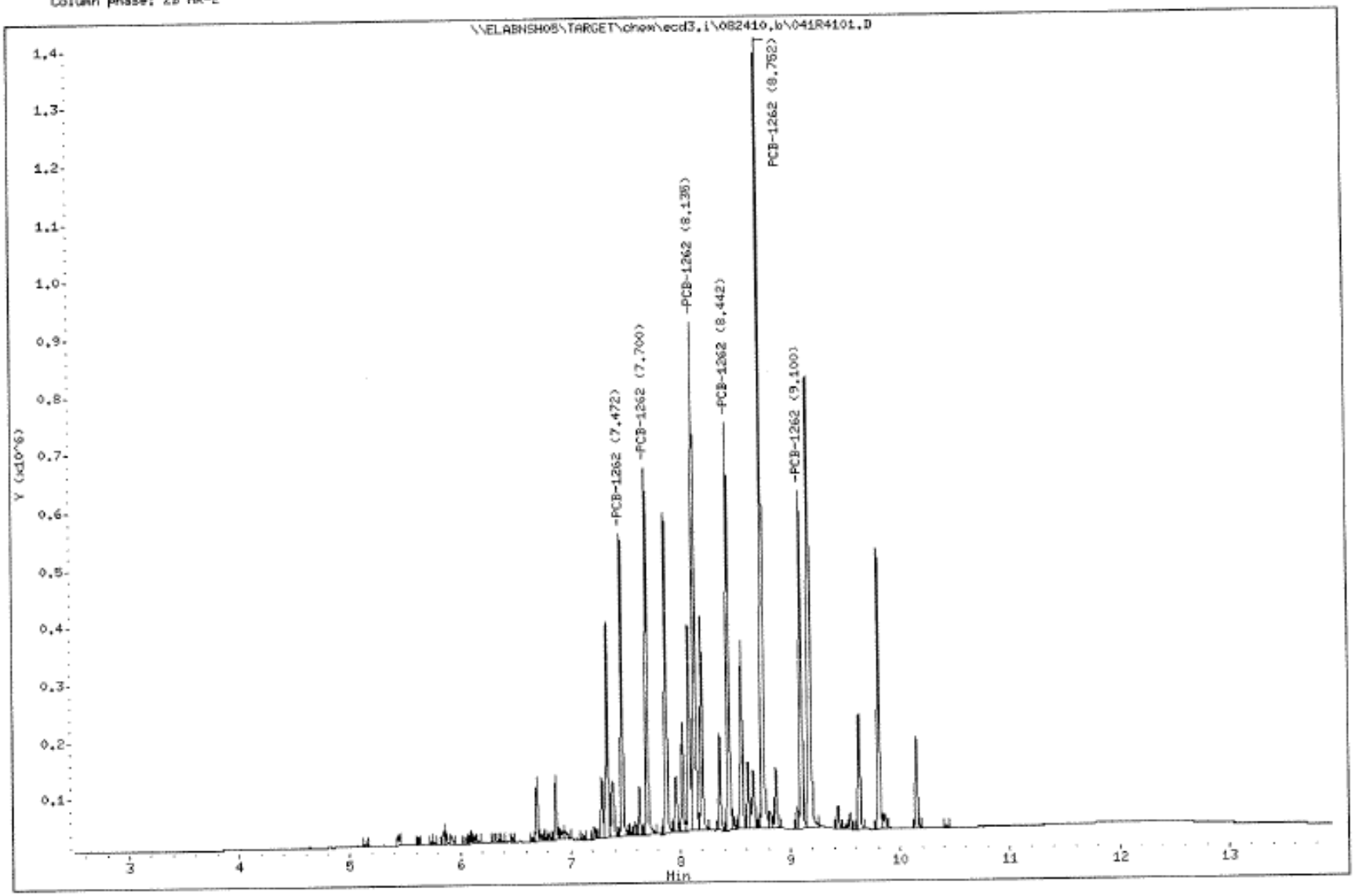
Column phase: ZB MR-2



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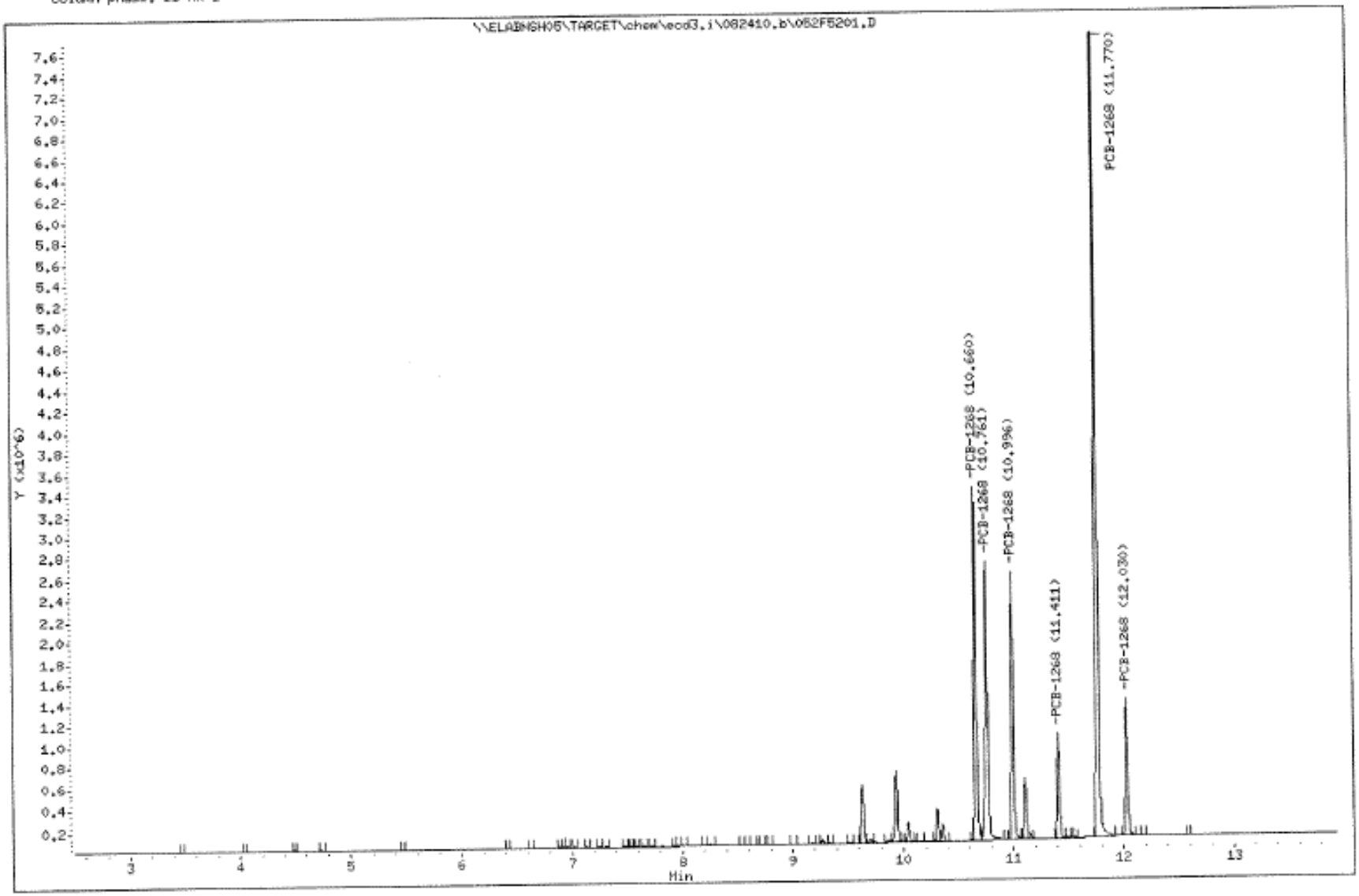
Column phase: ZB MR-1



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Page 1

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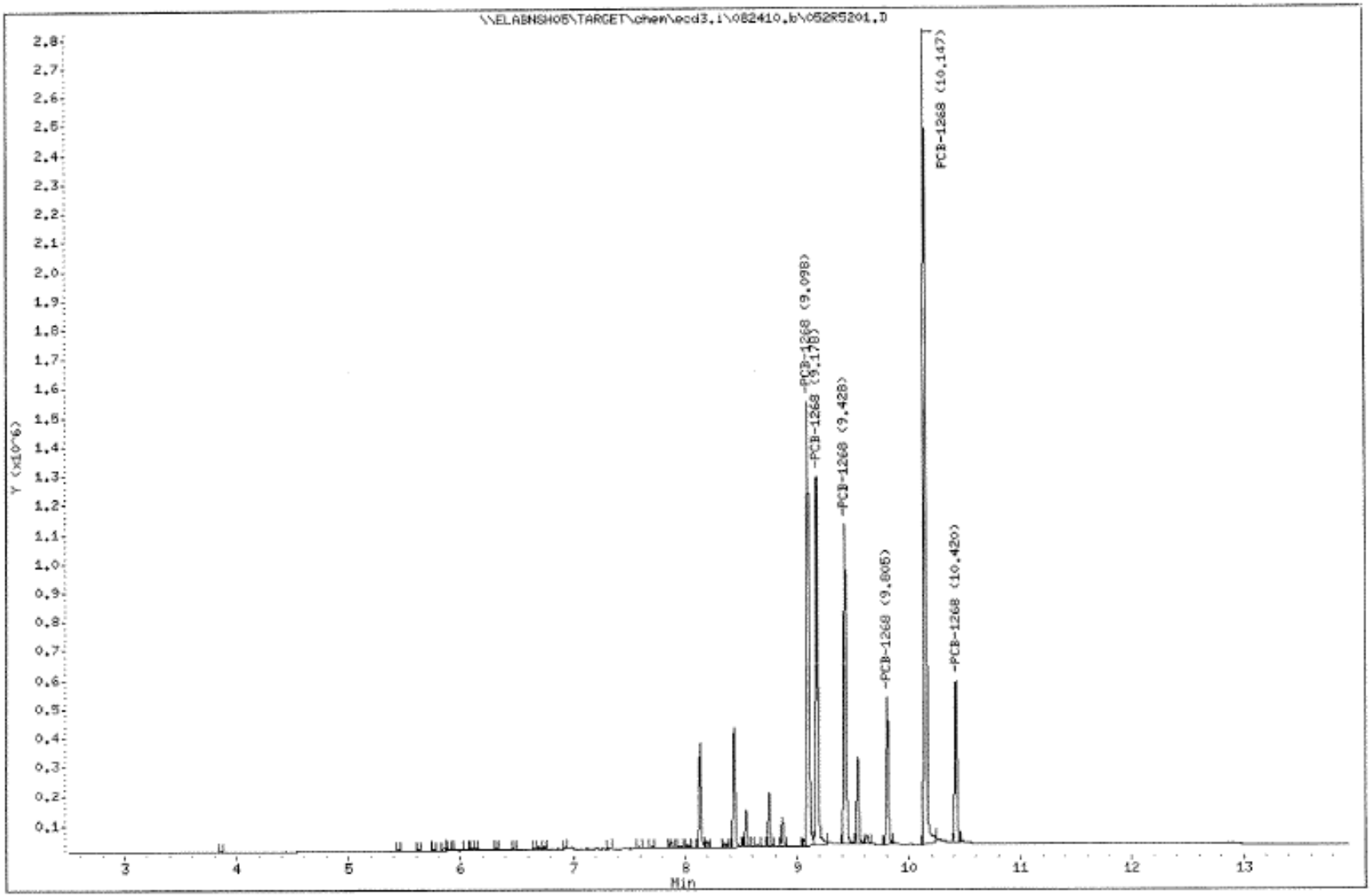
Instrument: ecd3.i

Sample Info: SEQ-CALE

Operator: WAL

Column phase: ZB HR-2

Column diameter: 0.32



EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP221 REVISION #: 18 EFFECTIVE DATE: 20151221

TOTAL ORGANIC CARBON (TOC)
Also DISSOLVED ORGANIC CARBON (DOC)
By SM 5310 C-2011, SW846 METHOD 9060/9060A or
Lloyd Kahn Method "Determination of TOC in Sediment"

APPROVALS:

Lab Director: _____ Date: **20151221**

Data Quality Manager: _____ Date: **20151221**

Group Leader: _____ Date: **20151221**
Betty Quillen

Changes Summary

SOP221_R18_20151221_TOC

- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.

SOP221_R17_20150922_TOC

- All references to supervisor updated to reflect group leader.
- Section 10 updated to reference QS04 and to add Stirbar/stirplate to 10.7.
- Section 15.2 updated for worksheet location
- Section 22 updated with troubleshooting tips.
- Updated soil procedure – review carefully.
- Date formats updated to YYYYMMDD.

Revision 16, 07/31/2014

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark updated to include proprietary reference.
- Added DOC analysis references.
- MSDS sheet reference updated to reflect vendor website access.
- Section 17.2 added.
- Sections 22-26 populated/updated

Revision 15, 10/11/2013

- Added water system software TOC-Vws/TOC-Control V version 1.07.00.00sp1 and soil system software WinTOC3.0.

Revision 14, 08/13/2013

- References to quarterly calibration removed – no method criteria identified.

Revision 13, 07/15/2013

- Procedure updated to remove single analysis option on 9060/9060A method water analysis.

Revision 12, 07/01/2013

- Standard method reference updated to reflect "-2011" and SOP verified to include necessary QC.
- Added demonstration of capability requirement to section 12.
- Clarified 5310C limits for CCVs.
- Underlined section 8.7 indication that strict 9060 samples are analyzed in quadruplicate.

Revision 11, 09/12/2012

- Standard method reference format updated and QS references updated
- QC frequencies and criteria updated for SM 5310 C.
- Duplicate/Quadruplicate analysis requirements clarified.
- 6.3 - standard makeup updated.
- 6.7 - solution stabilization added.
- 8.2 - instrument warmup added.
- 8.3 and 9.6 - sample sequence updated.
- 9.11 - added instructions for printing the run log
- 10.7 - added MRL standard analysis.

- Listing of attachments added as section XVI with data review checklist added as Attachment 2.

Revision 10, 08/22/2011

- Section 8.9 added "When verifying the sample calculations, note the instrument manual (p46) indicates **"The calibration curve is shifted through the origin to correct for the TC contained in purified water used for preparation of the standard solutions."** Therefore, the sample concentration is calculated by dividing the instrument response by the curve slope and ignoring the intercept."

Revision 09, 07/12/10

- The SOP is an update from Revision 08 dated 04/28/09
- The SOP has been reviewed and confirmed to be accurate.
- The soil TOC method calibration concentrations were updated.
- The LCS reference was changed to BS.

Table of Contents

TOTAL ORGANIC CARBON (TOC)
Also DISSOLVED ORGANIC CARBON (DOC)
BY SM 5310 C-2011, SW846 METHOD 99060A or
Lloyd Kahn Method "Determination of TOC in Sediment"

1. Identification of the Test Method
SM 5310C, SW-846 9060/A or Lloyd Kahn

2. Applicable Matrix or matrices
SM5310C is used to determine the concentration of organic carbon in source and drinking water, SW846 Method 9060/9060A is used to determine concentrations of carbon in saline waters, domestic and industrial wastes and can be modified for soil determination. The L1oyd Kahn Method is used for determination of TOC in solid/sediment matrices. Can also be used to determine dissolved organic carbon in field or lab filtered samples.

3. Limits of Detection and Quantitation

S/W	Analyte	LOQ	LOD	DL	Units
Water	Total Organic Carbon/Dissolved Organic Carbon	3.0	2.50	1.25	mg/L
Solid	Total Organic Carbon/Dissolved Organic Carbon	800	400	200	mg/Kg

4. Scope and Application, Including Parameters to be Analyzed
SM5310C is used to determine the concentration of organic carbon in source and drinking water, SW846 Method 9060/9060A is used to determine concentrations of carbon in saline waters, domestic and industrial wastes and can be modified for soil determination. The L1oyd Kahn Method is used for determination of TOC in solid matrices. These methods should be read over carefully by the analyst and any restrictions should be noted.

5. Summary of the Test Method
The organic carbon is measured using a Shimadzu Total Organic Carbon Analyzer (aqueous samples) and an OI Analytical Solids TOC Analyzer model 1010 (soil/sediment samples). The Shimadzu instrument converts the organic carbon in a sample using wet chemical oxidation. The CO2 formed is then measured by an infrared detector (replaces ultraviolet detector in SM 5310C). With the model 1010 Solids TOC analyzer, TOC is determined by acidifying a sample and heating it to 250°C to remove the TIC. The sample is then heated to 900°C to combust the remaining TOC. The resulting carbon dioxide from the TOC is detected by a non-disperse infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. This mass is proportional to the mass of TOC in the sample.

6. Definitions
6.1. Laboratory Quality System QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2. ANALYTICAL BATCH-The set of samples extracted /distilled/ or digested at the same time to a maximum of 20 samples.

6.3. CALIBRATION BLANK (CB)- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

6.4. CALIBRATION STANDARD (CAL)- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.5. FIELD BLANK (FB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, including exposure to a sample bottle holding time, preservatives, and all preanalysis treatments. The purpose is to determine if the field or sample transporting procedures and environments have contaminated the sample.

6.6. FIELD DUPLICATE (FD)- Two samples taken at the same time and place under identical circumstances which are treated identically throughout field and laboratory procedures. Analysis of field duplicates indicates the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.

6.7. LABORATORY BLANK (BLK)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

6.8. LABORATORY CONTROL SAMPLE (BS)- A solution prepared in the laboratory by dissolving a known amount of one or more pure compounds in a known amount of reagent water. Its purpose is to assure that the results produced by the laboratory remain within the acceptable limits for precision and accuracy. (This should not be confused with a calibrating standard, it must be prepared from a source other than the same source as the calibration standards).

6.9. LABORATORY DUPLICATE (DUP)- Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures.

6.10. QUALITY CONTROL CHECK SAMPLE, also known as INITIAL CALIBRATION VERIFICATION (ICV)- A sample containing analytes of interest at known concentrations (true value) of analytes. The ICV is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.

6.11. MINIMUM REPORTING LIMT (MRL) – the lowest standard concentration to be reported. Supported by the daily analysis of a standard at the specified concentration.

6.12. METHOD DETECTION LIMIT (MDL)- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero. Determined annually.

7. Interference

7.1. Water method

7.1.1. Removal or carbonate and bicarbonate carbon by acidification and purging with purified gas results in the loss of volatile organic substances. The volatiles also can be lost during sample blending, particularly if the temperature is allowed to rise. Another important loss can occur if large carbon-containing particles fail to enter the needle used for injection. Filtration although necessary to eliminate particulate organic matter when only DOC is to be determined, can result in loss or gain of DOC, depending on the physical properties of the carbon-containing compounds and the adsorption of carbonaceous material on the filter, or its desorption from it. Check filters for their contribution to DOC by analyzing a filtered blank. Note that any contact with organic material may contaminate a sample. Avoid contaminated glassware, plastic containers, and rubber tubing. Analyze treatment, system, and reagent blanks.

7.1.2. This procedure is applicable only to homogenous samples which can be injected into the apparatus reproducibly by means of a pipette. The openings of the pipette limit the maximum size of particles which may be included in the sample.

7.2. Soil Method

7.2.1. All materials must be routinely demonstrated to be interference –free under the analysis conditions by running blanks. Use high purity or purified reagents and gases to help minimize interference problems.

7.2.2. The infrared detector is sensitized to CO_2 and accomplishes virtually complete rejection of response from other gases that absorb energy in the infrared region.

8. Safety

8.1. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be follow lab wide.

8.2. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.

8.3. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.4. MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your group leader, lab director or data quality manager if there are any difficulties in accessing these records..

9. Equipment and Supplies

9.1. Equipment:

9.1.1 Shimadzu Total Organic Analyzer TOC-VWS

9.1.2 Shimadzu ASI-V Autosampler for Aqueous TOC determination holding 68 samples.

9.1.3 OIC Analytical Solids TOC Analyzer, Model 1010 (soil/sediment samples)

9.2 Supplies:

9.2.1 40 mL Glass Vials. Also, 100 mL and 200 mL volumetric flasks.

9.2.2 Quartz sample cups (soil/sediment samples)

9.2.3 50 µL syringe

10. Reagents and Standards

The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method. All reagents shall be made from ACS reagent grade chemicals. All reagents used for distillation and analysis are entered into Element. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the samples they were used with.

10.1. The laboratory reagent blank water used for TOC analysis is obtained from the Modulab Analytical water purification system in the analytical laboratory. **Boiling the water is not performed as the method requests. This has not presented a problem.**

10.2. Potassium hydrogen phthalate, primary stock solution, 1000 mg/L: Dissolve 2.165g of potassium hydrogen phthalate (primary standard grade) in 1000 mLs water preserved with 2 mLs of concentrated H2S04.

10.3. Potassium hydrogen phthalate, standard solutions : A 25 mg/L standard is prepared by diluting 5 mLs of the 1000 mg/L Primary stock solution to 200 mLs. Also, a 50 mg/L std. is prepared by diluting 10 mLs of the 1000 mg/L Primary stock solution to 200 mLs. These two standards are analyzed alternately during the analytical run.

10.4. The carbonate-bicarbonate solutions are not needed for this instrument.

10.5. Calibration Standards:

10.5.1. For the water method, calibration standard is Potassium Hydrogen Phthalate. Standards are made from dilutions of the stock 1000 mg/L standard as follows:
1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL
25.0 mg/L = 5.0 mL of 1000 mg/L -> 200 mL
50.0 mg/L = 10.0 mL of 1000 mg/L -> 200 mL
100 mg/L = 10.0 mL of 1000 mg/L -> 100 mL

10.5.2. A low level standard curve must be run for drinking water samples with the standards made as follows:

0.25 mg/L = 0.025 mL of 1000 mg/L -> 100 mL
0.50 mg/L = 0.050 mL of 1000 mg/L -> 100 mL
1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL
1.5 mg/L = 0.15 mL of 1000 mg/L -> 100 mL
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL

See 14.2.7 for soil curve

10.5.3. The soil method calibration standard is prepared by using an OI commercially prepared 30% carbon sucrose solution or by weighing 7.125 grams of reagent grade sucrose and diluting to 10 L with deionized water.

10.6. Laboratory Control Sample/Blank Spike (BS):
10.6.1. For the water method, the BS is normally made from a secondary lot of KHP. Prepared the same as the primary stock solution Refer to 10.2 for prep. This solution is given a unique identifier.
10.6.2. For the soil method, the BS is made using a second source of the 30% carbon sucrose made in 10.5.3. Use a 50 µL syringe to inject 25 µL of this solution onto the quartz wool in a quartz closed cup.

10.7. Persulfate oxidation solution: This solution is made by dissolving 60g of sodium persulfate in DI water, adding 15 ml of phosphoric acid and diluting to 500 ml. Allow solution to stabilize 24 hours while stirring with stir bar on stir plate. This is used for water analysis.

10.8. Phosphoric acid solution: Dilute 100 mL of concentrated 85% phosphoric acid in 500 mL of water. This is used for water analysis.

10.9. Phosphoric acid solution 5%: Dilute 59 mL of concentrated 85% phosphoric acid in 1000 mL of water. This is used for soil.

11. Sample Collection, Preservation, Shipment, and Storage

11.1. Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2. Sampling and storage in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples. NOTE 1: A brief study performed in the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

11.3. Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. The holding time is 28 days for waters and soils with the exception of the Lloyd Kahn method soils, which requires a 14 day holding time. Also, samples must be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

11.4. When water samples cannot be analyzed immediately, the sample is preserved by acidification to (pH </= 2) with HCl or H_2SO_4. Both water and soil samples are stored at 4°C.

12. Quality Control

12.1. Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2. The ICAL correlation coefficient requirement is less than 0.995 for r (0.990 for r^2 in LIMS).

12.3. Analyze a laboratory control sample (BS) for each batch of samples **(maximum of 20 soil/water samples per batch except 5310C is maximum of 10 water samples per batch).** If the BS does not fall within the control limits of 80 to 120%, corrective action must be taken to find and correct the problem. **(5310C limits are 85%-115%).**

12.4. Run a method blank (BLK) for each batch of samples **(maximum of 20 soil/water samples per batch except 5310C is maximum of 10 water samples per batch).** The BLK should be less than 1/2 the reporting limit or LOQ.

12.5. One matrix spike and matrix spike duplicate must be run per set of 20 samples. For water analysis, a spike and spike duplicate are made by mixing 20 mLs of sample with 0.30 mLs of stock 1000 mg/L standard using a mechanical pipette. The true value is 15 mg/L. The percent recoveries on a MS and a MSD should be within 75 and 125%. Relative percent difference (RPD) on duplicates should be less than 20%. If not, a non-conformance report (NCR) must be generated and approved by your group leader. Note – sample duplicate may be analyzed in place of MSD.

12.6. Analyze an initial calibration verification (ICV) immediately after the calibration curve. Analyze a calibration check verification (CCV) standard at the beginning of the sequence, after every tenth field sample and at the end of the sequence. The percent recoveries should be in the range of 80 to 120%. **(5310C limits are 90%-110%). For QSM projects, two passing CCVs immediately following a failed CCV can be used to report unqualified data.**

12.7. When analyzing water samples, all water blanks before samples and standards must be below the detection limit, otherwise the samples must be rerun.

12.8. Analyze an initial calibration blank (ICB) following the ICV. Analyze a continuing calibration blank (CCB) following each CCV. The ICB and CCB should be less than ± the MDL.

12.9. Analyze a CRL standard at the beginning of each soil sequence – it must recover in the range of 70 to 130%.

12.10. Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data. **Calculate spikes as follows where everything is in concentration.**

$$\%\ \text{Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

. Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

12.11. **For aqueous samples check an acidified 20mg/L inorganic carbon standard quarterly, to assure that purge gas flow is adequate to remove inorganic carbon. The result should be below the reported quantitation limit.**

13. Calibration and Standardization

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

14. Procedure

14.1. Aqueous Sample Procedure

14.1.1. Wearing lab coat, gloves and safety glasses, the standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. Also, remove samples from sample storage signing them out appropriately on the internal chain of custody form. Fresh acid and oxidation solutions should be poured into the appropriate containers on the front of the instrument.

14.1.2. Follow the instructions for operation of the instrument in Chapter 4, section 4.3 of the Shimadzu Model TOC-VWS User Manual. **See Appendix I. for Basic TOC start-up notes for analysis.**

NOTE: Instrument needs to warm up at least 30 minutes before analysis begins.

14.1.3. Following is a list outlining the order in which the samples should be analyzed**.** Each sample VOA vial should be numbered and its identity entered into the TOC schedule. Note: All blanks should be acidified to pH 2 to match the matrix of the samples analyzed. All 5310C samples are to be analyzed in duplicate while all 9060A samples are analyzed in quadruplicate. Analysis may begin with CCV/CCB/(CRL) if an ICAL is not analyzed.

14.1.3.1. 0.0 ppm
14.1.3.2. 1.0 ppm
14.1.3.3. 2.5 ppm
14.1.3.4. 5.0 ppm
14.1.3.5. 10.0 ppm
14.1.3.6. 25.0 ppm
14.1.3.7. 50.0 ppm
14.1.3.8. 100 ppm
14.1.3.9. Blank spike (BS/ICV/CRL)
14.1.3.10. Up to 10 field samples (+ QC)
14.1.3.11. 25 ppm CCV
14.1.3.12. Method and/or calibration blank (if 5310C, every 10 samples)
14.1.3.13. Blank spike (BS/ICV) (if 5310C, every 10 samples)
14.1.3.14. Up to 10 field samples (+ QC)
14.1.3.15. 50 ppm CCV

NOTE – A third analysis is automatically performed if sample results are not within ±10%. <u>Aqueous samples analyzed by 9060/9060A must be analyzed in quadruplicate, as specified by the test code.</u>

14.1.4. Instrument printouts are generated from the software. Normal procedure is followed for preparing reports and the data is second checked before being given to the group leader.

14.1.5. When verifying the sample calculations, note the instrument manual (p46) indicates **"The calibration curve is shifted through the origin to correct for the TC contained in purified water used for preparation of the standard solutions."** Therefore, the sample concentration is calculated by dividing the instrument response by the curve slope and ignoring the intercept.

14.2. **Soil / Sediment Sample Procedure**

14.2.1. A sample is introduced into the Solid Module via a conditioned sample cup. Once the sample has been introduced the entire analysis sequence is automatic. Please reference Chapter 4 of the OI 1010 Solid Module instrument manual for instrument states and configuration when initially setting the instrument methods up.

14.2.2. TC Mode Instrument Settings:

14.2.2.1. Analysis Temp: 900°C

14.2.2.2. Analysis Time: 6.5 minutes

14.2.2.3. Nitrogen Gas Flow: 60-100 psi (external regulator)

14.2.2.4. Oxygen Gas Flow: 40-60 psi (external regulator)

14.2.3. **Following is a step by step description of a routine soil TOC analysis**: The standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. The nitrogen and oxygen (internal regulator should be set at 50-60 psi) turned on allowing a nitrogen flow of 350-400mL/minute and an oxygen flow of 180 mL/minute (± 3 mL/minute).

NOTE: DO <u>NOT</u> TURN THE ANALYZER ON BEFORE TURNING THE GAS ON!

14.2.4. Let the gas flow through the instrument for a few minutes. The instrument should now be turned on and allowed to stabilize for 30 minutes.

14.2.5. Condition the cups (with quartz wool in them) using Diagnostics under Instrument Menu commands, (don't condition too many cups at a time since sitting in contact with the air can cause contamination).

14.2.6. Set up the subdirectory (using the current date to ID it - YYYYMMDD) under WinTOC output (F3 RUN SCRN then Setup then WinTOC Output, 2 places to save).

14.2.7. If doing an initial calibration curve use an appropriate µL syringe to make the following measurements of the sucrose standard in order to achieve the indicated concentrations. Make sure that there are no air bubbles in the syringe. Turn the syringe with the needle pointed up and vibrate the barrel and disperse any air from the syringe. Alternatively, use the solution meniscus on the line to measure the volume. To enter the calibration information on the instrument go to Instrument Cal Menu, type in the calibration standard values and save the file as the cal. <u>date analyzed</u>.

µL 30% Sucrose STD	Concentration (mg)
0	0
2.0 of 1:6 solution* (CRL)	0.10
3.0	0.90
25 (CCV/BS)	7.5
50	15

* The 1:6 solution of the 30% Sucrose standard is prepared by mixing 100µL of the 30% Sucrose standard with 500µL of water. The above standards are run in duplicate.

Note : Sucrose solutions gum up syringes quickly, rinse several times with DI water and then acetone after measuring standards.

14.2.8. Analysis may begin with CCV/CCB/CRL if an ICAL is not analyzed. Enter the sequence to be analyzed as listed below (Databases then Sequences, default weight of 250mg is used for QC, Long Method is used for really high samples or ones with a lot of matrix issues – has extra time to remove matrix before running the next sample):

14.2.8.1. CCV(CCV1+ date analyzed for ID) or Initial calibration – single analyses
14.2.8.2. CCB (Or ICB)
14.2.8.3. CRL standard (required for Lloyd Kahn)
14.2.8.4. Method Blank(MB + date analyzed for ID) – single analyses
14.2.8.5. BS, 15 mg dextrose (BS + date analyzed for ID) – single analyses
14.2.8.6. Up to 10 field samples (+ QC) in duplicate
14.2.8.7. CCV(CCV2+ date analyzed for ID) – single analyses
14.2.8.8. CCB (CCB2+ date analyzed for ID) – single analyses
14.2.8.9. Up to 9 field samples (+ QC) in duplicate
14.2.8.10. One field sample in quadruplicate
14.2.8.11. CCV (CCV3+ date analyzed for ID) – single analyses
14.2.8.12. CCB (CCB3+ date analyzed for ID) – single analyses
14.2.8.13. Up to 10 field samples (+ QC) in duplicate
14.2.8.14. CCV(CCV4+ date analyzed for ID) – single analyses
14.2.8.15. CCB (CCB4+ date analyzed for ID) – single analyses
14.2.8.16. Up to 9 field samples (+ QC) in duplicate
14.2.8.17. One field sample in quadruplicate
14.2.8.18. FCV(CCV5+ date analyzed for ID) – single analyses
14.2.8.19. FCB(CCB5+ date analyzed for ID) – single analyses

14.2.9. Samples are stored away from light and at 4°C. Wearing labcoat, gloves and safety glasses remove samples from sample storage signing them out appropriately on the internal chain of custody form.

14.2.10. Transfer a homogeneous aliquot(~5 g) of the sample into a small pre-labeled aluminum weighing pan. Label each pan in two places with the appropriate sample ID then add enough phosphoric acid (1-2 ml) to remove the Total inorganic carbon (TIC) when the sample is placed in an oven at 250°C. Use a tongue depresser to mix the sample/acid. Place the samples in the 250°C oven for 10 minute and begin prepping the sample cups to weigh 0.2g-1.0g of each sample (in duplicate or quadruplicate, as required). Limit the time that the cups are exposed to the atmosphere as to reduce potential contamination. **Note: Since the samples are dried in this manner before the sample aliquot it taken, a % solids determination and calculation is NOT necessary to report the sample concentrations in dry weight.** After samples are dried, crush samples using a clean mortar and pestle.

14.2.11. Set the OI 1010 to the TC Mode and start running the sequence beginning with the initial calibration or calibration verification standard as illustrated above. Weigh each sample in duplicate or quadruplicate, making sure to limit the time that samples are exposed to the atmosphere. Default is 250mg. If using less than 50mg, samples must be run in quadruplicate.

14.2.12. Each run takes 7 minutes. If the results are not as expected/needed, abort the run and troubleshoot.

14.2.13. The Excel file for calculations is located at: "V:\Standard Operating Procedures\Current SOP File Directory\Worksheets" or can be accessed through TOC_SOPs_Controlled_Documents spreadsheet at V:\Standard Operating Procedures. The sample identity, its corresponding mgC

reading, and the sample weight are entered into the appropriate columns. The Excel worksheet is self explanatory. Normal procedure is followed for preparing reports and the data is second checked before being given to the group leader.

14.2.14. To print the run log that contains the results, open the Utilities tab in the operating software. Select view Run Log, then select the file that contains the results. The file can then be printed from the window that is opened.

15. Data Analysis and Calculations

15.1. Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

16. Method Performance

16.1. The instrument used for the Water TOC analysis is a Shimadzu Total Carbon Analyzer with TOC-Vsw/TOC-Control V version 1.07.00.00sp1 software. An OIC 1010 soil/sediment carbon analyzer is used for soil samples with WinTOC3.0 software.

16.2. There is a Shimadzu autosampler which will hold 68 samples.

16.3. The corresponding data for each sample is obtained from the Shimadzu software for the water samples. The soil/sediment data are printed out at the organic GC printer.

16.4. **Precision and Bias for Total Organic Carbon (TOC) by Persulfate-Ultraviolet Oxidation. (Water samples)**

Characteristic Of Analysis Concentration determined, mg/L:	Spring Water	Spring Water +0.15 mg/L KHP*	Tap Water	Tap Water +10 mg/L KHP*	Municipal Wastewater Effluent
Replicate 1	0.402	0.559	2.47	11.70	5.88
Replicate 2	0.336	0.491	2.49	11.53	5.31
Replicate 3	0.340	0.505	2.47	11.70	5.21
Replicate 4	0.341	0.523	2.47	11.64	5.17
Replicate 5	0.355	0.542	2.46	11.55	5.10
Replicate 6	0.366	0.546	2.46	11.68	5.33
Replicate 7	0.361	0.548	2.42	11.55	5.35
Mean, mg/L	0.35	0.53	2.46	11.53	5.32
Std. Deviation: mg/L	0.02	0.03	0.02	0.21	0.23
%	6	6	1	2	4
Actual Value, mg/L	-	0.50	-	12.46	-
Recovery, %	-	106	-	93	-
Error, %	-	6	-	7	-

***KHP = potassium hydrogen phthalate.**

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed in the Quality Control SOP QS03 is completed by each analyst and then provided to the group leader for further processing and approval.

See SOP QS08 for criteria and corrective actions associated to the following method performance items:
16.5Method Detection Limit Study or Detection Limit Determination
16.6Limit of Detection Verification
16.7Limit of Quantitation or Reporting Limit Verification
16.8Demonstration of Capability (DOC)
16.9PT Studies

17. Pollution Prevention

17.1. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

17.2. All waste from Aqueous TOC Analysis is collected and labeled as corrosive and is placed in the appropriate area for login neutralization and disposal.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria.

19. Corrective Actions and Out-of-Control Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

19.1. Instrument Related

19.1.1. ICV not within ± 20% or ±10% (5310C)

19.1.1.1. If the problem is with the solution

19.1.1.1.1. Re-prepare, obtain new stock if necessary.

19.1.1.2. If the problem is with the calibration

19.1.1.2.1. Recalibrate through analysis of appropriate standards and recheck ICV.

19.1.2. CCV (CRL for Lloyd Kahn) not within ± 30% (Soil) or ±20% (Water) or ±10% (5310C)

19.1.2.1. If the problem is with the solution

19.1.2.1.1. Re-prepare, obtain new stock if necessary.

19.1.2.2. If the problem is with the calibration

19.1.2.2.1. Recalibrate through analysis of appropriate standards and re-prepare /reanalyze the previous ten samples according the following guidelines.

19.1.2.2.1.1. If the CCV was biased high, any of the previous samples which were below the minimum detection limit do not require reanalysis.

19.1.2.2.1.2. If the CCV was biased low, the previous samples must be reanalyzed.

19.1.3. BS not within ±20% (±15% 5310C)

19.1.3.1. If the problem is with the solution

19.1.3.1.1. Re-prepare, obtain new stock if necessary.

19.1.3.2. Discuss with group leader if recovery continues to exceed limits.

19.2. Sample Matrix Related

19.2.1. Replicate analysis RPD not within ±20% aqueous or ±50% soil/sediment

19.2.1.1. The associated sample data must be qualified on the final report unless third analysis performed within requirements.

19.2.2. Spike analysis recovery not within ±25% aqueous or ±50% soil/sediment (BS limits for DoD)

19.2.2.1. If the analyte level in the sample is greater than 4X the spiking level, the %recovery cannot be evaluated and no action is taken.

19.2.2.2. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A non-conformance report must accompany the data and be emailed or given to the group leader.

20. Contingencies for Handling Out-of-Control or Unacceptable Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

21. Waste Management

21.1. Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.2. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

22. Equipment / Instrument Maintenance

22.1. Equipment:

22.1.1 Shimadzu Total Organic Carbon Analyser (TOC-VWS)

22.1.2 Shimadzu ASI-V Autosampler

22.1.3 OIC Analytical Solids TOC Analyzer (Model 1010)

22.2 Maintenance (Shimadzu Total Organic Carbon Analyser (TOC-VWS):

22.2.1 Replace the CO2 absorber once annually.

22.2.2 Replace halogen scrubber after it changes to a black color.

22.3 Maintenance (Shimadzu ASI-V Autosampler):

22.3.1 Periodically wipe down autosampler instrument.

22.4 Maintenance (OIC Analytical Solids TOC Analyzer (Model 1010)):

(See chapter 5 maintenance in the O.I. Solids TOC Analyzer manual for instructions).

22.4.1 Replace combustion tube as needed.

22.4.2 Replace particulate filter (quartz wool) if the filter is dirty or discolored.

22.4.3 Replace gases as needed.

22.4.4 NDIR zero

22.4.5 Permeation Tube

22.4.6 Conditioning sample cups

23. Computer Hardware and Software

23.1. Computer Hardware: Windows XP 20gb hard drive with 512mb ram.

23.2. Software for Water Instrument: TOC-Vsw/TOC-Control V version 1.07.00.00sp1 software

23.3. Software for Soil Instrument: Win TOC Solids 3.0 (2001 01 Analyzer)

24. Troubleshooting

24.1. Refer to TOC-VWS user manual section 5.6 Troubleshooting for error messages list and troubleshooting charts.
24.2. Refer to OI Solids TOC Analyzer manual; Chapter 6.

25. Reference

25.1. Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).
25.2. EPA SW-846, Method 9060/9060A.
25.3. Lloyd Kahn Method, *"Determination of Total Organic Carbon in Sediment"*
25.4. Standard Methods – 22nd Edition.
25.5. DoD Quality Systems Manual for Environmental Laboratories, version 5.0, 7/2013 [Based on ISO/IEC and the NELAC Institute (TNI) Standards, Volume 1, (September 2009)].

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Appendix I – Basic TOC start-up notes for analysis.
26.2. Data Review Checklist

APPENDIX I. Basic Water TOC start-up notes for analysis.

1. Power up the lamp for warm –up, check reagents inside instrument cavity to make sure all are filled before starting the run.
2. Fill Fresh DI water in 1 gallon jug; DI squirt bottle and 1 L plastic
3. Label and load VOA vials with standards and samples into round tray.
4. Place round tray onto autosampler, get a final sample count for end point and replace lid.
5. Make sure that round tray fits down flush onto the autosampler.
6. On computer screen, select "TOC-Control V" icon.
7. Then select "Sample Table Editor"
8. Enter user name: "analyst initials" select OK.
9. Under "File" select "calibration curve" "OK".
10. Under system select Shimadzu TOC-BWS Enter/next
11. Select Edit Calibration points manually Enter/next
12. Under "Analysis" select "NPOC" then make up your file name (use today's date) Enter/next.
13. Calibration Measurement Parameters are default: Just hit "next"
14. Select "ADD" and enter calibration points (Analysis may begin with CCV/CCB/(CRL) if an ICAL is not analyzed.). After 8 points it should show 0.00 mg/L first and 100 mg/L eighth. If so "next"
15. Put a check mark in "Correlation Coefficient" check box "next"
16. "next"
17. "finish"
18. Go to file and select "new", "sample run" "ok" "ok" enter file name: user date "save"
19. Now go to insert and select "calibration curve" then scroll till you find your file name/date should have .cal after date "select" the "open"
20. You should now see the sparging /acid addition page which shows a picture of the round sample tray. Under vial manually enter "1"beside 0.00 mg/L.
21. manually enter "2" beside 1.0 mg/L and "3" beside 2.5 mg/L and so on and so forth all the way to "8" this shows what order they ar loaded on the tray. "Enter/OK"
22. Then a screen with your filename/date and all info should be in row 1 only with vial column showing. 1,2,3,4, etc.
23. Select the lightning bolt symbol then enter "use PC settings" this will start initializing wait till screen goes away then you will see the stop light symbol appear with green light showing, select that icon select "keep running" select "standby"
24. Sparging/acid addition page will re-appear just hit "OK"
25. Start ASI tray screen will appear hit "Start"
26. The instrument should start establishing the baseline and move auto tray into position – Lid must be on and samples loaded into correct position will take almost 3 hours to finish. Can view data as its coming off by selecting "view" "sample window". After calibration is done review.
27. Select "File" then "New" then "sample run" "ok"
28. General information screen: No change select "ok"
29. Save as screen: Use today's date for file name example YYYYMMDD
30. Select "save"
31. Sample Table Screen: Select "insert" then select " auto generate" enter
32. **Page 1** sample group wizard sample source: select "calibration curve" then double click on box with 3 dots …
33. Open latest curve from calibration curves file
34. Highlight latest curve and select "open"
35. Should send you back to page 1 with calibration curve info submitted. Select "next"
36. **Page 2** Sample Parameter: Enter final sample count for "number of samples" select "next"
37. **Page 3** Calibration Curves: No changes Select "Next"

38. **Page 4** Calibration Checks: No changes Select "Next"
39. **Page 5** Controls: No changes select "finish", Select "ok" on "Sparging/ Acid page.
40. Type sequence as they are loaded on tray: ICV, ICB, BSW, Sample #, client,etc.
41. Once everything is typed in, double check that it matches the way samples and QC are loaded..
42. Click or select the lightning bolt symbol then select "use settings on PC". Wait for initializing. When screen goes away, the traffic light symbol should appear next to the lightning bolt symbol. Click on the traffic light symbol.
43. Click or select "shut down Instructions". Then select "standby" Sparging/ Acid addition screen will appear so you can confirm your tray is loaded the wax things are highlighted in blue. Select "OK" if it looks the same.
44. Start ASI measurement: External acid addition should have a check mark click on "start" analysis should begin to start.
45. Click on view and chose "sample window" to watch curves come off and to see beginning values.

Attachment 2

TOC Data 2nd check

- ❑ Analyst authorization
- ❑ All general information complete
- ❑ Calibration meets criteria/ Date_____
- ❑ Base MDL
- ❑ Correct Units
- ❑ Corrections crossed out, initialed and dated, with reason (if necessary)
- ❑ Curve correlation & date good
- ❑ Dilution factors or Soil sample amounts correct
- ❑ TV's & sources indicated
- ❑ CRL performed and within control
- ❑ CCV/CCB done where required & in control
- ❑ BLK/BS per day & per 20 samples & in control (per 10 samples if 5310C)
- ❑ MS/MSD per 20 & in control
- ❑ MS prep included
- ❑ Blanks before samples all <DL
- ❑ 10% check of transcription from instrument
- ❑ Samples noted when <DL
- ❑ 2nd check of LIMS entry
- ❑ BS, MS/MSD prep. indicated &/or calculation shown
- ❑ Reagents/Standards verified accurate on bench sheets and in LIMS.
- ❑ Holding time met
- ❑ 9060/A samples analyzed in quad
- ❑ One sample per 20 analyzed in quad for Lloyd Kahn
- ❑ Problems discussed with manager
- ❑ Necessary NCR's attached
- ❑ Sample _____ used for recalculation from raw data to final LIMS concentration
- ❑ Additional information needed for reports:

2nd checked by/date:

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 225 REVISION #: 12 EFFECTIVE DATE: 20150921

GC or GC/MS VOLATILE NON-AQUEOUS MATRIX EXTRACTION USING SW-846 METHOD 5035/A FOR 8015B/C/D or 8260B/C ANALYSIS

APPROVALS:

Lab Director: _____ Date: **20150921**

Data Quality Manager: _____ Date: **20150921**

Group Leader: _____ Date: **20150921**

This SOP was reviewed for accuracy by Antonio Monteiro 9/5/16 and found to need no changes.

Changes Summary

SOP225_R12_20150921_5035

- All references to supervisor updated to reflect group leader.
- References to SOP202 updated to add SOP219.
- Section 9 updated to reference QS11 instead of appendix and remove instrumentation references.
- Section 10 update in reference QS04.
- Section 14 updated for storage and vial preparation.
- Section 15 updated to remove analytical instrument and method references.

Revision 11 20140722

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Section 14 updated to include procedure for bulk jar sub-sampling.
- Appendix I removed – located in SOP QS11.
- Reference to analysis by 8260C added.

Revision 10, 20120306

- The SOP is an update from Revision 09 dated 09/07/10
- The SOP has been updated to reflect QEC vials and in-house preparation of preserved vials including an appendix detailing the process for in-house vial preparation.
- References to standard logbooks have been updated to reference LIMS data entry.
- References to the Organic Lab Manager have been updated to Section Supervisor.
- Additional detail has been provided in sections 8 and 9.3.

Revision 09, 09/07/10

- The SOP is an update from Revision 08 dated 09/24/08
- The SOP has been updated to include reference to 5035A and preservation by freezing for unpreserved Terracores and Encores.

Table of Contents

GC or GC/MS VOLATILE NON-AQUEOUS MATRIX EXTRACTION USING SW-846 METHOD 5035/A FOR 8015B/C/D or 8260B/C ANALYSIS

1. ID of the method

This SOP details soil sample preparation for EPA method SW-846 5035 / 5035A.

2. Applicable matrix and matrices

This SOP details soil sample preparation for EPA method SW-846 5035 / 5035A.

3. Limits of detection and quantitation:

Not applicable

4. Scope and application, including parameters to be analyzed:

The purpose of this SOP is to detail soil sample preparation for EPA method SW-846 5035 and 5035A. Soil samples should be sampled in the field using the EnCore™ sampler or prepared VOA vials (sometimes referred to as Terracore samplers) then shipped to the lab within 24 hours for preservation, storage and analysis. This SOP should be used in conjunction SOP-202 or SOP219, which detail the analytical techniques.

5. Summary of the method

Samples are collected in EnCores or prepared VOA vials and submitted to the laboratory for preparation/analysis. EnCore samplers have to be frozen or prepared within 48 hrs of collection. Pre-preserved VOA vials (sometimes referred to as Terracores) are shipped already prepared in water, methanol or preservative solution. If prepared in water, freezing is required within 48 hours. If preservative is used, the sample must be cooled to 4°C and analyzed within 14 days of collection.

6. Definitions

Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

7. Interferences

Sample vials can be a source of contamination. Vials should be checked for contamination before use. Samples can be contaminated during sample prep. Prep blanks should be prepared at the same time as the samples to check for contamination.

8. Safety

8.1. Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be follow lab wide.

8.2. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex or nitrile gloves and lab coats is highly recommended.

8.3. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.4. MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your Group Leader, lab director or data quality manager if there are any difficulties in accessing these records.

9. Equipment and supplies

9.1 Sample Containers – 40mL VOA vials with low bleed septa. Available from QEC (Part No. 2112-40ml-EMP pack of 72), alternate sources are possible but must be checked for contaminants before use. ESS also supplies pre-prepped vials with the preservative and stirbar (Part No. PC4039-5035, pack of 72).

9.2 Top-loading balance – capable of accurately weighing to 0.01g.

9.3 1-10 mL Adjustable Dispenser from Thermo-scientific. Also available from Oxford (Part No. 8885-040009).

9.4 Wooden Tongue Depressors

9.5 Magnetic stirring bars – PTFE- or glass-coated, of the appropriate size to fit the sample vials. Available from A. Daigger (Part No. WX22782A, case of 50).

9.6 EnCore™ sampler – (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent. Necessary for field sampling crew.

9.7 Terracore Vials- Available from QEC or prepared in-house (see QS11).

9.8 Balance weights – used to calibrate the balance.

9.9 Labels

9.10 Gloves – Evolution One Microflex or equivalent

10. Reagents and standard

Quality Systems SOP QS04 " TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method. All reagents shall be made from ACS reagent grade chemicals. All reagents used for distillation and analysis are entered into Element. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the samples they were used with.

10.1. Reagent Water - Reagent water is NANO PURE WATER from source in the VOA instrument lab.

10.2 Methanol, CH_3OH – purge-and-trap quality, or equivalent. Store away from other solvents.

10.3 Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).

10.4 Sodium bisulfate solution – Prepare by adding 200 grams of $NaHSO_4$ (ACS reagent grade, or equivalent) to 1000 milliliters of reagent water. Record the vendor and lot number of the $NaHSO_4$ in the LIMS. Each standard/reagent that is prepared is recorded in the LIMS and receives a sequential number. The label is completed with the standard/reagent number, name, preparation date, expiration date, solvent and analyst initials. The solution should be discarded after six months or sooner if it shows signs of contamination.

11. Sample collection, preservation, shipment and storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 EnCores are prepped within 48 hrs of collection or frozen until preparation can be completed. Preparation can be in sodium bisulfate with refrigeration at 4°C or in reagent water with freezing. Prepared VOA vials are received already prepared in water, methanol or sodium bisulfate solution. If prepared in water, freezing is required within 48 hours of collection. If preservative is used, refrigeration at 4°C is the only requirement. Holding Time is 14 days from collection once preserved.

11.3 As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of volatile compounds. Always wear gloves whenever handling the tared sample vials. Several techniques may be used to perform the transfer of the sample to the relatively narrow opening of the low concentration soil vial such as the EnCore™ sampler, a cut off disposable plastic syringe, or a wooden tongue depressor. We prefer to use the EnCore™ sampler or Terracore sampler with prepared VOA vials.

11.4 The EnCore™ sampler is both a sampler and a container for low-level and high level soils. It is designed to collect an average weight with the exact weight to be determined in the lab. It is disposable and is also designed to have zero headspace. The EnCore™ sampler will require the field personnel to get the sample to the laboratory within 24-36 hours of collection. The laboratory needs to be contacted prior to sample collection to ensure that all necessary containers (with or without preservative) are available and that the proper sampling technique is used.

11.5 All low-level soil samples must be collected in duplicate to allow the laboratory an additional sample for reanalysis. A third sample should be collected for preparation of a high-level sample. This sample would be prepared at the same time as the "low-level" sample. (Some projects may not require the "low-level" detection limits, in this case only the high level sample preparation would be required.). A fourth sample may be collected to enable the laboratory to perform a pretest on the soil to determine if the soil sample contains carbonate minerals that will effervesce upon contact with the acidic sodium bisulfate preservative solution in the low concentration sample vial. The additional soil samples must be collected from the same soil stratum or the same section of solid waste being sampled and within close proximity to the location from which the original sample was collected. **Additional bulk samples should be collected for screening and dry weight determination without the preservative.**
Note: If the low-level sample cannot be preserved with sodium bisulfate, the remaining low-level sample aliquot(s) is (are) transferred to a pre-weighed vial containing 5 mL of reagent water. The sample in the unpreserved vial must either be analyzed immediately (within 48 hours of collection) or frozen within the 48 hour time frame and then analyzed within the 14 day holding time.

12. Quality control

Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

13. Calibration and standardization

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

14. Procedure

14.1 Sample Receiving personnel will log the samples in, place them in the VOA Soil Hobart assigned for volatile sample storage (if pre-preserved) or the VOA freezer (if preserved in water or received in Encore samplers).

14.2 If samples are received in Encores or bulk jars, Sample Receiving personnel will notify the Group Leader that samples are in-house for VOA soil preparation.

14.3 The Group Leader or designee will determine the amount of time remaining on the 48 hour holding time and assign the task of preserving the samples. On EnCore™ samplers, holding time is extended to 14 days when stored in the freezer.

14.4 Samples received from the field should be designated for low-level, high-level or % solids/screening (this fraction should be in a regular soil jar, if it is not, it will require transfer to a VOA vial). Each low-level and high-level sample must be preserved appropriately as follows:

14.4.1 Organize the VOA vials required and label them with the sample number (including letter designation), date and LOW (water), LOW (NaHSO4) or HIGH (MeOH) for either low-level water, low-level NaHSO4 or high-level preservation.

14.4.2 Get the samples from storage and log them out.

14.4.3 Enter the sample numbers (including letter designation) in the soil sample preparation logbook and add a sample preparation/storage blank to the book for each level being prepared (HIGH/LOW). There must be a line in the logbook for each sample vial being prepared (i.e. if there are 2 low-level samples and 1 high-level sample, the sample number should be listed in the logbook 3 times- use a,b,c to designate each vial associated with the same sample). Make sure each samples letter designation is accurately assigned for LOW or HIGH preparation.

14.4.4 For Encore™, place the vial (LOW/HIGH) on the top-loading balance, tare the vial then extrude the sample into the vial and record the weight of the sample in the sample preparation logbook. For bulk jars, remove top layer of soil and discard. Loosen and mix the middle layer. From the center of the jar, take and add approximately 5 grams of soil to vial then record weight in the logbook. Make sure the lip of the vial does not have any soil on it, which might cause a leak, and cap the vial tightly.

14.4.5 Using an adjustable pipettor, add 5 mL P&T methanol to each of the vials marked HIGH. Then record the vendor & lot number of the vials, vendor & lot number of methanol and the volume of methanol added to each sample in the sample preparation logbook. If the vial is not to be used immediately, weigh the vial to the nearest 0.01g and record the weight on the vial. The vial weight must be verified to be within ±0.01g of this value before using for sample preparation.

14.4.6 For each of the vials marked LOW (NaHSO4), add 5 mL of sodium bisulfate. Then record the vendor & lot number of the vials, vendor & lot number of NaHSO4 and the volume of NaHSO4 added to each sample in the sample preparation logbook. Add a magnetic stir bar to each vial. If the vial is not to be used immediately, weigh the vial to the nearest 0.01g and record the weight on the vial. The vial weight must be verified to be within ±0.01g of this value before using for sample preparation. If pre-prepped vials are used, this step is unnecessary but the lot number and the pre-prepped status must be recorded in the preparation log.

14.4.7 For each of the vials marked LOW (water), add 5 mL of reagent water. Then record the vendor & lot number of the vials, source of the reagent water and the volume of reagent water added to each sample in the sample preparation logbook. Add a magnetic stir bar to each vial. If the vial is not to be used immediately, weigh the vial to the nearest 0.01g and record the weight on the vial. The vial weight must be verified to be within ±0.01g of this value before using for sample preparation. If pre-prepped vials are used, this step is unnecessary but the lot number and the pre-prepped status must be recorded in the preparation log.

14.4.8 Place the preserved samples in a box, return them to the VOA freezer (LOW water w/HIGH MeOH) or Soil Walk-in Cooler (LOW NaHSO4 w/HIGH MeOH) assigned for volatile sample storage and log them back in.

NOTE 1: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the samples lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and place low concentration samples in vials that contain 5ml water and a stir bar. This sample must be frozen in a slanted position until analysis or analyzed within 48 hours of sampling. Notify the Group Leader if this occurs, note this in the sample preparation logbook and generate an NCR to document the problem.
NOTE 2: Excessive weights (>7.5g) may plug the purge needle and cause significant down time for the instrument. Notify the Project Manager and Group Leader whenever samples are received with excessive weights.

15. Data analysis and calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Calibration of the analytical instrument with subsequent analysis of the samples is covered under SOP202 or SOP219.

15.3 Determination of % Dry Weight

15.3.1 Weigh 5-10 grams of the sample from the bulk jar used for dry weight analysis in a tared crucible or aluminum pan.

15.3.2 Dry overnight at 105°C.

15.3.3 Allow to cool in a desiccator before weighing.

15.3.4 Calculate % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

15.4 If an extra bulk jar was not received for percent moisture determination, an alternate procedure incorporating the methanol vial can be used. If pre-preserved vials are received, start with bullet 3 and proceed to bullet 5:

15.4.1 Weigh the sample in the VOA vial to the necessary degree of accuracy for % solids (recommend a tare weight on the vial to the same degree of accuracy).

15.4.2 Preserve the vial as normal.

15.4.3 After all required analyses have been confirmed completed from the methanol extract, allow the methanol to evaporate and dry as necessary for % solids determination. Note – it is essential to retain the cap with the vial as the cap is part of the original tare weight.

15.4.4 Weigh the sample in the VOA vial (with cap) to the necessary degree of accuracy for % solids.

15.4.5 If sample was received preserved and it was not possible to obtain an original vial weight to the accuracy required by % solids, clean and dry the vial as necessary for % solids (including the cap) the obtain the vial weight for use as the original vial weight in % solids calculation.

16. Method performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within QS03 is completed by each analyst and then provided to the group leader for further processing and approval.

See Table 2 of SOP202 or SOP219 for criteria and corrective actions associated to the following method performance items:

19.1 Method Detection Limit Study or Detection Limit Determination
19.2 Limit of Detection Verification
19.3 Limit of Quantitation or Reporting Limit Verification
19.4 Demonstration of Capability (DOC)
19.5 PT Studies

17. Pollution prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data assessment and acceptance criteria for QC measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria.

19. Corrective actions and out-of-control data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20. Contingencies for handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating

Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

21. Waste management

Please see Waste Disposal SOP QS14 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

22. Equip./instrument maintenance

Not applicable

23. Computer hardware and software

Not applicable

24. Troubleshooting.

Not applicable

25. References

25.1.Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 5035, December 1996.

25.2.Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Other Methods; Method 5035A, July 2002.

26. Tables, diagrams, flowcharts and validation data

Not applicable

EMPIRICAL LABORATORIES, LLC (EL)
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 236 **REVISION #: 10** **EFFECTIVE DATE: 20161108**

METHANE, ETHANE, ETHENE IN AQUEOUS SAMPLES BY MODIFIED RSK-175 (AUTOMATED HEADSPACE)

APPROVALS:

Lab Director: _____ Date: **20161108**

Data Quality Manager: _____ Date: **20161108**

Group Leader: _____ Date: **20161108**
Jade Holliman

Changes Summary

SOP236_R10_20161108_MEE

- Calibration and associated calculations updated to reflect initial calibration in water instead of gas.
- Measurement of water for calibration and QC samples updated to use of 25mL gastight syringe.

SOP236_R09_20160812_MEE

- Added reference to 8000D for retention times.
- Updated instrument settings in section 14.1.
- Table 1 updated for header definitions.
- LLOQ and IDP terminology added from SW-846 Update V.
- MDL/LOD/LOQ references simplified in table 2.
- Revised checklist to include linear calibration read-back with list/discuss and to remove extra calculation check line.

SOP236_R08_20151221_MEE

- Checklist updated to include verification that reagents/standards are recorded on the bench sheet and in the LIMS.

SOP236_R07_20150921_MEE

- All references to supervisor updated to reflect group leader.
- Section 10 updated to reflect QS04.
- Table 2 updated with DoD 2 CCV allowance.

Revision 06, 20140722

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark has been updated to include proprietary reference.
- DL/LOD/LOQ table moved from section 3 to Table 1 and updated to include QSM 4.2 and QSM 5.0 control limits.
- Section 8.0 MSDS sheet reference updated to reflect vendor website access.
- Section 9.0 Equipment and supplies updated.
- Sections 22.0, 23.0 and 24.0 added/populated.
- Main text limit references updated to direct analyst to table 2 throughout the document.

Revision 05, 20130711

- Section 15.2: Updated to include column and row references indicated in calculations.

Revision 04, 20130612

- Section 10.3: indicate gas standards are purchased at concentration of 1.
- Section 11.0: Indicate samples can be received unpreserved or preserved with HCl like VOA samples.
- Section 13.1: correct RSD criteria to indicate must be “less” than 20 percent.
- Section 13.2: add MS/MSD to BS reference.
- Section 14.3.2 and 14.3.3: reference section 13.2 for spiking protocol.
- Section 14.4.1: indicate volume verification daily for representative vial.
- Section 15.2: Improved fit to make sure no information is truncated.
- Table 2: Analyst Review Checklist updated.

Revision 03, 20120716

- Autosampler information updated in sections 9 and 14.
- Example MEE run sequences added to sections 13.4 and 14.6.
- Process for preparing individual ICAL, ICV, CCV and LCS standards added to section 13.
- Redundant references to demonstration of capability removed at 12.2 and 14.6 with update in section 16.1.
- Section 15.2 calculation example updated.
- Table references updated.

Revision 02, 09/07/2010

- The SOP is an update from Revision 01 dated 04/28/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.
- Calculations have been added.

Table of Contents

METHANE, ETHANE, ETHENE IN AQUEOUS SAMPLES BY MODIFIED RSK-175 (AUTOMATED HEADSPACE)

1. **Identification of the Test Method**
   The GC/FID/Headspace system is used to analyze methane, ethane, and ethene in aqueous samples using method RSK-175.

2. **Applicable Matrix or matrices**
   Aqueous samples.

3. **Limits of Detection and Quantitation**
   See Table 1.

4. **Scope and Application, Including Parameters to be Analyzed**
   The GC/FID/Headspace system is used to analyze methane, ethane, and ethane in aqueous samples.

5. **Summary of the Test Method**
   This test is used to determine the amount of Methane, Ethene and Ethane in an aqueous sample. Samples are collected using 40mL VOA vials without headspace. Each sample is analyzed using a Headspace autosampler and GC with FID detector.

6. **Definitions**
   Laboratory Quality System QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7. **Interference**
   Methane is found in the lab environment and can be a source of contamination.

8. **Safety**
   8.1. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be follow lab wide.
   8.2. Care should be used in handling all samples.
      8.2.1. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.
      8.2.2. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
      8.2.3. MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your section group leader, lab director or data quality manager if there are any difficulties in accessing these records.
      8.2.4. Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

9. Equipment and Supplies

9.1. Gas Chromatograph: HP 5890 Series II (temperature programmable).
9.2. Autosampler: HP 7694 Headspace Autosampler
9.3. Columns – Capillary Columns: Carbonex 1006 PLOT column – 30 meter x 0.53mm ID (or equivalent)
9.4. Data Acquisition and Processing Software: HP Chemstation system is interface to the Hp-GC for data acquisition, processing, and storing acquired data.
9.5. Detector: Flame Ionization Detector
9.6. Glassware
9.6.1.25ml Syringe
9.6.2.20ml headspace crimp vials with crimp tops (Restek #24685/#21761 or equivalent)
9.6.3.Gastight syringes - 10µL, 100µL, 500µL and 1mL

10. Reagents and Standards

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards.

10.1. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method. All reagents shall be made from ACS reagent grade chemicals. All reagents used for distillation and analysis are entered into Element. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the samples they were used with.
10.2. Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Restek, Protocol, Ultra and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system.
10.3. Gas standards are purchased from Restek and other vendors depending on their availability. The date they are received is noted on the container they are received in. The standards are given a sequential number the day they are opened and this is noted on the tank. Standards for MEE are Scotty gases purchased in pressurized tanks at concentration of 1% – Part#23462.

11. Sample Collection, Preservation, Shipment, and Storage

11.1. Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.
11.2. Samples are collected in 40 ml VOA vials (with 1:1 HCl preservative or without preservative) and shipped to the lab in coolers with ice. Water samples are stored in the water walk-in cooler at a temperature of 0°C-6°C.

12. Quality Control

Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

13. Calibration and Standardization

13.1. Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2. A minimum five point calibration curve must be introduced into the GC and analyzed for each analyte of interest using the appropriate instrument parameters prior to running samples. If the percent relative standard deviation (% RSD) of the calibration factor is less than 20 percent over the working range, linearity through the origin can be assumed and the average calibration factor can be used in place of a calibration curve. Check acceptability of linear and quadratic curve fits by re-calculating each calibration point back to the curve. The curve is then verified using a second source standard – separate lot from the calibration standards. See table 2 for criteria.

13.3. Calibration standards at a minimum of five levels are prepared as follows:

13.3.1. Document temperature of the area in the MEE temperature logbook and on the analytical listing for transfer to the data review checklist. Acceptance limits are 21.1°C+/-4°C (inclusive).

13.3.2. Clean syringes by rinsing with methanol then acetone then pumping with air 3 times to ensure no plugging.

13.3.3. Add 15ml DI water to a 20ml headspace vial using the 25mL gastight syringe.

13.3.4. Cap immediately and check cap for secure fastening

13.3.5. Turn on Scotty Gas.

13.3.6. Insert appropriately cleaned gas-tight syringe, remove plunger, and listen for hissing.

13.3.6.1. If no hiss – clean syringe or change flow meter septum.

13.3.6.2. If hiss, insert plunger to appropriate volume and remove syringe.

13.3.7. Insert syringe at periphery of cap's septum and inject gas through below water's surface watching for bubbles.

13.3.8. Shake all bubbles free and analyze immediately.

13.3.9. Common Calibration/Spike Standards are listed below:

Description	Syringe (µL)	Amount (µL)
Cal1	10	2.5
Cal2	10	5.0
Cal3	10	10
Cal4	100	50
Cal5	100	100
Cal6	1000	500
Cal7	1000	1000
Cal8	1000	2000
ICV	1000	500 (2^{nd} lot)
CCV	1000	500
BS/MS/MSD	1000	500 (2^{nd} lot)

13.4. The calibration curve must be verified every day samples are run through the analysis of a mid-level standard at the beginning of the sequence, after every 10 field samples and at the end of the sequence. The percent difference back to the curve must not exceed 20 percent. If this requirement is not met, corrective action must be taken before sample analysis continues. Usually this involves recalibration or checking the gastight syringes.

13.5. A typical calibration sequence follows:
Instrument Blank

Calibration Point 1 through calibration Point 8
Initial Calibration Verification
Sample Analysis could continue per Section 14.6

14. Procedure

The following information describes the instrument and QC requirements to analyze by this method:

14.1. Instrumentation

14.1.1. GC

14.1.1.1. Initial Temperature: 35 º C, no hold
14.1.1.2. Ramp: 24 ºC/minute to 150 º C.
14.1.1.3. Final Temperature: 150 º C, no hold
14.1.1.4. Detector Temperature: 230 º C.
14.1.1.5. Injector Temperature: 150 ° C

14.1.2. Headspace Autosampler

14.1.2.1. Oven Temp: 30 º C/Platen Equilibration Time: 0.50 min.
14.1.2.2. Vial Equilibration Time: 5.0 min.
14.1.2.3. Pressurization Time: 1.0 min.
14.1.2.4. Loop Fill Time: 1.0 min. / Loop Equilibration Time: 0.5 min.
14.1.2.5. Injection Time: 1.5 min.
14.1.2.6. Loop and Transfer Line Temp.: 90 º C
14.1.2.7. GC Cycle Time: 14.5 min.

14.2. Retention Time (RT) Windows - RT criteria set forth in SW-846 method 8000D section 11.6 are used to set retention time windows. New in-house retention time windows are established after major changes to the system (new temperature program, different column phase or diameter). If the established retention time window is less than +/ 0.03 minutes, the window defaults to +/ 0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed in the same sequence as a curve.

14.3. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

14.3.1. A method blank is required every 20 samples. Any detected concentration should not exceed ½ the project reporting limit (LOQ).

14.3.2. A Laboratory Control Sample (LCS) or Blank Spike (BS) is required every 20 samples. An LCS Duplicate (LCSD) or BS Duplicate (BSD) is used if sample volume is insufficient for running MS/MSD. See section 13.2 for spiking instructions.

14.3.3. An MS/MSD pair is run (if sufficient volume is received) every batch - maximum 20 samples per batch. See section 13.2 for spiking instructions. (If insufficient volume is received, MS on one sample and duplicate on another or LCSD/BSD may be used to demonstrate accuracy and precision.)

14.4. Sample preparation includes the following steps:

14.4.1. Verify the volume of a representative crimp-top vial from the lot being used by filling with water and measuring to the nearest decimal place with the 25ml syringe. Subtract 15ml then record the result in the LIMS bench sheet as headspace volume.

14.4.2. Rinse the 25mL gas-tight syringe with DI water.

14.4.3. Remove the plunger and invert with gloved finger over the tip.

14.4.4. Fill the syringe to greater than 15mL with sample and replace the plunger.

14.4.5. Invert the syringe and displace any air bubbles.

14.4.6. Measure 15mL and add to headspace vial.
14.4.7. Cap immediately and check for secure fastening.
14.4.8. Load into the autosampler along with a similarly prepared method blank containing 15 ml of DI water, a blank spike/blank spike duplicate (BS/BSD, BSD only if no Matrix Spike/Matrix Spike Duplicate or MS and DUP volume).
14.4.9. A mid-level standard (continuing calibration verification or CCV) must be run at the beginning and end of the sequence and after every 10 field samples within the sequence. The mid-level standard cannot exceed table 2 criteria.
14.4.10. A typical sample sequence follows:
Instrument Blank
CCV 1
Method Blank
Blank Spike
Blank Spike Duplicate (NA if MS/MSD volume available)
Field Sample 1 – Field Sample 10
Matrix Spike
Matrix Spike Duplicate
CCV 2
Field Sample 11 – Field Sample 20
CCV 3

14.5. Following sample analysis, the data is reduced using the Chemstation data system. The retention times are updated with the first midpoint check of the day or from the midpoint of the calibration curve if analyzed before the samples. The following must be checked to see if the samples will require re-analyses or dilution.
14.5.1. The analyte concentration/area count must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the top half of the curve.

15. Data Analysis and Calculations

15.1. Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.
15.2. Calculations:

$$\text{Calibration Factor (CF)} = \frac{\text{Response}}{\text{Concentration}}$$

15.3 Densities at 21.1°C (derived using PV=nRT at 70°F or 21.1°C and 1ATM as in 15.4):

Analyte	MW (g/mol)	Density (g/L) at 21.1°C (70°F)
Acetylene	26.04	1.078
Methane	16.04	0.6643
Ethane	30.07	1.254
Ethene	28.05	1.162

15.4 Derivation of density using PV=nRT

R = 0.082057338 L ATM K^{-1} Mol^{-1}

Mass/Vol = (MoleMass *g/mol* * Pressure *atm*) / (R * Temp *K*)

Methane example = (16.04 * 1) / 0.082057338 * 294.25 = 0.6643 g/L

15.5 Standard concentration calculated from above density for 1% volume standard assuming 1ATM pressure:

Standard Concentration = (microliters of standard * 1/100 * density) / sample volume (L)

Methane CCV example = 500 * 1/100 * 0.6664/0.015 = 222.1ug/L

16. Method Performance

Initial Demonstration of Capability (DOC)/Performance (IDP): Each analyst must perform an IDOC/IDP prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples (may be prepared from same source as calibration). The data is calculated for accuracy and precision requirements. The IDOC form is completed by each analyst and then provided to the group leader for further processing and approval. See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

16.1. Method Detection Limit Study or Detection Limit Determination
16.2. Limit of Detection Verification
16.3. Limit of Quantitation or Reporting Limit Verification
16.4. Initial Demonstration of Capability (IDOC)/Performance (IDP)
16.5. PT Studies

17. Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

18.1. Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria.

18.2. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Corrective Actions and Out-of-Control Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20. Contingencies for Handling Out-of-Control or Unacceptable Data

20.1. Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20.2. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

21. Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

22. Equipment / Instrument Maintenance

22.1. Cool oven & injection port to 40°C.
22.2. Remove septum nut and replace septum
22.3. Remove upper weldment and replace glass liner and o-ring.
22.4. Remove the column nut & injection port nut and replace the inlet seal.
22.5. Clip/discard about one loop (40-55cm) off the inlet column. Measure 28mm from the back of the column nut to the tip of the column inlet and place back into the bottom of the injection port.
22.6. Close oven door and load the appropriated ChemStation method on the acquisition PC.

23. Computer Hardware and Software

HP Chemstation system is interfaced to the HP-GC for data acquisition, processing and storage.

24. Troubleshooting- If routine maintenance in section 22.0 did not bring the instrument back into control, more involved maintenance may be required (i.e. detector maintenance, analytical column maintenance, column splitter, etc.). Bring to the attention of the Group leader.

25. References

25.1. Newell, Bryan, RSKSOP-175, Rev.0, August 1994.
25.2. Newell, Bryan, RSKSOP-147, Rev.0, January 1993.
25.3. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Methods 8000C and 8015C.
25.4. DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003.) Dated 10/25/2010
Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories. Based on ISO/IEC 17025:2005(E) and The NELAC Institute (TNI) Standards, Volume 1, (September 2009) Dated July 2013

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. **Table 1, DL/LOD/LOQ, and recovery limits**
26.2. **Table 2, quality control criteria**
26.3. **Table 3, analyst review checklist**

Table 1, **DL/LOD/LOQ, and recovery limits**

Analyte	CAS	LOQ	LOD	DL	Units	LL	UL	LL5.0	UL5.0	RPD
Ethane	74-84-0	4.00	2.00	1.00	ug/L	80	120	74	131	30
Ethene	74-85-1	4.00	2.00	1.00	ug/L	80	120	72	133	30
Methane	74-82-8	4.00	2.00	1.00	ug/L	80	120	73	125	30

LL= Low Limit, UL = Upper Limit, RPD = relative percent difference, LL5/UL5 are limits from QSM5,

Table 2. Organic Analysis by Gas Chromatography (Method RSK-175)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of DoD QSM 4.1).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial demonstration with quarterly verification from LOD, LOQ. May be required annually for specific certifications.	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis with quarterly verification.	Refer to SOP QS08.			
LOQ establishment and verification	Prior to initial analysis with quarterly verification.	Refer to SOP QS08.			
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study or 0.03min., whichever is greater.	NA.	NA.	
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte ≤ 20% Option 2: linear least squares regression: $r \geq 0.995$ or $r^2 \geq 0.990$ Option 3: non-linear regression: coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order). Note: Linear/quadratic curve fits should have calibration standards 70%-130% when back calculated against curve.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.

Table 2. Organic Analysis by Gas Chromatography (Method RSK-175)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples (maximum of 20 for non-DoD projects), and at the end of the analysis sequence.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL Per DoD: 2 passing CCVs immediately following failing CCV allows reporting of unqualified data	If analyte exceeds with a positive bias and sample result is < LOQ, request client approval to qualify and narrate. If analyte exceeds with a negative bias, correct problem then rerun CCV. If that fails, repeat ICAL. Reanalyze all samples since last acceptable CCV. If reanalysis cannot be performed, Apply qualifier to results in all samples since last acceptable CCV and explain in the case narrative.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem, If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table 2. Organic Analysis by Gas Chromatography (Method RSK-175)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS) [LCSD (if no sample volume available for MSD)].	One per preparatory batch.	All project analytes within limits indicated in table 1.	If the LCS recoveries are high but the sample results are <LOQ request client approval to qualify and narrate. Otherwise, if sample volume available and <2x holding time, reprep and reanalyze.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS (BS). Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	One per preparatory batch per matrix.	For matrix evaluation, use LCS (BS) acceptance criteria from table 1. MSD: RPD ≤ 30% (between MS and MSD).	Corrective action will not be taken for samples when recoveries are outside limits and LCS criteria are met unless RPD indicate obvious extraction/analysis difficulties. Then, if sample volume available and <2x holding time, reprep and reanalyze MS/MSD.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS (BS) limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ.	

Table 3.
ANALYST DATA REVIEW CHECKLIST

Sample Number(s):			
Batch Number(s):			
Temperature of area during standard preparation _____ °C (acceptance limits 21.1°C ± 4°C)			
Sequence Number:	**Run Date:**	**Instrument ID:**	
Method: RSK-175	**Calibration:**	**NCR:**	

QA/QC Item	Yes	No	NA	2nd Review
A. Initial Calibration				
1. Does the curve consist of at least five Calibration Standards (six for quadratic curve)?				
2. Is the low standard equal to or below the LOQ?				
3. Are the %RSD or fit criteria within QC limits for all analytes?				
4. Is recalculation of calibration points for linear/quadratic curve fits within 70%-130% (should)? List exceedences and discuss with management prior to use.				
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 10 samples (20 non-DoD) or every 12 hours and at the end of the sequence?				
2. Are the % differences within QC limits for all analytes?				
D. Sample Analysis				
1. Are all sample holding times met?				
2. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?				
E. QC Samples				
1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than the LOD?				
2. Is the Laboratory Control Sample/Blanks Spike extracted at the desired frequency and are its percent recoveries within QC limits?				
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and are the percent recoveries/RPDs within QC limits?				
F. Others				
1. Are all nonconformances included and noted?				
2. Did analyst initial/date the appropriate printouts and report sheets?				
3. Are all manual integrations checked by a second reviewer to verify they were performed correctly?				
4. Data uploaded to LIMS correctly with associated primary analyst correct?				
5. Sample_____ showing full re-calculation from raw data through to final concentration in LIMS.				
6. Reagents/Standards verified accurate on bench sheets and in LIMS.				

Comments on any "No" response: _____

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 300 **REVISION #: 26** **EFFECTIVE DATE: 20170515**

AQUEOUS MATRIX EXTRACTION USING SW-846 METHOD 3510C FOR ANALYSIS BY: GC/MS SEMI-VOLATILE BNA-8270/625, GC/ECD Pest/PCB-8081/8082, GC/FID DRO/ORO/EPH 8015 or TNEPH

APPROVALS:

Lab Director: _____ Date: **20170515**

Data Quality Manager: _____ Date: **20170515**

Group Leader: _____ Date: **20170515**

Donald Hensel

Changes Summary

SOP300_R26_20170515_SepFunnel

- Added 3KG balance plus 500g and 1000g weights to parts section.
- Removed 1L comparator reference from section 10.3.
- Updated procedure for measurement of samples in section 14.

SOP300_R25_20161121_SepFunnel

- Added section 12.2 on quarterly rotation of PCB spiking solution to cover all certified aroclors.
- Updated 10.3 1L comparator reference to include validation per SOP QS08.

SOP300_R24_20160831_SepFunnel

- Combined SOP300, SOP302, SOP322 for separatory funnel extraction.
- Section 9: Removed duplicate reference to glass wool in reagents.
- Section 10: Added Fisher sodium sulfate option.
- Section 14: Inserted note at beginning of to indicate validation performed for method permitted modifications.
- Section 14: Added acetone rinse prior to use and hexane rinse prior to use for low-level analyses.
- Section 14: Clarified addition of BSD if no MS/MSD volume when 5 or more samples in batch.
- Section 14: Clarified pre-rinse required for non-dedicated syringes.
- Section 14: Added intermediate separatory funnel option for handling emulsions.
- Section 14: Updated to add 40mL hexane prior to concentration for pest/pcb.
- Section 14: added option to add two droppers of hexane to TV tube prior to transfer.
- Added check-off/flow chart for routine extraction steps as appendix.

SOP300_R23_20151013_BNAw

- Nitrogen tank reference updated to indicate liquid nitrogen as source.
- 1mL comparator vial added to section 10.
- Ring-stand and drying column removed from section 9.
- Procedure for overnight storage of unconcentrated extracts added.
- Added instructions for preparation timeline throughout procedure.

SOP300_R22_20150921_BNAw

- All references to supervisor updated to reflect group leader.
- SVOC regular and SVOC Low-Level clarified throughout.
- Section 10 updated to reference QS04.
- References to short-range pH paper replaced with wide-range pH paper.
- Techniques for breaking down emulsion added to section 14.13.
- Section 16 updated to reference QS03.
- Sections 18, 19 and 20 updated to reference QS05.

Revision 21, 07/31/2014

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark update to include proprietary reference.
- MSDS sheet reference updated to reflect vendor website access.
- Reference to bulk sodium sulfate updated to reflect testing prior to use and cooling in desiccator.

- Section 14.8 updated to reference handling of samples containing solids

Reviewed 20140414 by Jacque Galland and found to need no updates. Approved by Data Quality Manager Marcia McGinnity.

Reviewed 20130315 by Jacque Galland and found to need no updates. Approved by Section Supervisor Betty Quillen.

Revision 20, 03/20/2012

- Equipment and Supplies/Reagents and Standards sections have been updated.
- Procedure has been updated throughout. A complete review of this section is required.

Revision 19, 11/17/2010

- SOP has been updated to reflect current procedure used to prepare the "Baked Sodium Sulfate" and 10N NaOH solution.
- Redundant statement concerning holding time under sec. 14.0 has been removed.
- Volume correction for MeCl2 added.
- Size and labeling of containers used as well as drying procedure prior to concentration of extracts updated.
- The requirement for a matrix spike duplicate for TCLP extracts has been added.

Revision 18, 04/26/10

- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

AQUEOUS MATRIX EXTRACTION
USING SW-846 METHOD 3510C FOR ANALYSIS BY:
GC/MS SEMI-VOLATILE BNA-8270/625,
GC/ECD Pest/PCB-8081/8082,
GC/FID DRO/ORO/EPH 8015 or TNEPH

1. **Identification of the Test Method**
   This SOP is compliant with SW-846 Method 3510C and Methods 608/608.2/625. Also applicable to TNEPH "Method from the Tennessee Division of Underground Storage Tanks, Effective April 1, 1992".

2. **Applicable Matrix or Matrices**
   This SOP is applicable to aqueous samples.

3. **Detection Limit**
   Not Applicable to this SOP

4. **Scope of Application, including components to be analyzed**
   Not Applicable to this SOP

5. **Summary of the Test Method**
   Aqueous samples are extracted with methylene chloride. The extracts are dried through sodium sulfate and concentrated to an appropriate final volume.

6. **Definitions**
   Laboratory Quality System SOP QS08 "Technical/Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7. **Interferences**
   - 7.1 Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
   - 7.2 Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
   - 7.3 Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware must be solvent rinsed to avoid this problem.

8. **Safety**
   - 8.1. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab wide.
   - 8.2. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.
   - 8.3. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
   - 8.4. MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9. **Equipment and Supplies**
   - 9.1 Separatory Funnel – 2L with Teflon stopcock

9.2 Beaker – 125mL, 250mL or 400mL
9.4 Filter funnel – Glass or High density polyethylene (HDPE)
9.5 Turbo-Vap evaporation tube – 200mL tube made by Zymark or equivalent
9.6 Metal rack – capable of holding six glass evaporation tubes
9.7 Turbo-Vap Evaporator – heated and capable of temperature control (+/-5˚C); the bath should be vented into a hood
9.8 Vials, 2.0 mL glass with Teflon-lined screw cap
9.9 pH indicator paper – wide range (1.0-12.0)
9.10 Syringes – 0.5 mL or 1mL
9.11 Graduated cylinder – 1000mL, 500mL, and 100mL, glass, Class A
9.12 Pasteur pipette – length 9"
9.13 Pasteur pipette bulb
9.14 Labels – DYMO
9.15 Teflon Centrifuge Bottles – 250mL or 500mL
9.16 Volumetric Flasks – 500mL, 100mL, 50mL, and 10mL, glass, Class A
9.17 Aluminum foil – heavy duty
9.18 Liquid Nitrogen tank – equipped with pressure regulator
9.19 Boiling chips – Teflon
9.20 Glass Wool – Roving, 9989 purchased from Fisher #11-388 or equivalent
9.21 Teflon coated metal rod
9.22 Centrifuge
9.23 Percussion Hammer
9.24 Furnace – for use in cleaning water turbovap tubes at 400°C for minimum 2 hours.
9.25 3200g top-loader balance (Intelligent Weighing Technology/ PBW-3200)
9.26 1000g weight (Troemner ISO17025 certified)
9.27 500g weight (Troemner ISO17025 certified)

10. Reagents and Standards

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. All reagents and standards must be documented within the LIMS and labeled accordingly. The following information relates to the specific reagents and standards used for the performance of the method:

10.1 Reagents:

10.1.1 Reagent Water - Reagent water is gathered in a carboy from the instrument lab water system, as needed.

10.1.2 Sodium Hydroxide Solution - (10N). Weigh 800g NaOH (purchased in a fiber drum from Tennessee Reagents # 2-31825-25lb, or equivalent) into a glass or plastic container. Add approximately 1000mL of reagent water to a 2000mL volumetric flask, add a stir bar, place on stir plate, and stir. Add pellets slowly and swirl until pellets are mostly dissolved. This mixture will get very hot. Continue to add reagent water while mixture is being stirred to keep volume at approximately 1000mL. Let stand until cool. Bring to final volume. Transfer to 1000mL Teflon containers.

10.1.3 Sodium Sulfate – Pre-baked sodium sulfate Anhydrous, ACS Low Organic, Crystals purchased from Jost Chemical Co. (#2796) in 2.5 kg. glass jars. Fisher sodium sulfate anhydrous (S415-212, Granular 10-60mesh/certified ACS) in 2.5 kg. glass jars may also be used. With SVOC testing prior to use, Granular, anhydrous, trace pure 10-60 mesh may be purchased in bulk containers from Fisher # S415-10S (or equivalent). It must be placed in a Pyrex tray and heated

at 400°C for 4 hours, minimum, removed and cooled in a desiccator then placed in a 2.5 kg glass amber jug and stored at room temperature.

10.1.4 1:1 Sulfuric Acid Solution - slowly add 500mL of H2SO4 (Fisher, suitable for trace metal analysis #A300C-212 or equivalent) to 500mL of reagent water in a 1000mL Teflon container. This mixture will get very warm. Allow to cool before use.

10.1.5 Extraction Solvent - Methylene Chloride (purchased from Fisher #D151-4 or equivalent) **Please see SOP 336 before handling this solvent in our laboratory.**

10.1.6 Methanol - suitable for use in gas chromatography (Fisher A452SK4 or equivalent)

10.1.7 Acetone - suitable for gas chromatography (Fisher A18SK4 or equivalent)

10.1.8 Hexane – suitable for gas chromatography (Fisher #H300-4 or equivalent)

10.2 The extraction analyst makes up surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required and/or the final volume of the extract is adjusted.

10.2.1 TCMX/DCB (2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl) – Surrogate solution is prepared, with a final concentration of 0.5ug/mL, by diluting a stock solution (purchased from Restek #32000) in acetone. This solution is named "Pesticide Surrogate for Extractions 500ppb" and expires 6 months after the date it is made. Use 1.0mL of this solution per sample.

10.2.2 Pesticide Spiking Solution – A spiking solution, with a final concentration of 1ug/mL, is prepared by making a dilution of the Pesticide AB ICV Intermediate (this is made in-house by GC operators) in acetone. This solution is named "Pesticide AB LCS for Extractions 1.0ppm" and expires 2 weeks after the date it is made. Use 1.0mL of this solution per BS/BSD/MS/MSD. For 608 samples, 1 out of every 10 samples must be spiked.

10.2.3 PCB (Polychlorinated biphenyl) Spiking Solution – For all routine extractions, a mixture of 1016/1260 is prepared and used. The stock standards (purchased from Accustandard, or equivalent, 1016 #APP-9-158-10X and 1260 #C260S-H-10X) are diluted in acetone to a final concentration of 5ug/mL. This solution is named "PCB 1660 LCS for Extractions 5ppm" and expires 6 months after the date it is made. Use 1.0mL of this solution per BS/BS/MS/MSD. The Client, Laboratory Director and/or Project Manager will specify if another PCB mixture is necessary, such as 1221, 1232, 1242, 1248, 1254, 1262 or 1268.

10.2.4 Toxaphene spiking solution – This matrix spike is prepared from Toxaphene (Accustandard #P-0935-H, or equivalent) at a concentration 10ug/mL in acetone and expires 6 months from the date it is made. Add 1.0mL of this solution for each TCLP MS/MSD.

10.2.5 Chlordane spiking solution – This matrix spike is prepared from Chlordane (Ultra Scientific #EPA-1086, or equivalent) at a concentration of 100ug/mL. Add 1.0mL of this solution for each TCLP MS/MSD.

10.2.6 BNA Surrogate – The base neutral and acid surrogate are mixed together in one solution (purchased from NSI #WL-371-C at concentrations of 100-200ug/mL). The expiration for this standard is 6 months from the date opened or the vendor expiration date, whichever is sooner. Spike 0.5ml for 1ml final extract volume.

10.2.7 BNA LCS#1 Spiking Solution –BNA LCS#1. This spiking solution contains all the compounds that are normally calibrated by GC/MS. This solution, with a final concentration of 100ug/ml, is prepared in Methanol by making a dilution of stock purchased from reputable vendors (BNA LCS #1 spike kit #K-943 and 1-methylnapthalene #1288-01-08 are purchased from NSI). Spike 0.5ml for 1ml final extract volume.

10.2.8 BNA LCS#2 Spiking Solution –BNA LCS#2. This solution, with a final concentration of 100ug/ml, is prepared in Acetone by making a dilution of stock purchased from NSI #Q-6104-0. Spike 0.5ml for each BS/BSD/MSD/MSD.

10.2.9 Low-level BNA Spiking Solution – This solution is made using 1000µl of BNA LCS#1 and 1000µl BNA LCS#2 diluted to 10 ml with methanol. Spike 1.0ml for 1ml final extract volume.

10.2.10 TPH Surrogate - Surrogate (OTP) solution is prepared in acetone at a concentration of 20 µg/ml. Use 1 ml per sample.

10.2.11 TPH Spike (DRO or TNEPH) - A spiking solution is prepared at a concentration of 1000 µg/ml in acetone. Use 1 ml per BS/BSD/MS/MSD.

10.3 1mL, 2mL, 5mL, 10mL comparator vials prepared and documented as LIMS standards quarterly.

11. Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage. Aqueous samples have a hold time of 7 days from the date of sampling.

12. Quality Control

12.1. Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2. In order to spike all aroclors over a two year period, the following quarterly schedule will be followed with the indicated spike added in one batch for blank spike, matrix spike and matrix spike duplicate:

Aroclor 1221 Odd year first quarter (for example Jan/Feb/Mar 2017)
Aroclor 1232 Odd year second quarter
Aroclor 1242 Odd year third quarter
Aroclor 1248 Odd year fourth quarter
Aroclor 1254 Even year first quarter
Aroclor 1262 Even year second quarter
Aroclor 1268 Even year third or fourth quarter

13. Calibration and Standardization

Not Applicable to this SOP

14. Procedure

Note – the following procedure contains validated/permitted adjustments to the referenced method to compress extraction timeframes and reduce solvent volumes in order to reduce solvent usage. IDOC and LOD/LOQ validation documentation is available as provided to NJDEP NELAP assessors July 30, 2012 and approved by NJDEP NELAP assessors August 02, 2012. Documentation is located at "V:\QAQC\External Audits\2011\20110124_20110127 NJ NELAP\20120730_FinalResponse".

14.1 Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with group leader):

14.1.1 Each day the extractions group leader will generate a sample backlog using LIMS.

14.1.2 This backlog is used to determine extraction priorities based on hold times and due dates.

14.1.3 Samples requiring RUSH turnaround time may be logged in throughout the day, which will require immediate attention. Sample receiving personnel will generally communicate this need.

14.1.4 Samples are placed in LIMS "batches" based on parameter and extracted accordingly.

- A method blank (BLK) and blank spike (BS) must be processed with each set of samples.
- A matrix spike and matrix spike duplicate (MS/MSD), may be requested by client but additional volume must be provided. If limited extra volume is provided, check with client to determine if full volume MS is requested or ½ volume MS and MSD.
- If no MS/MSD volume is provided and 5 or more samples are included in the batch, a blank spike duplicate must also be processed.
- If a sample requires both Regular and Low-level BNA analysis, spikes must be prepared for both regular (BS1/MS1/MSD1) and low-level (BS2). Regular MS/MSD is sufficient

to cover both regular and low-level unless designated sample is specified for low-level only.

- If a sample requires both Pesticide and PCB analysis, spikes must be prepared for both pesticides (BS1/MS1/MSD1) and PCBs (BS2/MS2/MSD2).
- If a sample requires both DRO and ORO analysis, an ORO spike may be required (BS2/MS2/MSD2).
- If the sample is a TCLP, matrix spikes for toxaphene and chlordane are required in addition to MS/MSD for pesticide mix.
- TCLP blank fluid may be provided along with the extracted TCLP sample(s) and would be processed similar to the TCLPs using only 100mL diluted to 1000mL with reagent water.

14.2 Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are:

14.2.1 SLUDGE – measure 100mL with a graduated cylinder and dilute to 1000mL with reagent water.

14.2.2 TCLP EXTRACT - measure 100mL with a graduated cylinder and dilute to 1000mL with reagent water. Matrix spike and matrix spike duplicate using 100mLs should be set up at the same time.

14.2.3 BAD MATRIX – Discuss with project manager for possible dilution or centrifuge.

14.2.4 SPLP extract- use 1000mL measured with a graduated cylinder.

14.2.5 NPDES client requesting 608 or 625:

- Methods 608 and 625 require that there be a spike every ten samples. In addition, the sample must be extracted and concentrated in the same day.
- Samples for method 608/608.2 are checked by login to make sure the pH of the sample is in the range of 5.0-9.0. If the sample is not in this range, extraction personnel will be notified. At that time, it is the responsibility of the extraction lab to adjust the pH of the sample to the appropriate range (pH of 5-9) using NaOH solution or Sulfuric Acid, as necessary) or to extract the sample within 72 hours of sampling. If a pH adjustment is made, the details of the adjustment must be recorded on the sample COC and in LIMS.

14.2.6 ACID EXTRACT WITH BAD MATRIX - a cleanup step may be added when only acid fraction is required. Samples are taken to a high pH, extracted with 60mL methylene chloride one time as explained below in the BASE NEUTRAL EXTRACTION section. This extract is discarded. The samples are then taken to a low pH and extracted as an acid extraction and taken through concentration steps. Acid only must be documented in the LIMS.

NOTE! BENCH SHEET DOCUMENTATION IS ESSENTIAL FOR ALL NON-ROUTINE SITUATIONS!

14.3 Get out enough separatory funnels and glass beakers to extract the number of samples you have plus any additional spikes and a method blank.

14.4 Rinse separatory funnels and beakers with acetone. If preparing for low-level analysis, include a hexane rinse.

14.5 Use labels to properly identify BLK, BS, BSD, MS, MSD and samples for each separatory funnel and beaker.

14.6 Calibrate the 3200g balance with the rack and empty separatory funnel in place – tare then verify bracketing weights of 500g and 1500g (500g + 1000g weights combined) meet criteria as specified in QS08 (500g must read 490g - 510g and 1500g must read 1470g - 1530g).

14.7 To measure the sample volume – place the labeled separatory funnel in the rack with the stopcock closed, tare the balance, carefully transfer the sample to the separatory funnel and record the weight in milliliters. For the purposes of this test, 1g is assumed to be equivalent to 1mL.

14.8 Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record pH on the LIMS bench sheet.

14.8 Using the 1000mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1000mL of reagent water from the carboy and transfer it to the separatory funnel for the method blank, blank spike and blank spike duplicate (if applicable). Transfer sample to labeled separatory funnel that corresponds to the lab # on the sample bottle.

14.9 Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes and requires a change to the final volume of the extract. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

14.10 Generally, 0.5mL of BNA surrogate is added to each sample, spike, and blank with a syringe designated for BNA surrogate. For DRO, ORO, EPH, or Pest/PCB surrogate, use the designated syringe to spike the amount indicated on the bench sheet. **If a designated syringe is not available, rinse the syringe with the surrogate solvent 10x.** Document with initials/date on preparation timeline. A second extraction person or member of management (GL, LD, DQM) must verify that the correct surrogate spike/concentration has been added by adding initials/date to preparation timeline.
NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution. Alternately, measurement can be made from bottom of meniscus providing the associated bubble spans the entire syringe barrel.

14.11 For the sample in each analytical batch selected for spiking, use the 0.5mL glass syringes designated for BNA BS #1 and BNA BS #2 spike, to add 0.5mL of BNA spiking solutions. **For low level PAHs/BNAs use 1.0mL of the 1.0ppm Low-Level spiking solution**. For DRO, ORO, EPH, Pest or PCB, use the designated syringe to spike the amount indicated on the bench sheet. **If a designated syringe is not used, rinse 10x with the surrogate solvent.** Someone must verify that the spike has been added by initialing the LIMS bench sheet. For DOD QSM projects, all target compounds will be spiked into the BS and MS/MSD.
NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution. Alternately, measurement can be made from bottom of meniscus providing the associated bubble spans the entire syringe barrel.

14.8.1 If samples have suspended solids, heavy sediment or dark samples, notify the Project Manager to determine how the client wants to proceed.

14.8.2 If the client wants to proceed with centrifuging the sample, split the sample between 4 – 250 mL Teflon centrifuge containers. Place in large yellow cups in centrifuge buckets. Turn rpm to 1500-2000 rpm for 10 minutes. Be sure to let centrifuge reach max rpm to be certain centrifuge is well balanced. Re-balance, if necessary. Once done, decant aqueous portion into separatory funnel, leaving the solids portion in the bottom of the centrifuge tube. BENCH SHEET DOCUMENTATION IS ESSENTIAL.

14.13 ACID EXTRACTION: For Pest/PCB samples, skip pH adjustment. For BNA/PAH samples or DRO/EPH samples received at neutral pH, adjust the pH to between 1.0 and 2.0, using 2mL of 1:1 H2SO4. Add to each sample (unless received acidified), spike and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with wide-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H2SO4 solution in small increments, as required to attain the proper pH. DOCUMENT additional acid required.

14.14 Add 60mL of Methylene Chloride to each empty sample bottle (including MS/MSD) and to the BS and method blank funnels. Swirl the 60mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel.

14.12 Seal and shake the separatory funnel **vigorously** for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon funnels may be used and placed in the shaker apparatus with the stopcocks slightly open. When this apparatus is used, the shake must be for 5 minutes.

NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.

14.15 Allow the sample to sit for 5 minutes (more, if necessary) after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), use steps below in progression to attempt to break-down. If the layers are clearly separated, drain the solvent layer into the previously prepared glass beaker labeled with the laboratory sample ID.

NOTE: It is critical for spike/surrogate recoveries that only solvent be drained into the beaker. Make sure the sample is fully separated and carefully drain only solvent into the beaker, leaving 2-5ml in the funnel is acceptable at this step.

EMULSION - If sample exhibits an emulsion – use the following steps (in order from simplest to most complicated emulsion) to attempt to break-down the emulsion:

1. Tap: **U**se Teflon coated Stirbar retrieval stick or percussion hammer to tap the outside of the separatory funnel.
2. Swirl: Swirl the solution in the separatory funnel.
3. Stir: Use cleaned and acetone (hexane) rinsed Teflon coated Stirbar to vigorously stir the solution in the separatory funnel.
4. Intermediate separatory funnel: Acetone (and hexane for low-level) rinse a 250mL separatory funnel then slowly drain emulsion down the side of the intermediate separatory funnel. Once complete, emulsion should be greatly reduced and solvent can be drained to the beaker. If not, proceed to centrifuge.
5. Centrifuge: Drain any solvent that can be separated into the beaker then drain the remaining emulsion into cleaned, methanol-rinsed, labeled centrifuge bottles to be centrifuged. Label should indicate sample ID/acid fraction (or base if redirect from 14.17 below)
6. Split: Drain the base/neutral fraction and acid fractions into separate beakers to be taken through filtration separately with the acid fraction added to the base/neutral fraction in the Turbovap.

14.14 Following Steps 14.11 through 14.13, extract one more time with 60mL of methylene chloride. Combine the two solvent extracts into the same glass beaker. Proceed to Turbovap concentration for Pest/PCB/DRO/EPH samples.

14.15 BASE NEUTRAL EXTRACTION (for BNA/PAH samples, only): Adjust the pH to 11 or slightly greater, using 10N NaOH. Start by adding 7.0mL to each sample, spike, and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with wide-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more 10N NaOH in small increments, as required to attain the proper pH. Extraction at pH 11 **IS** necessary when doing low level PAHs/BNAs. If addition base is required to attain pH 11, DOCUMENT additional base required.

NOTE: This step is critical to the extraction procedure. Too much NaOH solution could cause you to lose certain Base Neutral compounds. Be careful on this step.

14.16 Add 40ml methylene chloride to each separatory funnel. Seal and shake the separatory funnel **vigorously** for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon funnels may be used and placed in the shaker apparatus with the stopcocks slightly open. When this apparatus is used, the shake must be for 5 minutes.

NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.

14.17 Allow the sample to sit for 5 minutes (more, if necessary) after it has been shaken. It will separate

into two layers with the solvent layer on the bottom. Drain the solvent layer into the previously prepared glass beaker labeled with the laboratory sample ID. If emulsion forms, see section 14.13 above for steps to break-down.

NOTE: It is critical for spike/surrogate recoveries that only solvent be drained into the beaker. Make sure the sample is fully separated and carefully drain only solvent into the beaker, leaving 2-5ml in the funnel is acceptable until the final drain is performed.

14.18 Extract one more time with 40mL of methylene chloride following Steps 14.16-14.17. Document with initials/date on preparation timeline.

NOTE: SW-846 extracts may be stored overnight at this point to allow for concentration the next day. Extracts for 608/625 must be concentrated the same day. Cover beakers with double layer of aluminum foil tightly crimped to the top of the beaker, place on a cart and place cart in the water walk-in. Document storage on the extraction timeline.

14.19 Prepare to dry the sample by setting up a ring stand with funnels. **HDPE funnels may be used for BNA/PAH/TPH extracts while glass funnels must be used for Pest/PCB extracts.** Place a small amount of glass wool in the bottom of the funnels (hammock style), add ~2" sodium sulfate (1/3 cup) to the funnel and rinse with 20-30 mL methylene chloride. Discard this rinse into the Chlorinated Waste container in the hood.

14.20 If the extract was drained into a centrifuge bottle, at this point you will need to take it to the centrifuge. Push the "ON" button to turn the centrifuge on. Be sure that the large holders are available for the 250-mL centrifuge bottles. The sample must always be balanced. If necessary use a dummy bottle making it similar weight using reagent water. Set the rpm at 2500. Close the lid and be sure to press it down until you hear it click. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Open lid and remove sample. The sample will usually be in two layers with the extract on the bottom.

14.21 **Note – this step should not be required for most samples and may negatively impact surrogate/spike recoveries.** Remove any water layer from the extract in the beaker or centrifuge bottle, by one of two methods. First, remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer in the sink with rinse. Use the smallest amount possible of Na2SO4 by sprinkling the top layer with Na2SO4 until it hardens, separates, and drops to the bottom.

14.22 Document filtration with initials/date on preparation timeline.

14.23 TURBO-VAP CONCENTRATION

14.23.1 Rinse a Turbo-Vap tube with methylene chloride and arrange it underneath a rinsed, funnel. For Pest and/or PCB, funnel must be glass. Pour the extract through the funnel so that it will collect in the tube. Rinse the beaker, which contained the solvent extract twice with 10 to 15 mL of methylene chloride and add each rinse to the funnel to complete the quantitative transfer. After all the extract has passed through the funnel, rinse the funnel with 10 to 15 mL of methylene chloride. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap. **For Pest and/or PCB extracts, add 40mL hexane to the tube prior to concentration.**

14.23.2 Record the numbers of the Turbo-Vap tubes on the LIMS bench sheet and place the tube in a metal holder.

14.23.3 Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 45°C (+/-5°C).

14.23.4 Place the glass evaporator tube (covered with foil, if high level of solvent) in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange

light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).

14.23.5 Prepare sample vial tray using labels printed off from LIMS that identify sample numbers, initial/final volumes, client, parameter, and date extracted.

14.23.6 When the beep sounds indicating the end of concentration, the extract will be at approximately one mL (half way up tip of tube). Remove the tube from the bath.

14.23.7 Transfer the extract to vial:

14.23.7.1 BNA/PAH/DRO/EPH:

14.23.7.1.1 Use a 9" Pasteur pipette to draw up the sample and transfer it to the 2-mL vial. THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!! A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure the vial is close to the top of the turbovap tube and that a drop will not form on the end and fall off.

14.23.7.1.2 Draw ~0.25 mL of methylene chloride into a 9" Pasteur pipette and add this aliquot to the turbo-vap. Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the appropriately labeled 2-mL vial. Add methylene chloride from a clean pipette and repeat the rinsing process until you have ~ 1 mL in the sample extract vial. Compare this volume to a 2-mL comparator vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. The methylene chloride rinse volume must be adjusted to achieve this final volume. Cover the extract with a Teflon-sealed screw cap.

14.23.7.2 Pest and/or PCB (see trouble shooting section if gel forms on PCBs):

14.23.7.2.1 Use a 9" Pasteur pipette to draw up the sample and transfer it to the 10-mL vial. THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!! A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off. If final volume is 10mL, one or two droppers of hexane may be added prior to transfer.

14.23.7.2.2 Draw ~0.25 mL of hexane into a 9" Pasteur pipette and add this aliquot to the turbo-vap. Draw the hexane into a pipette and rinse the sides of the tube several times. Transfer this rinse to the appropriately labeled 10-mL vial. Add hexane from a clean pipette and repeat the rinsing process until you have the final volume indicated on the bench sheet (2 mL - 10mL) in the sample extract vial. Compare this volume to the appropriate comparator vial to insure that you have not exceeded the final volume. The hexane rinse volume must be adjusted to achieve this final volume. Cover the extract with a Teflon-sealed screw cap.

14.23.8 If extract exceeds final volume, use disposable pipette to swirl extract down side of vial to evaporate or use nitrogen to gently evaporate with documentation.

14.23.9 Document completion with initials/date on preparation timeline.

14.24 The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the batch, the analyst initials, and the date and time the samples were placed into the refrigerator.

14.25 Transfer handwritten extraction details from bench sheet to LIMS and scan bench sheet for PDF archival in the LIMS**. It is essential that all difficulties encountered with a sample or QC be transferred to the LIMS.**

14.26 Difficulties with QC must be communicated directly to the analyst/group leader. **If there are any problems, it may be necessary to have the extracts screened immediately.**

15. Data Analysis and Calculations

Not Applicable to this SOP

16. Method Performance

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP QS03 for guidance.

17. Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

19. Corrective Actions and Out-of-Control Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20. Contingencies for Handling out of control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

21. Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

22. Equipment/ Instrument Maintenance

22.1 Replace separatory funnels as needed.
22.2 Replace turbo vap sensors as needed.
22.3 Drain, clean, refill turbovap water bath.

23. Computer hardware and software

Not applicable.

24. Troubleshooting

24.1. Water in Turbovap tube:
  24.1.1. Filter again through funnel with Na2SO4 into new turbo.
24.2. Sample cooks down too low in turbovap water bath
  24.2.1. Sample too dark
  24.2.2. Sensor malfunction (replacement)
24.3. Sample slow to cook down
  24.3.1. Low pressure in nitrogen tank
  24.3.2. Low water in turbovap water bath.
24.4. **For PCBs Only** – Some wastewater samples will form a gel like substance when the hexane is concentrated. Proceed with these samples as follows, if necessary:
  24.4.1. Add just enough methylene chloride to make the gel go back into solution
  24.4.2. Acid clean the extract and re-concentrate.

24.4.3. Exchange with hexane again

24.4.4. If gel forms again, add enough methylene chloride to get gel back into solution

24.4.5. Transfer to a suitable container and record the final volume on the label and on bench sheet.

24.4.6. **Make sure to note the difficulty and the percentage of methylene chloride in sample.**

25. References

25.1 Test Methods for Evaluating Solid Waste, SW-846, Third Edition

25.2 40 CFR, Method 608, 625.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1 Check-off list for separatory funnel extractions

26.1 Check-off list for separatory funnel extractions

General Flow - See SOP for details!

	Water Extraction Steps	BNA/PAH	Pest/PCB	DRO/ORO/EPH
1	Pre-rinse labware w/Acetone (add hexane for low-level)			
2	Retrieve samples			
3	Record container ID			
4	Measure Sample/Record			
5	Place sample in SepFunnel			
6	Check pH from 1L amber/record	*neutral*	*neutral*	*pH<2 or adjust*
7	Add MeCl2 to sample jar			
8	Add Spike/Surr to SepFunnel			
9	Add H2SO4 to SepFunnel		NA	*Unless rcvd pH<2*
10	Verify pH ≤2		NA	
11	Add MeCl2 to SepFunnel			
12	Shake (2m manual/5m shaker)			
13	Sit (5 minutes), Record emulsions			
14	Drain/Repeat 11-13/Drain*			
15	Add NaOH to SepFunnel		NA	NA
16	Verify pH ≥11		NA	NA
17	Add MeCl2 to SepFunnel		NA	NA
18	Shake (2m manual/5m shaker)		NA	NA
19	Sit (5 minutes), Record emulsions		NA	NA
20	Drain/Repeat 17-19/Drain*		NA	NA
21	Filter (glass/plastic funnel), Record TV	Plastic	Glass	Plastic
22	Concentrate w/water TV tubes , Record position	Foil Tubes!	Add Hexane!	

* = if emulsion, check SOP for options (Drain to intermediate SepFun for separation, centrifuge, swirl, vibrate with percussion hammer, stir with clean teflon stir rod). Maintaining separation is **critical** for spike and surrogate recoveries.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 343 REVISION #: 10 EFFECTIVE DATE: 20161121

BNA, PESTICIDE/PCB & TPH NON-AQUEOUS MATRIX (MICROWAVE EXTRACTION) USING SW-846 METHOD 3546

APPROVALS:

Lab Director: _____ Date: **20161121**

Data Quality Manager: _____ Date: **20161121**

Group Leader: _____ Date: **20161121**
Donald Hensel

Changes Summary

SOP343_R10_20161121_MW

- Section 10.5 added for comparator vials.
- Section 12.2 added to contain quarterly rotation of extra aroclors for BS/MS/MSD.

SOP343_R09_20160831_Micro

- Placement diagram added to appendix with reference in section 14.15
- Pre-rinse of syringes updated to indicate for non-designated syringes.
- Section 14.9 clarified to indicate QC is lined up prior to samples in bench sheet order.
- Updated 14.21 to reflect addition of 40mL hexane prior to pest/pcb concentration.
- Section 14 concentration steps updated to clarify.
- Section 14.23: added option to add two droppers of hexane to TV tube prior to transfer.

SOP343_R08_20151013_Micro

- Removed references to drying column, ring stand and clamps in section 9.
- Updated nitrogen reference to indicate liquid nitrogen.
- Preparation timeline instructions added to procedure.
- Overnight storage option added before concentration step.

SOP343_R07_20150922_Micro

- All references to supervisor updated to reflect group leader.
- Section 10 updated to reflect QS04.
- Section 14.13 and 14.20 updated for pest/pcb solvent.

Revision 06, 07/30/2104

- The SOP has been formatted to include all 26-elements required per the TNI 2009 (23)/DoD Version 5.0 (26) standards.
- Watermark update to include proprietary reference.
- Section 14.6.3 note updated to indicate documentation of clay sample on bench sheet.
- Sodium sulfate specifications in reagents section updated to indicate bulk sodium sulfate must be tested prior to use and cooled in desiccator after baking.
- Note added following section 14.8.
- Sections 22, 23, 24 and 25 populated/updated.

Revision 05, 07/26/2013

- SOP was reviewed by Jacque Galland and found to need no revisions.

Revision 04, 06/04/2012

- Procedure has been updated to allow cooling of the sealed microwave extracts in the walk-in cooler and update the drying procedure.

Revision 03, 03/20/2012

- Equipment and Supplies/Reagents and Standards sections have been updated.
- Procedure has been updated to reflect current spiking, extraction and concentration techniques. A complete review of this section is required

Revision 02, 11/17/2010

- SOP has been updated to reflect added volume correction for BNA surrogate

- Clarified addition of sodium sulfate to microwave tubes
- Corrected # of samples associated to each of the specified microwave methods and added the name of the method identified on the instrument.

Revision 01, 09/09/2010

- SOP has been updated to reflect the correct QS SOPs and include missing solvent/spike information.

Revision 00, 08/01/09

- Review of SOP indicated no changes were necessary
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

BNA, Pesticide/PCB & TPH NON-AQUEOUS MATRIX (Microwave Extraction) Using SW846 METHOD 3546

1. **Identification of the Method**
   SW846 Method 3546

2. **Application Matrix or Matrices**
   Solids.

3. **Limits of Detection and Quantitation**
   Not applicable to this SOP.

4. **Scope and Application, Including Parameters to be Analyzed**
   This SOP describes the extraction of BNAs, pesticides/PCBs, and TPHs from soil, sediment, sludges and waste solids by an automated method (3546).

5. **Summary of the Test Method**
   Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Microwave extractor for BNAs, Pesticides/PCBs, or TPHs. The extracts are then concentrated by a Turbo Vap concentrator.

6. **Definitions**
   Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7. **Interferences**
   7.1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
   7.2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
   7.3. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

8. **Safety**
   8.1. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab wide.
   8.2. Wear appropriate personal protection equipment when working with chemicals or samples.
   8.3. Use the lab hoods when working with solvents.
   8.4. Use caution when mixing strong acids or bases. Solutions will become extremely hot when mixing with water. Avoid splashing these solutions so they won't come in contact with the skin or eyes. If this happens, flush with lots of water. Contact your group leader if serious and medical attention is needed.
   8.5. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.
   8.6. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
   8.7. MSDS sheets are available from vendor websites for all reagents and standards which have been purchased. See your group leader, lab director or data quality manager if there are any difficulties in accessing these records.

9. Equipment and Supplies

9.1. Stainless Steel spatula
9.2. Microwave extractor unit with 40 position carousel, electronic components, and ample ventilation
9.3. Microwave extraction Teflon tubes, capacity approximately 75mL
9.4. Suitable Teflon-lined cap and screw-top lid
9.5. Vial – 2mL clear with Teflon-lined screw cap
9.6. Vial – 12mL clear with Teflon-lined screw cap
9.7. Syringe – 1mL, 500uL
9.8. Pasteur pipet – 9" length
9.9. Pasteur pipet bulb
9.10. Labels – Staples, Dymo or equivalent
9.11. Aluminum foil – heavy duty
9.12. Liquid Nitrogen tank – equipped with pressure regulator
9.13. TurboVap Concentrator with 200mL concentrator tubes
9.14. Teflon or glass funnels
9.15. Balance – capable of weighing to 0.1grams
9.16. Aluminum pie pans
9.17. Filter paper – 185mm – VWR #28455-164 or equivalent
9.18. Wooden tongue depressors
9.19. Glass Wool - Silane Treated (purchased from Supelco #2-0410 or equivalent).

10. Reagents and Standards

Quality Systems SOP QS04 "TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS" contains all default requirements for laboratory reagents and standards. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. All reagents and standards must be documented within the LIMS and labeled accordingly. The following information relates to the specific reagents and standards used for the performance of the method:

10.1. Methylene Chloride (Please read SOP – 336 before handling this solvent in our laboratory) (Dichloromethane) – suitable for spectrophotometry and gas chromatography (VWR# EM-DX0831-1 or equivalent)
10.2. Hexane – suitable for spectrophotometry and gas chromatography (VWR# EM-HX0296-1 or equivalent)
10.3. Sodium Sulfate (Na2SO4) – Pre-baked sodium sulfate Anhydrous, ACS Low Organic, Crystals purchased from Jost Chemical Co. (#2796) in 2.5 kg. glass jars. Fisher sodium sulfate anhydrous (S415-212, Granular 10-60mesh/certified ACS) in 2.5 kg. glass jars may also be used. **With SVOC testing prior to use,** Granular, anhydrous, trace pure 10-60 mesh may be purchased in bulk containers from Fisher # S415-10S (or equivalent). It must be placed in a Pyrex tray and heated at 400°C for 4 hrs minimum, removed and cooled in a dessicator then placed in a 2.5 kg glass amber jug and stored at room temperature.
10.4. Surrogate/Spike Solutions – Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes or if the initial concentration of stock is different than that listed below:
    10.4.1. **BNA Surrogate (100ug/mL)** – The base neutral and acid surrogates are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from Restek or equivalent reputable vendor). Spike 0.5mL of this solution per 15g of non-aqueous sample (BNA and Low-Level BNA/PAH).
    10.4.2. **BNA LCS #1 for Extractions 100ug/mL –** Contains kit from NSI (K943) which includes 10.0 mL of Acids Mix, 5.0 mL of Basic Extractables, 5.0 mL of Base Neutrals Mix, and 2.5 mL of Benzidines Mix. Also contains 1 mL of 1-methylnapthalene and an additional 5.0 mL of Acids Mix (C-498-5) brought to final volume of 50 mL with acetone or methanol. Spike 0.5 mL of this solution per 15g of non-aqueous sample. **(Low-level BNA/PAH spike is listed below).**
    10.4.3. **BNA LCS #2 for Extractions 100ug/mL** – Add 1.0 of Custom Mix #2 (NSI #8-6421-0) and

dilute to a final volume of 50 mL in acetone. Spike 0.5 mL of this solution per 15g of non-aqueous sample.

10.4.4. **Low Level BNA LCS 1ug/mL** – add 100µL of BNA #1 and 100µL of BNA LCS #2 to acetone and dilute to 10 mL in volumetric flask. Spike 1.0mL of 1.0 ug/mL Low-Level spiking solution.

Note: The BNA Spiking solutions contain all targets that are calibrated for GC/MS. DOD QSM requires all target analytes be spiked in the LCS and MS/MSD.

10.4.5. **TCMX/DCB** (2,4,5,6-Tetrachloro-meta-xylene/ Decachlorobiphenyl) Surrogate solution is prepared in acetone by diluting the stock purchased from a reputable vendor at 0.5 ug/mL. Spike 0.5mL of this solution per 15g of non-aqueous sample.

10.4.6. **PCB Spiking Solution –** Aroclor 1016/1260 or the PCB of choice (1242, 1248, 1254, or 1260 are the most common) is prepared in acetone at a concentration of 5.0ug/mL. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Spike 0.5mL per 15.0g of non-aqueous sample.

10.4.7. **Pesticide Spiking Solution –** A spiking solution is prepared at 1.0 ug/mL. Spike 0.5mL per 15g of non-aqueous sample.

10.4.8. **TPH Surrogate –** Surrogate solution is prepared in acetone by diluting stock ortho-terphenyl standard to a final concentration of 20 ug/mL. Spike 1mL per 15 grams of non-aqueous sample.

10.4.9. **TPH Spike (DRO, DRO/ORO or TNEPH) –** A spiking solution is prepared by extractions analyst that has a concentration of 1000 ug/mL in acetone. Spike 1mL per 15 grams of non-aqueous sample.

10.5 1mL, 2mL, 5mL, 10mL comparator vials prepared and documented as LIMS standard quarterly.

11. Sample Collection, Preservation, Shipment, and Storage

11.1. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a Teflon-lined cap.

11.2. Samples are preserved by cooling to 4°C.

11.3. Holding time is 14 days from collection date to extraction.

12. Quality Control

12.1. Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2. In order to spike all aroclors over a two year period, the following quarterly schedule will be followed with the indicated spike added in one batch for blank spike, matrix spike and matrix spike duplicate:

Aroclor 1221 Odd year first quarter (for example Jan/Feb/Mar 2017)
Aroclor 1232 Odd year second quarter
Aroclor 1242 Odd year third quarter
Aroclor 1248 Odd year fourth quarter
Aroclor 1254 Even year first quarter
Aroclor 1262 Even year second quarter
Aroclor 1268 Even year third or fourth quarter

13. Calibration and Standardization

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

14. Procedure

NOTE: All non-designated syringes should be rinsed with spike solution solvent 10x prior to use.

14.1. All soils have a 14-day holding time counted from the day they are sampled. Determine the samples necessary to extract using the following information. (DO NOT extract samples for which you have no information.):

14.1.1. Each day a backlog is generated in the LIMS providing all relevant sample information, including samples numbers and respective analysis required.

14.1.2. Samples requiring RUSH turnaround time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.

14.1.3. Check the backlog throughout the day to re-evaluate priority if needed.

14.2. Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your group leader to find out if a microwave extraction is truly necessary.

14.3. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples). Using the LIMS, create a batch of samples and print off sample labels. The LIMS will create a unique batch sequence number.

14.4. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan. **Document on bench sheet.**

14.5. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

14.6. It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

14.6.1. The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.

14.6.2. Two quarters should then be mixed to form halves.

14.6.3. The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container. Document on bench sheet if this approach is used by noting that the sample was clay.

14.7. Place a labeled aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the balance calibration logbook. Using a tongue depressor, transfer the appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}, of a representative sample to the nearest 0.1 gram. Normally 15g sample weights are used. Record this amount on your bench sheet. **For MS/MSD spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc. unless a client sample has been specified.** Record balance ID on bench sheet. Document measurement with initials/date on preparation timeline.

14.8. Add ~ 15 grams of sodium sulfate to the aluminum pie pan. Using a tongue depressor, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the tongue depressor from the mixed sample, leave behind all the sample possible. Continue to weigh up the remaining samples. For the method blank and LCS, pour up approximately 15g of sodium sulfate in microwave tube. The matrix used for the method blank and LCS

must be free of the analytes of interest and processed through the same analytical steps as the samples.

NOTE: If samples do not become suitably free-flowing with the addition of sodium sulfate, they may be left to air dry for up to 4 hours with documentation on the bench sheet.

14.9. Carefully transfer samples to microwave tubes. Make sure samples are loaded in the rack in the order of the bench sheet with the exception that QC are loaded in front of samples.

14.10. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by watching and signing off on bench sheet.

14.11. Surrogate: BNA/PAH - using the 0.5-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. Pest/PCBs - using the 1.0-mL glass syringe marked Pesticide surrogate, add 0.5 mL of surrogate to each sample, blank and spike. TPH – use the appropriate 1.0-mL glass syringe to add 1.0 mL of the appropriate surrogate to each sample, blank and spike.

14.12. Spiking: For the BNA sample in each analytical batch selected for spiking, use the 0.5-mL glass syringes marked BNA LCS #1 and BNA LCS #2 to add 0.5 mL Spiking solution. (For low level BNAs/PAHs use 1.0 ml of the 1.0µg/mL Low-Level spiking solution.)

14.12.1. **For Pest/PCB samples**, determine if the sample will require a Pesticide Spike and/or a PCB Spike. Proceed as follows:

14.12.2. **Pesticide and PCB** - set up two LCS's – one for Pesticide getting an AB MIX spike and one for PCB, which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.

14.12.3. **Pesticide only –** To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.

14.12.4. **PCB only -** To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB.

14.12.5. **For TPH** - To the sample in each analytical batch selected for spiking, add 1mL of the appropriate spiking solution (i.e. DRO or TNEPH or MAEPH) using a 1.0 mL glass syringe dedicated to that spike.

14.12.6. **Document spiking with initials/date on preparation timeline. Document certification of correct spiking with second checker initials/date on preparation timeline.**

14.13. Solvent: Add 30mL methylene chloride for BNA/PAH/TPH/Pest/PCB extractions.

14.14. Place a Teflon cap and Teflon screw top on the Teflon microwave tube. Using the cap tightener station, tighten the caps then invert sample to mix and check for leaks in cap.

14.15. Place microwave tubes in microwave carousel making sure they are in order and spaced evenly throughout the carousel to insure proper heating while in microwave. See appendix for recommended distribution from manufacturer user guidelines.

14.16. Place microwave carousel in microwave making sure the carousel is properly lined up with the turning mechanism.

14.17. Choose saved program option based on total number of samples to extract and begin process by pressing the start button. The program is set to EPA method 3546 specifications. Note the method used by circling either 800W or 1600W on the bench sheet.

For 1-16 samples (800W):
Method: 3546 800W 100% 16-Express
Max power: 800W
Ramp time: 15:00
Control temperature (in Celsius): 110
Hold time: 10:00
Cool down: 5:00

For 17-40 samples (1600W):
Method: 3546 1600W 100% 40-Express
Max power: 1600W
Ramp time: 15:00
Control temperature (in Celsius): 110
Hold time: 10:00
Cool down: 5:00

14.18. Allow samples to cool in the carousel for an additional 30 minutes or place microwave rack in the walk-in cooler to cool quickly before attempting to handle the extracts. Document preparation with initials/date on preparation timeline. Note – extracts may be held overnight at this step.

14.19. Prepare to dry the sample by setting up a rack with funnels. Place folded filter paper and/or a small amount of glass wool in the bottom of the funnels, add ~2" sodium sulfate to the funnel and rinse with 20-30 mL methylene chloride. Discard this rinse into the Chlorinated Waste container in the hood.

14.20. Transfer the extract to a pre-rinsed turbo vap tube by passing through prepared drying funnels. After pouring the extract into the turbo, rinse the microwave tube 3 times with the extraction solvent and transfer the rinsate to the turbo. Finally, rinse the funnel with an adequate amount of the extraction solvent using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest. Document filtration with initials/date on preparation timeline.

14.21. **For Pest/PCB extracts**, add 40mLs of hexane to each turbovap tube before proceeding to concentration step.

14.22. Turbo Vap Concentration: Now concentrate the extract to 1.0mL using the turbovap concentrator.

14.22.1. Adjust the pressure of nitrogen gas tank to 50 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be at 45°C (+/-5°C). The pressure target range for the concentration is about 20 psi to provide proper vortex without splashing.

14.22.2. Place the turbo vap tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).

14.22.3. Prepare sample vial tray using labels printed off from LIMS that identify sample numbers, initial/final volumes, client, parameter, and date extracted.

14.22.4. When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.

14.23 **Transfer the Extracts to final vial:**

14.23.1 For BNA/PAH/DRO/EPH:

14.23.1.1 Use a 9" Pasteur pipette to draw up the sample and transfer it to the 2-mL vial. THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!! A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure the vial is close to the top of the turbovap tube and that a drop will not form on the end and fall off.

14.23.1.2 Draw ~0.25 mL of methylene chloride into a 9" Pasteur pipette and add this aliquot to the turbo-vap. Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the appropriately labeled 2-mL vial. Add methylene chloride from a clean pipette and repeat the rinsing process until you have ~ 1 mL in the sample extract vial. Compare this volume to a 2-mL comparator vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. The methylene chloride rinse volume must be adjusted to achieve this final volume. Cover the extract with a Teflon-sealed screw cap.

14.23.2 For Pest and/or PCB (see trouble shooting section if gel forms on PCBs):

14.23.2.1 Use a 9" Pasteur pipette to draw up the sample and transfer it to the 10-mL vial. THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!! A single drop

represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off. If final volume is 10mL, one or two droppers of hexane may be added prior to transfer.

14.23.2.2 Draw ~0.25 mL of hexane into a 9" Pasteur pipette and add this aliquot to the turbo-vap. Draw the hexane into a pipette and rinse the sides of the tube several times. Transfer this rinse to the appropriately labeled 10-mL vial. Add hexane from a clean pipette and repeat the rinsing process until you have the final volume indicated on the bench sheet (2 mL - 10mL) in the sample extract vial. Compare this volume to the appropriate comparator vial to insure that you have not exceeded the final volume. The hexane rinse volume must be adjusted to achieve this final volume. Cover the extract with a Teflon-sealed screw cap.

14.23.3 If extract exceeds final volume, use disposable pipette to swirl extract down side of vial to evaporate or use nitrogen to gently evaporate with documentation.

14.23.4 Document completion with initials/date on preparation timeline.

14.24 The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the batch, the analyst initials, and the date and time the samples were placed into the refrigerator.

14.25 Transfer handwritten extraction details from bench sheet to LIMS and scan bench sheet for PDF archival in the LIMS**. It is essential that all difficulties encountered with a sample or QC be transferred to the LIMS.**

14.26 Difficulties with QC must be communicated directly to the analyst/group leader. **If there are any problems, it may be necessary to have the extracts screened immediately.**

NOTE: If the final extract is yellow or dark in color or the matrix is oily and viscous, further cleanup may be desired. Discuss cleanup possibilities with the group leader.

15. Data Analysis and Calculations

Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

16. Method Performance

16.1. Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP QS08 for guidance.

16.2. Refer to SOP-201, SOP-211 and SOP-219 for method performance**.**

17. Pollution Prevention

17.1. Please see Waste Disposal SOP QS14 for the proper disposal of waste generated from this area.

17.2. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria.

19. Corrective Actions and Out-of-Control Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

20. Contingencies for Handling Out-of-Control or Unacceptable Data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data.

21. Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

22. Equipment / Instrument Maintenance

22.1 Replace separatory funnels, as needed.
22.2 Replace turbovap sensors, as needed.
22.3 Drain, clean, refill turbovap water bath.

23. Computer Hardware and Software

Not applicable.

24. Troubleshooting

24.1. Water in Turbo tube:
  24.1.1. Filter through funnel with Na2SO4 into new turbo.
24.2. Sample cooks down too low in cooker
  24.2.1. Sample too dark
  24.2.2. Sensor malfunction (replacement)
24.3. Sample slow to cook down
  24.3.1. Low nitrogen in tank or pressure
  24.3.2. Low water in turbovap water bath.
  24.3.3. **For PCBs Only** – Some wastewater samples will form a gel like substance when the hexane is concentrated. Proceed with these samples as follows, if necessary:
    24.3.3.1. Add just enough methylene chloride to make the gel go back into solution
    24.3.3.2. Acid clean the extract and re-concentrate.
    24.3.3.3. Exchange with hexane again
    24.3.3.4. If gel forms again, add enough methylene chloride to get gel back into solution
    24.3.3.5. Transfer to a suitable container and record the final volume on the label and on bench sheet.
    24.3.3.6. **Make sure to note the difficulty and the percentage of methylene chloride in sample.**

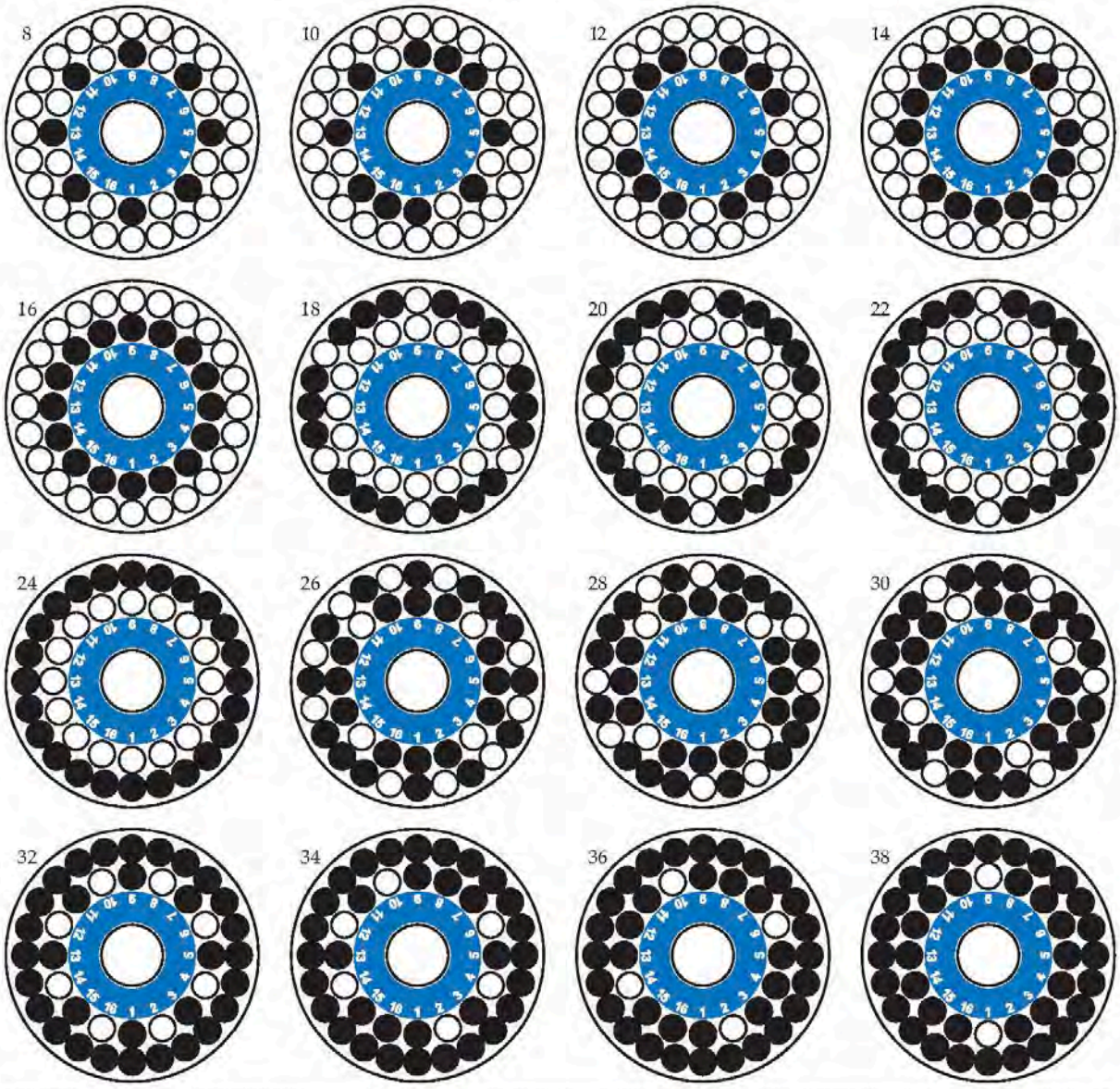
25. References

25.1. EPA Methods SW-846, Method 3546

26. Tables, Diagrams, Flowcharts, and Validation Data

Appendix – Recommended Distribution

Recommended distribution of MARSXpress vessels when turntable is less than capacity



Incorrect distribution: What not to do

Title: Determination of Low-Level Volatile Organics in Ambient / Indoor Whole Air Samples Using GC/MS-Scan Mode

[Methods EPA TO-14A and EPA TO-15]

Approvals (Signature/Date):

7-22-15	7/22/15
Koroush Vaziri Date Department Manager	Joe Schairer Date Health & Safety Manager / Coordinator
7/29/2015	7.24.15
Lisa Stafford Date Quality Assurance Manager	Crystal Pollock Laboratory Director

Facility Distribution No. Uncontrolled **Distributed To:** Sacramento Bids Folder

1. SCOPE AND APPLICATION

1.1. This standard operating procedure (SOP) is applicable to the analysis of low-level volatile organic compounds (VOCs), having molecular weight in the general range of 40-200 g/mol and vapor pressure greater than 0.10 Torr at 25°C and 760 mm Hg in ambient air, by gas chromatography/mass spectroscopy (GC/MS) technique. This SOP is based on the EPA TO-14A/TO-15 method specifications and is applicable to various air matrices that include ambient air and indoor air.

1.2. Target analytes and reporting limits with this SOP are listed in Attachment 1. Reporting limits will be proportionately higher for samples that require dilution.

1.3. On occasion, clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the laboratory's Quality Assurance Manual (WS-QAM) in the section that discusses Service to the Client

1.4. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. An air sample and internal standards (IS) are metered by a mass flow controller onto a cryogenically cooled trap (using either a Microscale Purge and Trap or a Cold Trap Dehydration technique described in Section 11). The trap is heated and the contents are transferred to a Tenax trap to remove water. The Tenax trap is heated and the analytes are transferred to a cryofocusing module. The cryofocuser is heated to transfer the analytes to the gas chromatographic column for separation and detection by a mass spectrometer operated in the Scan Mode.

3. DEFINITIONS

3.1. Note that "must" and "shall" in this SOP denote required activities.

3.2. Air Sample Bag: Commonly referred to as FlexFilm or Tedlar bag, in 1.0-L or 3.0-L volumes, that is constructed of proprietary material (e.g., SKC or ESS).

Note: Use of air sample bags as sample collection media constitutes a modification to the method (see Section 17.5) that is defined in the final report.

3.3. Part per billion volume to volume (ppbv or ppb v/v): Concentration expressed as part of gaseous (vapor) volume of pure target compound contained in a billion part of gaseous volume of sample.

Note: This reporting unit is NOT equivalent to the common ppb unit used in soil or water analysis.

3.4. Particulate Filter: A cylindrical stainless steel fitting containing a fritted metal disc, which is connected to the valve of a passivated canister or to the vacuum flow regulator (VFR), to prevent particulate matter from entering and damaging the passivated canister or VFR.

3.5. Passivated canister: Commonly referred to as SUMMA canister, SilcoCan, or T.O.-Can in 1.0-L, 1.8-L, 6-L, 15-L, or 33-L volumes.

3.5.1. SUMMA canister: A spherical stainless steel container, of which the interior has been specially treated by a process (SUMMA passivation), that renders all surfaces inert to VOCs.

3.5.2. SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most VOCs.

3.5.3. T.O.-Can: A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek using a proprietary electropolishing process and is extensively cleaned using an ultrasonic method that ensures a high-quality, passivated surface that maintains the stability of VOCs during storage.

3.6. Standard molar volume = 24.45 L/mol at standard conditions (i.e., room temperature of 25°C and standard pressure of 1 atmosphere).

3.7. Standard pressure = 1.0 atmosphere or 14.6 pounds per square inch absolute (psia) or 0 inches of mercury or 0 pound per square inch gauge (psig), based on laboratory elevation and average barometric pressure.

Note: Full vacuum (0 psia) = -30 inches of mercury vacuum

3.8. QC section: Surrogates: Organic compounds which are similar to the target analytes in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Although not required by the method, each client and QC sample is spiked with surrogate standards via the analytical trap. Surrogates are used to monitor method performance with each sample. Surrogates are only reported to the client by request.

3.9. Vacuum Flow Regulator: A device which, when connected to a passivated canister, regulates the flow of sample into the passivated canister so that a timed, representative sample can be obtained (also called a composite sample), as opposed to an unregulated, instantaneous sample (grab sample).

3.10. Vacuum/Pressure Gauge: Device used to measure the vacuum or pressure in a passivated canister. Units of measure range from -30 to 0 inches of mercury (for vacuum) to 0 to 30 psig (for positive pressure). All pressure units are converted to psia (psig + 14.7 = psia).

3.11. Further definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

4. INTERFERENCES

4.1. Gas regulators are cleaned by the manufacturer using Freon 113, a target analyte in this SOP. Before using Ultra High Purity Nitrogen (UHP N2), hydrocarbon-free air, IS mix, or target compound standard mix cylinders, each regulator should be purged with the appropriate gas.

4.2. Contamination may occur in the sampling system if passivated canisters are not properly cleaned prior to use. Passivated canisters shall not be used for the collection of samples until a batch blank analysis indicates that no target compounds are present above the RL, or a level previously agreed upon between the laboratory and the client. When more stringent canister cleaning acceptance criteria are warranted based on project-specific or regulatory requirements, and then the more stringent criteria must be used. Further information regarding the cleaning and certification of passivated canisters may be found in TestAmerica SOP WS-QA-0032. All other sampling equipment including pumps, flow controllers, and filters must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

4.2.1. Passivated canisters may be batch-certified or individually-certified, depending on client request.

4.2.2. Passivated canisters will be certified-clean down to the MDL of the target analytes of interest if sample results need to be evaluated down to those limits. However, the laboratory must be provided advanced notification of the requirement.

4.2.2.1. Common laboratory contaminants like Acetone and Methylene chloride may be present above the MDL. In this instance, client approval must be received prior to sending out these passivated canisters.

4.3. Carry-over may occur when samples with high levels of contaminants are analyzed. The sample immediately following a high-level sample shall be re-analyzed if carry-over is suspected.

4.4. Air sample bags may contain low levels of target analytes.

4.5. Only compounds having both similar mass spectrum and GC retention time (RT) would be expected to interfere in the method. This situation most commonly occurs with structural isomers.

4.6. Large concentrations of water, Methane, or Carbon dioxide may limit the size of the sample aliquot that can be effectively cryo-trapped. This may elevate the RLs for samples of this type.

4.7. Matrix interferences may be caused by non-target contaminants that are present in the sample. The extent of matrix interference will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Pressurized gas equipment is used in this procedure. Be sure all valves and gauges are operating properly and that no equipment is over-pressurized. After changing cylinders, check all gas line connectors for leaks, with soapy water. Release of high pressure gas can cause rapid suffocation.

5.1.2. This analysis may require transfer of the sample from an air sample bag to a canister (Section 11.2). Because of the flexible nature of the air sample bag, insertion of the syringe needle into the bag may cause the septa to flex or the shaft of the syringe needle may flex within the septa. This may allow sample to escape along the shaft of the needle. All samples being manually removed from an air sample bag and transferred into a canister must be handled inside a fume hood with chemical protective gloves, lab coat, and safety glasses.

5.1.3. When pressurizing canisters or changing cylinders, face shield must be worn over safety glasses.

5.1.3.1. Passivated canisters should never be pressurized over 40 psig.

5.1.4. Pressurized gas cylinders must be securely retained. The use of a face shield is required when changing regulators.

5.1.5. Air sample bags should not be pressurized, as seam splitting will result.

5.1.6. The preparation of standards and reagents will be performed in a fume hood with the sash set at the level indicated on the side of the hood.

5.1.7. Both the GC and the MS contain elevated temperature zones. These zones must be cooled prior to an analyst or technician working on the instrument.

5.1.7.1. Temperature-appropriate gloves must be worn when working with hot or cold items.

5.1.8. Latex and vinyl gloves provide no protection against organic solvents. Nitrile or similar gloves must be used.

5.1.9. The effluents from the sample splitters for the GC and the roughing pumps for the MS must be vented to a fume hood or at a minimum, must pass through a charcoal filter.

5.1.10. The MS is under deep vacuum and must be brought to atmospheric pressure before working on the source.

5.1.11. Due to high voltage risk, power to the GC and/or MS must be turned off or disconnected before work can be done on the instrument.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Helium	Simple Asphyxiant	NA – Keep oxygen levels at 19.5%	Oxygen Deficient atmosphere may cause headaches, ringing in ears, dizziness, drowsiness, unconsciousness, nausea, vomiting and depression of all the senses.
Liquid Nitrogen	Simple Asphyxiant Cryogenic liquid	NA – Keep oxygen levels at 19.5%	Oxygen Deficient atmosphere may cause headaches, ringing in ears, dizziness, drowsiness, unconsciousness, nausea, vomiting and depression of all the senses. Contact with skin may cause frostbite-changes in skin color to white or grayish-yellow.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Instrumentation

6.1.1. Gas chromatograph – capable of sub-ambient temperature programming and electronic pressure control (Hewlett Packard 6890 or equivalent).

6.1.2. Mass-selective detector – equipped with computer and appropriate software (Hewlett Packard 5973/5975 or equivalent with Chemstation data acquisition software).

6.1.3. Sample concentrator – equipped with a cryogenic trap and appropriate systems for the control of moisture (Entech 7100 or equivalent).

6.1.4. Chrom version 2.1 data processing software.

6.2. Supplies

6.2.1. Chromatographic grade stainless steel or nickel tubing and stainless steel plumbing fittings.

6.2.2. Chromatographic column – Rtx-Volatiles, 0.32 mm ID, 1.5 μm df, 60 m length, methyl polysilicate liquid phase (Restek Corporation or equivalent).

6.2.3. Transducer and process meter capable of measuring 0 psia to 50 psia, for preparing standards (Ashcroft Digital Vacuum/Pressure Gauge or equivalent).

6.2.3.1. The process meter must be calibrated quarterly, at a minimum, against the master gauge.

6.2.4. Pressure regulators for carrier gas and standards – 2-stage, stainless steel diaphragm (single stage acceptable for standards).

6.2.5. Stainless steel vacuum/pressure gauge capable of measuring from -30 inches of mercury to 40 psig (Span Instruments or equivalent).

6.2.6. Air sample bags and passivated canisters used for the preparation of standards and the dilution of samples.

6.2.7. Screen can for preparation of method blanks. This is a cleaned canister certified to be free of analytes at levels greater than or equal to the MDL or levels specified by client or program requirements, whichever is appropriate to the samples being analyzed.

6.2.8. Gas-tight syringes of various sizes (Hamilton or equivalent).

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Reagents

7.1.1. UHP N_2 – used for Method Blanks and preparing dilutions of samples and standards

7.1.2. UHP Helium – used as the gas chromatograph carrier gas

7.1.3. Pressurized air source for Entech 7100 heater gas

7.1.4. Liquid N_2

7.1.5. Distilled or NanoPure water

7.2. Standards

7.2.1. Gas calibration stock standards containing the target compounds, at a nominal concentration of 1 part per million volume/volume (ppmv or ppm v/v), are purchased from NIST-approved vendors or prepared from neat in passivated canisters. Suppliers are required to provide certification of the analyte concentrations.

7.2.2. IS and surrogate stock standard mix (see Attachments 5 and 6, respectively), at a concentration of 300 ppbv, are purchased from NIST-approved vendors. Suppliers are required to provide certification of the analyte concentrations.

7.2.2.1. The surrogate mix is also used to tune the mass spectrometer.

7.3. Standard Preparation

Static dilutions and other standard preparation activities are performed in accordance with TestAmerica SOP WS-QA-0017.

7.3.1. Static dilutions of the stock standard gas mixtures are made in 6- or 15-L passivated canisters to create working standards. A high precision vacuum gauge is flushed with UHP N_2 and attached to the top valve of a clean, evacuated passivated canister, and the absolute pressure is recorded.

7.3.1.1. Distilled or NanoPure water (50 µL) is added to calibration standards prior to mixing.

7.3.1.2. The IS mix does not contain water.

7.3.2. Depending on the concentration of each stock standard gas mixture, a particular pressure of each is added to the passivated canister to achieve the

desired concentration in the working standard.

7.3.2.1. As an example, the daily working standard, at a nominal concentration of 10 ppbv, is created by adding 5.0 psia of the 100 ppbv standard mix and adding UHP N_2 to the passivated canister to achieve a final pressure of 50 psia.

7.3.2.2. Care should be taken to flush each regulator and transfer line with standard prior to transfer to the passivated canister.

7.3.3. Detailed preparation of each standard is recorded in the Laboratory Information Management System (LIMS) reagent module.

7.3.4. Other preparation techniques may be used to obtain the desired standard concentration, provided these techniques do not compromise the integrity of the standards used, and that the details of the preparation are properly documented in the LIMS.

7.3.5. Working standards are valid for a period of 30 days, after which fresh standards are prepared.

7.4. Expiration dates for source standards and reagents are based on vendor specification. If no vendor expiration date is assigned, the laboratory assigns an expiration date of two years from the date of receipt. Refer to TestAmerica SOP WS-QA-0017 for further information on standards and expiration dates. Expiration dates must be documented on the gas cylinders.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample container, preservation techniques, and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Passivated Canister	2000 mL	None	30 days	EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air

Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Passivated Canister	2000 mL	None	30 days	Advisory – Active Soil Gas Investigations, April, 2012 (DTSC, LARWQCB, and SFRWQCB)
Air Sample Bag	500 mL	None	72 hours	N/A

8.2. Passivated canisters used for sample collection must be certified clean (< RL or program specific limit). Canisters are cleaned in accordance with TestAmerica SOP WS-QA-0032. Filters (e.g., 7-micron or 2-micron) should be placed on the inlet of the canister to protect the valve from particulates.

8.3. Samples should be shipped at room temperature, in packaging suitable to prevent puncture and exposure to light.

8.4. If air sample bags are to be shipped by aircraft, they should be filled about 75% full to allow for expansion during shipment.

8.5. The pressure of a passivated canister should be recorded before and after sample collection in the field to help detect canister leakage and document proper sampling.

8.6. Samples are stored at room temperature.

8.7. Samples should be protected from extreme temperatures.

9. QUALITY CONTROL

9.1. Batch

A batch is defined as a set of up to 20 client samples of the same matrix processed using the same procedures and the same lot(s) of reagents within the same time period. A batch must contain a Laboratory Control Sample (LCS) and a Method Blank, but they do not count towards the maximum 20 samples in a batch.

9.1.1. In some cases, an LCS Duplicate may be required by a client or program to provide batch precision. In that instance, the acceptance criteria and corrective actions appropriate for the LCS are applied.

9.1.2. Rerun of the same client sample is counted as part of the 20 in a batch (i.e., a client sample analyzed twice in the same batch must be counted as two client samples).

9.1.3. Field quality control (QC) samples (e.g., trip blanks, equipment blanks, and field duplicates) count as client samples; therefore, they add to the batch

count.

9.1.4. Laboratory QC samples, including duplicates and clean canister blanks (screen cans), do not add to the batch count.

9.1.5. The batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

9.1.6. Refer to the laboratory's QC Program document (WS-PQA-003) for further details of the batch definition.

9.2. Laboratory Control Sample– For each batch, an LCS must be analyzed. The LCS is analyzed after the calibration standards and before the Method Blank and client samples. The LCS is spiked with the target analytes in Attachment 1, from which a sub-set may be reported..

9.2.1. Refer to the QC Program document (WS-PQA-003) for details on the requirements for LCS composition.

9.2.2. If any analyte is outside established recovery and precision control limits, or any surrogate is outside the established recovery control limits, the system is out of control and corrective action must occur. Corrective action typically includes reanalysis of the batch.

9.2.3. If the batch is not reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable reasons for not reanalyzing include evaluation of sporadic marginal exceedances (ME), or an elevated recovery (indicating a high bias) with samples non-detect for the failing analyte. Refer to the QC program document (WS-PQA-003) for more details regarding evaluation and acceptance of out of control LCS data.

9.2.4. Exceedance outside the ME limits require corrective action, regardless of whether the associated result is positive or ND. See Section 9.2.6.

9.2.4.1. For failures that exceeded the ME at the high end, the ND analyte may be flagged and reported only if the program or project-specific requirements allow.

9.2.4.2. For all other ND results that failed the ME requirements, the client must approve to flag and report the data since the nonconformance does not meet the NELAC or TNI Standard.

9.2.5. All data reported with out of control LCS values will be flagged by the LIMS. Analysts shall also file a nonconformance memo (NCM) within the LIMS

detailing why the data is reported, and any corrective actions performed.

9.2.6. Corrective actions to occur before batch reanalysis:

9.2.6.1. Evaluate the analytical run for errors and anomalies. Re-analyze the LCS.

9.2.6.2. Consult the troubleshooting guidelines in Section 11.8. Evaluate the instrument status and perform maintenance.

9.2.6.3. Re-analyze the continuing calibration verification (CCV) standard and LCS, or recalibrate.

9.2.7. Current LCS control limits are stored in the LIMS. Control and ME limits are subject to change based on periodic evaluation of LCS control charts by Quality Assurance personnel, in accordance with the procedures detailed in policy WS-PQA-003.

9.3. Method Blank

9.3.1. For each batch, an acceptable Method Blank must be analyzed. The Method Blank is analyzed after the calibration standards and LCS and prior to client samples. The Method Blank is a 6-L screen can (Section 6.2.7) humidified with 50 µL of Distilled or NanoPure water and then pressurized to 40 psia with UHP N_2.

9.3.2. If a method blank is requested to be analyzed using an air sample bag similar to which samples are collected the client is required to submit a bag for that purpose. The laboratory does not maintain an inventory of air sample bags, therefore, Method blanks in bags is by client request only.

9.3.3. The Method Blank must <u>not</u> contain any analyte of interest ≥ RL (or ≥1/2 RL, as dictated by the QSM or project-specific requirements), except common laboratory contaminants, (Section 9.3.2). Otherwise, the Method Blank is further evaluated and corrective actions must be performed, as stated below. See troubleshooting guidelines in Section 11.8.

9.3.3.1. Re-analyze the Method Blank once to determine if an error or an anomaly occurred during sample analysis. If the re-analysis is acceptable, then the Method Blank can be considered in control.

9.3.3.2. If there are no results greater than the RL in the samples or if the results in the samples are greater than 10X the Method Blank level, the data may be reported with qualifiers. In this case, the elevated Method Blank result is not believed to impact data quality. The anomaly must be reported in an NCM.

9.3.3.3. If there are results greater than the RL in the samples and if these results are less than 10X the Method Blank level, the samples must be re-analyzed.

9.3.3.3.1. If re-analysis is not possible due to limited sample volume or other constraints, the Method Blank is reported and all associated samples are flagged. The client must be consulted. The anomaly must be reported in an NCM. The laboratory Project Manager (PM) must record the client's decision in the NCM.

9.3.4. If the analyte detected in the Method Blank is a common laboratory contaminant (Methylene chloride, Acetone, and 2-Butanone), the data may be reported with qualifiers if the concentration of the analyte is less than 5X times the RL. Otherwise, corrective actions, as stated in Section 9.3.1, must be performed. The anomaly must be reported in an NCM.

9.3.5. If surrogates are a project-specific requirement, then the Method Blank must have acceptable surrogate recoveries. If surrogate recoveries are unacceptable, the data must be evaluated to determine if the Method Blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-analysis of the Method Blank and affected samples must be performed.

9.4. Surrogate Standards.

Surrogates are not a method requirement. The laboratory routinely adds surrogates to all QC and client samples via the analytical trap and will report these results only if defined in a project/contract or at client's request. The surrogate compounds used in this SOP are listed in Attachment 6.

Surrogate recoveries in QC and client samples may be assessed to ensure that recoveries are within laboratory control limits. If any surrogate is outside these limits and if surrogates are a project-specific requirement, the following corrective actions must be performed:

- Check all calculations for error.
- Ensure that instrument performance is acceptable. See troubleshooting guidelines in Section 11.8.
- Re-analyze the QC/client sample.

9.4.1. It is only necessary to re-analyze a client sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst has reason to believe that the repeated out of control results are due to problems other than matrix effect.

9.4.2. If re-analysis is not possible due to limited sample volume or other constraints, the surrogates are reported with a flag. The client must be consulted. The anomaly must be reported in an NCM. The nature of the matrix interference must be noted in the NCM. The PM must record the client's decision in the NCM.

9.4.3. Current surrogate control limits are stored in the LIMS and are subject to change based on periodic evaluation of surrogate control charts by Quality Assurance personnel, in accordance with the procedures detailed in policy WS-PQA-003.

9.5. Internal standard (IS)

IS compounds are added to each calibration standard, LCS, Method Blank, and client sample via the analytical trap. IS compounds are monitored for each shift by comparing the IS areas and retention times in each client and QC sample against those of the associated CCV standard. The IS compounds used in this SOP are listed in Attachment 5.

9.5.1. IS evaluation criteria for the initial calibration (ICAL) may be found in Section 10.3.8.

9.5.2. For all other QC and client samples, IS areas are considered acceptable if they fall between 60% and 140% (for TO-15) or –50% and 200% (for TO-14A Low-level) of the CCV IS areas. The RTs are considered acceptable if they fall within ±20 seconds (±0.33 minutes) of the IS RT of the associated CCV.

9.5.3. Any QC or client sample exceeding the acceptance criteria above must be re-analyzed. If the IS fails upon re-analysis, the failure must be documented in an NCM. All corrective actions performed must also be documented in the NCM.

9.6. Sample Duplicate Analysis

9.6.1. A client sample duplicate is analyzed and reported with the batch when requested by the client.

9.6.2. The acceptance criteria for the duplicate analysis is an RPD $\leq$ 25 for target analytes detected >5X the RL. No criteria are established for duplicate results <5X the RL.

9.7. Calibration standards and other QC samples (e.g., BFB, LCS, Method Blank, etc.) may not be analyzed more than twice without documented corrective action. If the initial run fails acceptance criteria, re-inject the calibration standard or QC sample. If second run passes, analysis may proceed. Otherwise, conduct instrument maintenance or perform corrective action. Completely document failure, corrective action performed,

and return to control in the Instrument Maintenance Logbook. Section 11.8 lists troubleshooting guidelines.

9.7.1. Refer to WS-PQA-021 for corrective actions specific to DoD programs.

10. CALIBRATION

For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.1. Initial/Daily Tuning of the Instrument

10.1.1. After a successful autotune per manufacturer's recommendations, each instrument is manually tuned using perfluorotributylamine (PFTBA) so that mass-to-charge ratio (m/z) 69 is 100%, m/z 131 is approximately 34%, and m/z 219 is approximately 36%. The width and axis parameters are set using the routines in the software. This initial tune should remain stable for extended periods of time, and retuning with PFTBA should not be necessary every day.

10.1.2. At the beginning of each 24-hour shift, prior to any analytical runs, it must be verified that the GC/MS system meets acceptable tune performance criteria. This is done through the analysis of 50 ng of 4-bromofluorobenzene (BFB); the acceptance criteria are listed in Attachment 3 (for TO-14A) or in Attachment 4 (for TO-15).

10.1.2.1. Using the BFB method in the cryotrap software, the IS mixture volume required is 50 ng of BFB on column (currently 24 mL of 300 ppbv standard).

10.1.2.2. An alternate way to load 50ng BFB is to use 50mL of a 20ppbv IS/SURR mix and 20mL of a 3000ppbv IS/SURR mix.

10.1.2.3. The mass spectrum of BFB must be acquired using the peak apex and the scans immediately before and after the apex and averaged. A background subtraction is applied using a scan prior to the elution of BFB.

10.1.3. If any of the key ions fail the ion abundance criteria listed in the attachments, the system is considered out of tune and any subsequent sample/standard analysis shall be considered unacceptable. The BFB must be re-analyzed and re-evaluated. If the BFB continues to fail, the GC/MS system must be evaluated. See troubleshooting guidelines in Section 11.8.

10.1.3.1. Adjustments to the mass axis calibration, the electron multiplier voltage, or other tune parameters may be required. All parameter changes must be recorded in the Instrument Maintenance Logbook.

10.1.4. BFB tunes may be analyzed more frequently depending on documented client requirements.

10.2. Initial Calibration

10.2.1. Instruments are calibrated at initial setup and as needed thereafter, and at least annually.

10.2.2. An ICAL curve consisting of a minimum of five points is analyzed to determine the linear working range of the analytical system for each compound. An average response factor (RF), or sometimes called the relative response factor (RRF), and the percent relative standard deviation (%RSD) are calculated for each target analyte using the equations in Section 12.5.

10.2.3. The ICAL is considered acceptable if the calculated %RSD for the RF (or RRF) for each analyte not listed in Section 10.3.4 is <30, with at most two exceptions up to a limit of 40%.

10.2.4. Any four of the following analytes, which are not listed as target analytes in the published methods and are considered poor performers, may have a %RSD limit up to 55:

1,1,2-Trichloro-1,2,2-trifluoroethane	Dichlorodifluoromethane
1,2,3-Trichlorobenzene	Naphthalene
2-Hexanone	Propene
Acetone	Tetrahydrofuran
alpha-Methylstyrene	

10.2.5. Linear calibration using least squares regression may be used with the appropriate number of calibration points. Details regarding its use and the calculations involved may be found in TestAmerica Corporate QA SOP CA-Q-S-005. This Corporate SOP must be consulted prior to using this curve. The analyst must read and understand the topics regarding Forcing Through Zero and Curve Weighting.

10.2.5.1. The coefficient of determination for a line fit must be greater than or equal to 0.990

10.2.5.2. The absolute value of the intercept (printed on the calibration curve plot in Chrom) should be less than ½ the reporting limit (-RL ≤ intercept ≤ +RL). If the intercept is outside the limits, any values

below the reporting limit must be evaluated to ensure that false positives or false negatives are not being reported.

10.2.6. If the ICAL acceptance criteria are not met, corrective action (documented in the Instrument Maintenance Logbook) must be performed and a new ICAL generated. See troubleshooting guidelines in Section 11.8.

10.2.7. The nominal concentrations of the ICAL standards are typically 0.20, 0.30, 0.40, 0.80, 2.0, 4.0, 8.0, 20, and 40 ppbv, but these may vary depending on the certified mix used to prepare the standards or the volume trapped. The low standard must be at or below the RL. The standards are analyzed by preparing stock standards at the required concentration or by varying the trapped volume of the working standards from the default volume of 250 mL. For example, the 0.20, 0.40, and 0.80 ppbv standards are analyzed by trapping 62.5, 125, and 250 mL, respectively, of a 0.80 ppbv working standard.

10.2.7.1. At times, the default volume may be changed, depending on required instrument sensitivity for a project.

10.2.8. Internal Standards in the ICAL

10.2.8.1. The IS response at each calibration level must fall between 60% and 140% (for TO-15) or –50% and 200% (for TO-14A) of the IS response in the mid-point calibration standard. The mid-point standard is normally 8ppbv.

10.2.8.2. The RT shift for each of the IS at each calibration level must be within ±20 seconds (0.33 minutes) of the RT of the IS in the mid-point calibration standard.

10.2.8.3. Any calibration level exceeding the above acceptance criteria must be re-analyzed.

10.2.9. The analyst may elect to drop points from the calibration curve to improve subsequent quantitation, in accordance with Policy CA-T-P-002, Selection of Calibration Points.

10.3. Essential components for ICAL evaluation

10.3.1. The signal-to-noise (S/N) in the low point of the ICAL must be ≥ 2.5:1 for the compound to be considered valid at that level. Evaluate the EICP for the S/N determination.

10.3.2. Qualitative compound identification criteria must be met for all calibration levels; (primary and secondary ions >10% must be present in each standard level. Consult the technical director or QA for clarification.

10.3.3. Check retention times for isomers to ensure correct peak assignment.

10.3.4. Check co-eluting peaks for the proper reference spectrum.

10.3.5. The 're-fit' or 'read-back' for each point of the calibration curve is evaluated for the % error. The guideline is ≤ 20% for each level of the curve. This can be observed in the calibration summary in Chrom for each compound.

10.4. Initial Calibration Verification (ICV)

10.4.1. Each new ICAL must be verified using a second-source standard.

10.4.2. Since the regulatory agencies have not provided guidance on second-source verification, the ICV is considered acceptable if the %Recovery for each analyte not listed in Section 10.4.3 is 70–130%, with at most two exceptions up to a limit of 60–140%.

10.4.3. Any four of the following analytes, which are not listed as target analytes in the published methods and are considered poor performers, may have a %Recovery of 45–155:

1,1,2-Trichloro-1,2,2-trifluoroethane	Dichlorodifluoromethane
1,2,3-Trichlorobenzene	Naphthalene
2-Hexanone	Propene
Acetone	Tetrahydrofuran
alpha-Methylstyrene	

10.4.4. For samples analyzed in the same batch where the ICAL and ICV were analyzed, every time an allowed exception to the 70–130% Recovery ICV criteria is used, the sample results (whether J-value, ND, or positive) for the affected analyte must be flagged and explained in an NCM.

10.4.5. If the ICV acceptance criteria are not met, the following corrective actions must be performed. See troubleshooting guidelines in Section 11.8.

10.4.5.1. Rerun the second-source check standard.

10.4.5.2. Re-prepare or acquire a new standard.

10.4.5.3. Evaluate instrument conditions.

10.4.5.4. Regenerate a new ICAL.

10.4.6. Due to the limited availability of second-source manufacturers for the air standard mixes and some neat compounds, the following options may be considered as second-source:

10.4.6.1. Different certified lot from the same manufacturer.

10.4.6.2. Same certified lot from the same manufacturer but the stock standard used for the second-source is prepared by an analyst other than the one who prepared the stock standard used for the ICAL. This option is only allowed if the program or project-specific requirements allow.

10.5. Continuing Calibration Verification

10.5.1. Unless the QC batch follows a new ICAL and an ICV, for every 24 hours of operation, a CCV standard is analyzed to verify the ICAL average RF. The %D of the CCV RF from the ICAL average RF is calculated for each target analyte using the equation in Section 12.5.4.

10.5.2. The CCV is considered acceptable if the %D for each analyte not listed in Section 10.5.3 is ±30.

10.5.3. Any four of the following analytes, which are not listed as target analytes in the published methods and are considered poor performers, may have a %D ±55:

1,1,2-Trichloro-1,2,2-trifluoroethane	Dichlorodifluoromethane
1,2,3-Trichlorobenzene	Naphthalene
2-Hexanone	Propene
Acetone	Tetrahydrofuran
alpha-Methylstyrene	

10.5.4. Any time an allowed exception to the ±30%D CCV criteria is used, the sample results (whether J-value, ND, or positive) for the affected analyte must be flagged and explained in an NCM.

10.5.5. The following NELAC requirements (NELAC Quality Systems, June 5, 2003, 5.5.5.10e, page 217 of 324) and TNI Standard EL-V1M4-2009, Quality Systems for Chemical Testing, Section 1.7.2e, page 92) apply when the CCV acceptance criteria are not met.

10.5.5.1. If routine corrective action procedures fail to produce a second consecutive (immediate) CCV within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive CCVs, or a new ICAL must be generated.

10.5.5.2. When the acceptance criteria for an analyte in the CCV are exceeded high (i.e., high bias), and the analyte was ND in the associated samples, the ND analyte may be reported with a flag. An NCM must be generated and the high bias discussed in the case narrative of the final report.

10.5.5.3. When the acceptance criteria for an analyte in the CCV are exceeded high (i.e., high bias), and the analyte was detected at a positive hit in the sample, the sample must be re-analyzed after a passing CCV or after a new ICAL has been established, evaluated, and accepted.

10.5.5.4. When the acceptance criteria for an analyte in the CCV are exceeded low (i.e., low bias), sample results may be reported if they exceed a maximum regulatory limit/decision level (if known). Otherwise, the affected samples must be re-analyzed after a passing CCV or after a new ICAL has been established, evaluated, and accepted. An NCM must be generated and the low bias discussed in the case narrative of the final report.

10.5.6. CCVs may be analyzed more frequently depending on documented client requirements.

10.5.7. CCVs must be monitored on a routine basis using the control chart program (refer to SOP WS-QA-0035) to evaluate the data for trends. The frequency is dependent on the frequency of the analysis. In the LIMS control chart module, select the CCV chart. Once the chart has been evaluated, click "Save Log" to save the chart evaluation in the LIMS.

10.6. Calibration standards and other QC samples (e.g., BFB, LCS, Method Blank, etc.) may not be analyzed more than twice without documented corrective action. If the initial run fails acceptance criteria, re-inject the calibration standard or QC sample. If second run passes, analysis may proceed. Otherwise, conduct instrument maintenance or perform corrective action. Completely document failure, corrective action performed, and return to control in the Instrument Maintenance Logbook. Section 11.9 lists troubleshooting guidelines.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Preparation – Air Bag Samples

11.2.1. For air sample bag samples, the air sample bag is checked for damage and is analyzed as received.

11.2.2. Air sample bags are analyzed directly from the bag or transferred to an evacuated can within 72 hours of sampling.

11.2.2.1. If the entire bag is transferred to a can, the bag needle valve septum is pierced with a needle attached to a 1-L or a 6-L evacuated can, and the entire contents transferred.

11.2.2.2. If only a portion of the bag is to be transferred, a measured aliquot of the bag is transferred via a clean syringe through a septum attached to the top of a 1-L or a 6-L humidified can.

11.2.3. After transfer, the can is then pressurized to a positive pressure and the pressure is recorded.

11.3. Sample Preparation – Canister Samples

11.3.1. For passivated canister samples, the initial pressure is checked by attaching the process meter line connector to the passivated canister. The process meter line connector must be rinsed before use, with the pressurization gas (UHP N_2 or UHP He, if requested) by physically holding it against the gas outlet and flushing for 10 seconds, as this avoids possible carry-over concerns from high concentration samples. With the process meter line connector attached, the passivated canister valve is opened briefly and the pressure is recorded. If the pressure is less than 6 psig, the passivated canister is pressurized to 10 psig with the pressurization gas. The initial and final pressure must be recorded in the Canister Pressurization Logbook (see Attachment 10) and in the individual Canister Field Data Record.

11.3.1.1. Samples received above ambient pressure (14.6 psia) do not require pressurization unless additional volume is needed to perform multiple analyses. If samples are received below ambient pressure, UHP N_2 should be added. The default final pressure is 24 to 26 psia, however, the final pressure should be above ambient but not more than three times the initial pressure.

11.3.2. When the passivated canister vacuum/pressure is increased, a dilution factor (DF) is calculated and is applied to results. The calculation is provided in Section 12.5.5.

11.3.3. Passivated canisters received as trip blanks (without sample collected) are pressurized to 24-26 psia. These samples are considered to have a DF =1.0.

11.4. Sample Screening

Samples are screened to check for contamination before analysis or if suspected to contain significant contamination, using a GC/MS. Screening is performed to determine a proper dilution or the optimum volume of sample for the calibrated range, and to prevent overloading the analytical instrument. The screening instrument is generally calibrated at a single-point for common analytes of interest. The sample screen data are stored in TALS within the Chrom module.

11.5. Tuning

Refer to section 10.2 for details regarding instrument tuning.

11.6. Calibration

11.6.1. Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

11.6.2. Refer to Sections 10.3 through 10.5 for details regarding instrument calibration.

11.7. Sample Analysis

11.7.1. The calibration standards and the sample QC are analyzed in the same manner as client samples. After the calibration standards are analyzed and evaluated (Sections 10.3 through 10.5), the LCS is analyzed and evaluated (Section 9.2), and then the Method Blank is analyzed and evaluated (see Section 9.3), all prior to client sample analysis.

11.7.2. Each passivated canister is attached to the autosampler and recorded in the instrument sequence. A sequence is created in the GC/MS software to prepare the instrument for data acquisition. The sequence information controls the GC/MS method, data file creation, sample parameters, and report output. A second sequence must be created in the autosampler control software to control the sampling process such as line position, sample volume, trap temperatures, flow rates, and times. The sequence is verified by another analyst. This analyst verifies the autosampler sequence, port position, chem. station sequence, and Chrom worklist. The analyst annotates in the instrument maintenance log if the sequence has been verified.

11.7.3. The valves are opened on all passivated canisters and the autosampler and GC/MS sequences are started.

11.7.4. The pressure DF must be compensated for by trapping more than the default volume. For example, a sample received at 12.0 psia and pressurized to 24.6 psia has a pressure DF of 2.05. If the default volume is 250 mL, then 510 mL should be trapped. The recorded volume is rounded to three significant figures.

11.7.5. A sample that requires only a small dilution can be analyzed by trapping a volume less than the standard volume. The minimum volumes that can be trapped are 10 mL for an Entech 7200 and 20 mL for an Entech 7100. The maximum volume that can be trapped is three times the default volume. Larger dilutions may require analysis using different methodology.

11.7.6. Sample dilutions may also be performed by transferring an aliquot of the sample (originally from either an air sample bag or a passivated canister) into an air sample bag and filling it up to volume or by removing an appropriate amount of the original pressurized sample from a passivated canister and then re-pressurizing to approximately 24 to 26 psia or no more than 3x the initial pressure with UHP N_2. Serial dilutions can be performed, as necessary.

11.7.6.1. Air sample bag dilutions can be used for reporting analytes that exceeded calibration range in the original analysis, but not as a reportable analysis for all other analytes due to the low-level contamination inherent in the air sample bag.

11.7.6.2. When air sample bag dilutions are performed, the syringe used for transferring sample must be fitted with a valve (e.g., Luer-Lok) that allows the syringe contents to be isolated from the room air during transfer between containers.

11.7.7. For routine analysis, either the Microscale Purge and Trap or the Cold Trap Dehydration technique is used.

11.7.7.1. For Microscale Purge and Trap technique, the autosampler will follow the sequence of events below (parameters may be modified based on instrument performance):

11.7.7.1.1. Glass Bead trap (Module 1) is cooled to -150°C

11.7.7.1.2. Internal standard is trapped

11.7.7.1.3. Sample is trapped

11.7.7.1.4. Tenax trap (Module 2) is cooled to -15°C and the Glass Bead trap is heated to 10°C. Any remaining sample is transferred to Module 2 by passing UHP Helium through Module 1. Conditions may vary based on

instrument performance. This step is designed to remove water from the sample.

11.7.7.1.5. When GC is ready, the cryofocuser (Module 3) is cooled to –170°C. Module 2 is heated to 200°C. The sample is transferred to Module 3.

11.7.7.1.6. Module 3 is heated and the GC/MS column flow is routed through Module 3 to inject the sample and begin the run.

11.7.7.1.7. The system is pre-flushed with the next sample and the system is baked to limit carry-over.

11.7.7.2. For Cold Trap Dehydration technique, the autosampler will follow the sequence of events below (parameters may be modified based on instrument performance):

11.7.7.2.1. Blank (empty) trap (Module 1) is cooled to -40°C and the Tenax trap (Module 2) is cooled to -40°C.

11.7.7.2.2. Internal standard is trapped.

11.7.7.2.3. Sample is trapped.

11.7.7.2.4. Blank trap is heated to 10°C. Any remaining sample is transferred to Module 2 by passing UHP Helium through Module 1. Conditions may vary based on instrument performance. This step is designed to remove water from the sample.

11.7.7.2.5. When GC is ready, the cryofocuser (Module 3) is cooled to –155°C. Module 2 is heated to 200°C. The sample is transferred to Module 3.

11.7.7.2.6. Module 3 is heated and the GC/MS column flow is routed through Module 3 to inject the sample and begin the run.

11.7.7.2.7. The system is pre-flushed with the next sample and the system is baked to limit carry-over.

11.7.8. Upon completion of the analytical sequence, the Entech software generates a QA/QC report that records data from the sampling event (i.e. actual volume trapped, temperature at the time of trapping, sample pressure, etc.).

11.8. Troubleshooting Guidelines

11.8.1. Many problems encountered during analysis are due to low standard pressures or carrier/detector gas supply issues. Always confirm that adequate pressure remains in the standards and that the instrument gas supplies are sufficient before working on the instrument hardware.

11.8.2. Low response – typically caused by leaking sample lines or valves or contaminated/dirty sources. Instrument software can perform automated leak checks of the system. Specific components can be checked by isolating the component in question from the system (disconnect and cap or plug the ends) and then performing a leak test using a pressure gauge and passivated canister at positive pressure. Leaking components will not hold pressure when the passivated canister is closed. Low internal standard areas may be caused by degradation of the MS performance and increasing the electron multiplier (EM) voltage may solve this concern.

11.8.3. Baseline noise – check for supply gas contamination and leaking fittings. Carrier gas filters may need to be changed, including the pencil filters inside the GC. Sample carry-over or contamination may also be an issue and baking the system while flushing sample lines will remove most carry-over. A dirty source or leaking MS may also cause issues. The use of automated leak check routines in the MS software can indicate if a leak is present. Source-cleaning should be performed according to the manufacturer's instructions.

11.8.4. Tune issues – if an instrument will not pass tune the first step is to perform a mass axis calibration and peak-width adjustment. If the failure is due to ratios of ions with large differences, the tune parameters should be adjusted to achieve the desired ratios. The final corrective action is to clean the source according to the manufacturer's instructions.

11.8.5. Instrument issues – if data loss or error messages are encountered, consult the instrument troubleshooting guidance found in the operator's manual. The manual is in the help section of the GC software.

11.9. Maintenance or Repair of Analytical Instruments or Support Equipment

11.9.1. When analytical instruments or support equipment require repair or maintenance, they shall be taken out of operation or otherwise isolated, and tagged as 'out-of-service' until such a time as the repairs or maintenance have been made and the instrument or support equipment can be demonstrated as operational by calibration and/or verification or other tests to demonstrate acceptable performance. Details on the tag-out procedures to be followed may be found in the section of the QAM that discusses Equipment and Calibrations.

11.9.2. A new ICAL must be generated following major maintenance such as

changing the column, cleaning or repairing the source, replacing filaments, changing electronics, replacing the multiplier or changing the concentrator.

11.9.3. Minor maintenance includes cleaning the injector port, replacing filters, changing the pump oil, autotuning, switching filaments (instrument contains two filaments under vacuum), replacing the syringe or injector tower, changing/refilling the calibration vial, changing seals and o-rings, ballasting pump, replacing fuses, replacing roughing pumps or transfer lines.

11.9.4. Schedule for routine maintenance of analytical instruments may be found in Attachment 9.

11.9.5. All maintenance or repair must be documented in the Instrument Maintenance Logbook.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Analyses

Two criteria must be satisfied to verify positive identification:

12.1.1. Elution of sample component at the same GC relative or absolute RT as those of the standard component.

12.1.1.1. The sample component relative retention time (RRT) must compare within ±0.06 RRT units of the RRT of the standard component.

12.1.1.2. As an option, RT must compare within 0.33 minutes of the standard component absolute RT. For reference, the RT standard must be run within the same 24-hour shift as the sample.

12.1.2. Correspondence of the sample component and the standard component mass spectra.

12.1.2.1. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

12.1.2.2. The relative intensities of ions must agree within ±30% between the standard reference and sample spectra. For example, for ions with ratio of 50% in the reference spectra, the corresponding sample ratio must be between 20 and 80%.

12.1.2.3. Standard reference mass spectra must be obtained on each individual GC/MS system.

12.1.3. If an analyte cannot be verified by all of the criteria in the above sections, but in the technical judgment of the analyst the identification is correct, then the analyte may be reported

12.1.3.1. Technical judgment may be based on whether a compound is present when co-elution occurs and a determination is made based on retention time and mass spectrum.

12.1.3.2. The difference in the spectra and the reason for the decision to report results must be explained in an NCM.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analysis being conducted. The following sections identify the guidelines for making tentative identification:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2. Relative intensities of the major ions should agree within ±30%.

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.2.1.6. Only peaks having a total ion current greater than 10% of the nearest eluting IS total ion current will be evaluated for reporting.

12.2.2. TICs will be given general names consisting of major functional groups and number of carbon atoms unless an RT reference is available.

12.2.3. When TICs are requested to be reported using specific compound names, the following procedure must be followed:

12.2.3.1. Choose characterized ions of the specific compounds from the mass spectrum.

12.2.3.2. Search ions from expected RT range or entire RT range if the RT of the specific compound is unknown or uncertain.

12.2.4. Semi-quantitative results will be calculated for TICs using total ion current areas and assuming an RRF = 1.0.

12.2.5. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the analyst assign a tentative identification.

12.3. Quantitative Analysis

12.3.1. When an analyte has been identified, the quantification of that analyte will be based on the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. Quantitation will take place using the IS technique.

12.3.2. A sample must be analyzed and reported at a dilution if one or more target analytes have an on-column amount above the upper calibration level. Dilutions are acceptable if at least one of the following criteria are met:

12.3.2.1. Any target analyte in the diluted sample is at or above the mid-point calibration standard (e.g., 8 ppbv on-column if 8 ppbv is the mid-point).

12.3.2.2. The peak height of any non-target analyte in the diluted sample exceeds the largest peak height of the highest calibration standard.

12.3.2.3. A heavy hydrocarbon matrix in the diluted sample raises the baseline two times that of the relative IS.

12.3.3. Analyte quantitation must be performed from the ICAL response and not from the CCV response. Test results must be qualified in reports when analyte quantitation is based on the CCV at client's request. This request must also be documented in an NCM and reported in the case narrative of the final report.

12.4. All manual or re-integration of chromatograms must be documented in accordance with TestAmerica Corporate SOP CA-Q-S-002. Documentation includes, at a minimum, before and after copies of the chromatograms with a reference to the reason for re-integration, dated, and initialed. All manual integrations must undergo a secondary-level review.

12.5. Calculations

12.5.1. Calculation for RPD

$$RPD = \frac{Value\ A - Value\ B}{Average\ of\ Values} X\ 100$$

12.5.2. Calculation for RRF

$$RRF = \frac{Area\ cpd\ in\ Std.}{Area\ I.S.} X \frac{Conc.\ I.S.}{Conc.\ cpd\ in\ Std.}$$

The area of the primary quantitation ion is used in the calculation.
I.S. = Internal Standard

12.5.3. Calculation for %RSD

$$\%RSD = \frac{Std.\ Dev.\ of\ RRFs}{Mean\ of\ RRFs} X 100$$

12.5.4. Calculation for %D

$$\%D = \frac{Average\ RRF\ from\ ICAL - RRF\ CCV.}{Average\ RRF\ from\ ICAL} X 100$$

12.5.5. Calculation for pressure DF

$$DF = \frac{Y_a}{X_a}$$

Where:

X_a = absolute canister pressure before dilution (initial pressure)
Y_a = absolute canister pressure after dilution (final pressure)

12.5.6. Calculation for Determining the Concentration of Compounds
The data system automatically quantitates the sample results based on a standard sample size of 250 mL. The default result units are in ppbv. If a sample size other than 250 mL was used or a canister sample was pressurized, the result must be adjusted as shown below:

$$Final\ result\ \ ppbv = raw\ result\ ppbv\ X \frac{250\ mL}{sample\ vol\ injected} X \frac{final\ psia}{initial\ psia}$$

Where:

$$raw result\ ppbv = \frac{Area\ cpd\ in\ sample}{Area\ I.S. in\ sample} X \frac{Conc. I.S.}{RRF\ \ ICAL.}$$

Note: The area of the primary quantitation ion is used in the calculation.
I.S. = Internal Standard

12.5.7. Calculation for Percent Recovery (%Rec)

$$\%\,\mathrm{Rec} = \frac{Amount\ cpd.\ recovered}{Amount\ cpd.\ spiked}\ X\ 100$$

12.5.8. Standard reporting units are ppbv (also ppb v/v). If results are to be reported in ng/L or ug/m^3, use the following equation:

$$result\ ppbv\ X\ \frac{Molecular\ weight\ of\ cpd}{24.45} = results\ ng/L\ \ or\ ug/m^3$$

Note: 24.45 is the molar volume of ideal gas in liters at 25°C and 1 atmosphere.

12.6. Estimates of uncertainty are based upon LCS historical control limits, and are provided on request only.

12.7. "J" values (results below the RL but above the MDL) are reported on request only.

12.8. No conversion of the analytical results to standard conditions is made.

12.9. The number of significant figures to be used when reporting sample and QC results are defined in QA Policy WS-PQA-004.

12.10. Technical Data Review

Technical data review is performed in accordance with Policy WS-PQA-012, and is documented utilizing review checklists. Examples of appropriate review checklists are found in Attachments 7 and 8.

12.10.1. One aspect of technical review is to ensure that the test instructions are clear, and that all project-specific requirements have been understood and followed. If directions to the analyst are not clear, the analyst must consult the Department Manager or the appropriate PM, who must clarify the instructions.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix

B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. This requires analysis of QC check samples containing all of the standard analytes for the method. It may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Expired standards will be part of the Lab Pack waste stream. They will be identified as expired, stored under the manufacturer's recommended conditions, and then packed for disposal as outlined in SOP WS-EHS-0001.

15.1.1. Gas standards in non-returnable, non-refillable cylinders, such as Scotty® Transportables, are slowly vented in the fume hood when empty. They are then turned over to the hazardous materials specialist, who ensures that they are damaged (e.g., a hole is drilled into the cylinders) so they cannot be reused. The damaged cylinders are then either recycled or scrapped.

15.1.2. Gas standards in returnable, refillable cylinders are returned to the manufacturer.

15.2. Air sample bags are slashed in a hood and then placed into an orange high VOA lab trash can. When the can is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, 2nd edition, January 1999

16.1.1. Compendium Method TO-14A, Determination of Volatile Organic Compounds (VOCs) in Ambient Air using Specially Prepared Canisters With Subsequent Analysis Gas Chromatographic Analysis

16.1.2. 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)

16.2. TestAmerica Sacramento QAM, current revision

16.3. TestAmerica Sacramento SOP WS-QA-0032, Cleaning, Certification, and Preparation of Sampling Equipment, current revision

16.4. TestAmerica Corporate Environmental Health and Safety Manual CW-E-M-001, current revision

16.5. Advisory – Active Soil Gas Investigations, April 2012 (CAEPA, DTSC, LARWQCB, and SFRWQCB)

16.6. EPA/600/R-04/003, 2003 NELAC Standard, June 5, 2003

16.7. The NELAC Institute (TNI) Standard 2009, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis

16.8. TestAmerica Sacramento SOP WS-PQA-001, Quality Control Program, current revision

16.9. TestAmerica Corporate SOP CA-Q-S-005, Calibration Curves (General), current revision

16.10. TestAmerica Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices, current revision

16.11. TestAmerica Sacramento Policy WS-PQA-004, Rounding and Significant Figures, current revision

16.12. TestAmerica Corporate SOP CA-Q-S-006, Detection Limits, current revision

16.13. TestAmerica Sacramento SOP WS-EHS-0001, Sample and Chemical Waste Characterization, Collection, Storage and Disposal, current revision

17. METHOD MODIFICATIONS

17.1. Method TO-15 describes the use of zero air when making standards. TestAmerica Sacramento uses UHP nitrogen in the standards preparation.

17.2. UHP Nitrogen is used for dilution/pressurization purposes.

17.3. Method TO-15 describes canisters leak check being performed using pressure. TestAmerica performs the canisters leak check using vacuum unless specified by the project.

17.4. Method TO-15 presents criteria for evaluating canister cleanliness as less than 0.2 ppbv of any target VOC. TestAmerica Sacramento works with clients to ensure that the canisters meet their data quality objectives, by providing canisters evaluated to the reporting limit or method detection limit as required by their program.

17.5. Method TO-14A describes an inlet system that uses a vacuum to pull the sample through the trap. TestAmerica Sacramento optionally uses the pressure of the sample canister to drive the sample through the trap.

17.6. Method TO-14A describes the use of a Nafion dryer to remove excess moisture from air matrices. TestAmerica Sacramento does not use a Nafion dryer since polar compounds may be lost during this removal step.

17.7. Method TO-14A describes the BFB tune check to be a gas sample introduced via a sample loop. TestAmerica Sacramento traps and analyzes BFB using the same analytical technique used with samples.

17.8. Methods TO-14A and TO-15 describe the use of passivated steel canisters for sampling and analysis. No mention is made of the use of air sample bags. TestAmerica Sacramento analyzes samples in air sample bags for VOCs using the same procedures described herein. A modification to the method is noted in the NCM submitted with the final report.

17.9. Methods TO-14A and TO-15 indicate that in order for the ICAL to be acceptable, all compounds must have a %RSD <30 (with allowance for two that could be up to 40% in TO-15). For routine analysis, for those analytes not listed in the published methods and are considered poor performers, TestAmerica Sacramento accepts the ICAL if up to four of these analytes have a %RSD ≤ 55. See Section 10.3.4. This modification accounts for analytical issues that arise from poor performing analytes.

17.10. For the continuing calibration criteria, Method TO-14A states that the RPD of each RF (in the CCV) from the mean RF of the ICAL curve should be <30%; for Method TO-15, the %D for each target compound must compare to the ICAL at ±30%. For routine analysis, for those analytes not listed in the published methods and are considered poor performers, TestAmerica Sacramento accepts the CCV if up to four of these analytes have a %D ±55. See Section 10.6.3. This modification accounts for analytical issues that arise for poor performing analytes.

17.11. Method TO-15 recommends maintaining control charts of the %D values for CCV standards. Due to limitations of the LIMS control chart module, these control charts are maintained using the % recovery values for the CCVs. This modification still permits monitoring for adverse trends.

17.12. Surrogates are not required by the methods. This SOP adds surrogates to every QC and client sample to help monitor for matrix effects and method performance. These compounds are included in the initial calibration at a single concentration for each calibration point. However, surrogates are not reported unless requested.

17.13. Method TO-15 states that the scan time must give 10 scans per peak, not to exceed 1 second per scan. The GC/MS software is set for a sampling rate of 3, which corresponds to approximately 2 to 3 scans per second, depending on the instrument. See the GC/MS operator's manual or "help" on the software for more information about the sampling rate.

17.14. The transfer of sample from Tedlar bags to canisters is supported by the conclusions of an EPA poster titled LOSS/GAIN OF VOCS FROM TEDLAR BAGS AND OTHER SAMPLING EQUIPMENT, by C. Loss Paul as presented at the Presented at The 17th Annual Association for Environmental Health and Sciences Meeting, San Diego, CA, March 21 - 22, 2007. According to the synopsis,

> Soil gas samples are collected to evaluate human health risk from vapor intrusion into homes and other buildings. In order to meet risk assessment goals, the analytical reporting limit for many compounds of concern are down to part per billion ranges. The appropriate sampling tubing, sample containers and leak check compounds should be selected in order to ensure data quality objectives. Mechanisms which can impact sample integrity include adsorption of volatile organic compounds (VOCs) onto the sampling media and diffusion of VOCs through the sampling media which may result in artificially low values, and desorption of compounds from the sampling media into the sample which may results in artificially high values. A literature

search was conducted to compile published results of impacts on soil gas samples from sampling equipment (i.e., tubing) and containers (i.e., Tedlar® bags). The literature search revealed that the recommended holding time for samples stored in Tedlar® bags not exceed 48 hours. A laboratory study was conducted to evaluate the holding times for certain VOCs in 1 L Tedlar® bags stored at two different temperatures. VOCs used in this experiment include 1,1,1-trichloroethane (1,1,1-TCA), trichloroethylene (TCE), benzene, and toluene that were combined in a gas mixture with nitrogen. Bags were filled with the gas mixture and then stored for different times and temperatures. Two incubators were set at two temperatures, C to simulate field temperatures and the bags were stored from 8°C and 25°C 15 hours to 2 weeks. Results of this study show that 1,1,1-TCA, TCE, and benzene can be stored up to one week without significant impact on concentrations. Results for toluene are less conclusive. However, soil gas samples collected in Tedlar® bags should be analyzed as quickly as practical or samples can be transferred to another container with longer holding times (i.e., Summa canister).

18. ATTACHMENTS

18.1. Attachment 1: Standard Analytes, Reporting Limits, and Characteristic Ions

18.2. Attachment 2: BFB GC Operating Conditions, EPA TO-15

18.3. Attachment 3: BFB Acceptance Criteria, EPA TO-14A

18.4. Attachment 4: BFB Acceptance Criteria, EPA TO-15

18.5. Attachment 5: Internal Standards

18.6. Attachment 6: Surrogate Standards

18.7. Attachment 7: Example GC/MS Initial Calibration Curve Review Checklist

18.8. Attachment 8: Example GC/MS Technical Data Review Checklist

18.9. Attachment 9: Schedule for Routine Maintenance of Analytical Instrument

18.10. Attachment 10: Canister Pressurization Logbook (Example Page)

18.11. Attachment 11: GRO Analysis

19. REVISION HISTORY

19.1. WS-MSA-0015, Revision 1.6, Effective 07/31/2015

19.1.1. Changed Copyright Information statement on Title page.

19.1.2. Table A3 –Changed gasoline calibration standard 1 from 200 ppb v/v to 100

ppb v/v.

19.1.3. Editorial changes.

19.2. WS-MSA-0015, Revision 1.5, Effective 04/03/2015

19.2.1. Changed Section 10.1.2.1 from "28mL of 250pbv" to "24mL of 300ppbv".

19.2.2. Changed Section 10.1.2.2 from: An alternative way to load 50ng BFB is to use 100ml of a 10ppbv IS/SURR mix and 24mL of a 250 IS/SURR" to "An alternate way to load 50ng BFB is to use 50mL of a 20ppbv IS/SURR mix and 20mL of a 3000ppbv IS/SURR mix."

19.2.3. Editorial changes

19.3. WS-MSA-0015, Revision 1.4 Effective 03/06/2015

19.3.1. Added Section 7.3.5 to read, "Working standards are valid for a period of 30 days, after which fresh standards are prepared."

19.3.2. Added Section 10.5.7 to read, "CCVs must be monitored on a routine basis using the control chart program (refer to SOP WS-QA-0035) to evaluate the data for trends. The frequency is dependent on the frequency of the analysis. In the LIMS control chart module, select the CCV chart. Once the chart has been evaluated, click "Save Log" to save the chart evaluation in the LIMS."

19.3.3. Inserted Section 17.4 to read, "Method TO-15 presents criteria for evaluating canister cleanliness as less than 0.2 ppbv of any target VOC. TestAmerica Sacramento works with clients to ensure that the canisters meet their data quality objectives, by providing canisters evaluated to the reporting limit or method detection limit as required by their program."

19.3.4. Inserted Section 17.11 to read, "Method TO-15 recommends maintaining control charts of the %D values for CCV standards. Due to limitations of the LIMS control chart module, these control charts are maintained using the % recovery values for the CCVs. This modification still permits monitoring for adverse trends."

19.3.5. Section 17.1.2, inserted the sentence, "These compounds are included in the initial calibration at a single concentration for each calibration point."

19.4. WS-MSA-0015, Revision 1.3, Effective 08/15/2014

19.4.1. Section 8, revised second row of table to reflect the more recent "Advisory, Active Soil Gas Investigations" (from 2012 rather than 2003). Reference in 16.5 also changed.

19.4.2. Inserted Section 5.1.2, safety requirements when transferring sample from air bags into syringes or canisters.

19.4.3. Section 8 – in accordance with the most recent DTSC advisory, changed the holding time to 30 days for canisters under DTCS requirements as well.

19.4.4. Changed Section 7.2.2 from 'concentration of 250 ppbv…' to 'concentration of 300 ppbv…'.

19.4.5. Changed Section 7.3.1.1 from 125 µL to 50 µL Distilled or Nanopure water.

19.4.6. Inserted Section 9.7.1, referring to DoD requirements.

19.4.7. Added Section 10.2.5.1, "The coefficient of determination for a line fit must be greater than or equal to 0.990

19.4.8. Added Section 10.2.5.2, The absolute value of the intercept (printed on the calibration curve plot in Chrom) should be less than ½ the reporting limit (-RL ≤ intercept ≤ +RL). If the intercept is outside the limits, any values below the reporting limit must be evaluated to ensure that false positives or false negatives are not being reported.

19.4.9. Changed Section 10.2.7 to – "The nominal concentrations of the ICAL standards are typically 0.30, 0.40, 0.80, 2.0, 4.0, 8.0, 20, 40 and 60 ppbv, but these may vary depending on the certified mix used to prepare the standards or the volume trapped. The low standard must be at or below the RL. The standards are analyzed by preparing stock standards at the required concentration or by varying the trapped volume of the working standards from the default volume of 250 mL. For example, the 0.30, 0.40, and 0.80 ppbv standards are analyzed by trapping 30, 40, and 80 mL, respectively, of a 2.5 ppbv working standard."

19.4.10. Removed Section 11.2.4, 11.2.4.1, and 11.2.4.2, which discussed the laboratory default to analyze air sample bags at a 20x dilution.

19.4.11. Removed the subheadings from Section 11.4 (Screening).

19.4.12. Section 11.7.2, appended the following, "The sequence is verified by another analyst. This analyst verifies the autosampler sequence, port position, chem. station sequence, and Chrom worklist. The analyst annotates in the instrument maintenance log if the sequence has been verified."

19.4.13. Changed Section 11.7.4 from – 'If the default volume is 250 mL then 410 mL should be trapped' to 'If the default volume is 250 mL then 510 mL should be trapped.'

19.4.14. Combined GC operating conditions and BFB operating conditions (Attachment 2 and Attachment 3) as they are the same. Renumbered the attachments.

19.4.15. Updated Attachments 7, 8, and 10 to current versions.

19.4.16. Editorial comments.

19.4.17. Inserted Attachment 11 – GRO Analysis using Method TO-15

19.4.18. Editorial changes.

19.5. WS-MSA-0015, Revision 1.2, Effective 10/25/2013

19.5.1. Added psig to psia conversion in section 3.10.

19.5.2. Removed reference to Costa Mesa SOP LA-QAS-002.

19.5.3. Added language to clarify "certified clean" in section 8.2.

19.5.4. Changed humidification of method blank canister from 100uL to 50uL with distilled or NanoPure water in section 9.3.

19.5.5. Added language to evaluate EICP for S/N determination in section 10.4.1.

19.5.6. Insert section 10.4.2 to state qualitative compound identification criteria must be met for all concentrations.

19.5.7. Insert 10.4.5 to add language for re-fit / read-back evaluation for % error.

19.5.8. Removed section 11.5 from previous version regarding water addition.

19.5.9. Added language in section 11.7.6 for sample dilution that can is pressurized to 24-26 psia or no more than 3x the initial pressure.

19.5.10. Added language in method modification to use UHP Nitrogen for standards preparation instead of zero air, section 17.1.

19.5.11. Added language in method modification that UHP Nitrogen is used for dilution / pressurization, section 17.1

19.5.12. Added language in method modification that leak check is performed by measuring vacuum instead of pressure as stated in the source method, section 17.3.

19.6. WS-MSA-0015, Revision 1.1, Effective 04/22/2013

19.6.1. Inserted Section 5.1 to address transfer of sample from Tedlar bags to canisters.

19.6.2. Added Section 17.11 regarding the supporting evidence for transferring samples from Tedlar bags to canisters to prolong the holding time.

19.6.3. Updated Attachment 1 to reflect the analytes evaluated at this time.

19.6.4. Removed Attachment 2 until such time as an additional mix is introduced to the analysis.

19.6.5. Section 9.2, removed references to LCS Duplicate.

19.6.6. Inserted Section 9.1.1 regarding LCS duplicates.

19.6.7. Removed all other references to LCS duplicates (aside from Section 9.1.1)

19.6.8. Replaced all references to LA-SRA-002 with WS-QA-0032.

19.7. WS-MSA-0015, Revision 1, Effective 04/08/2013

19.7.1. Revised Attachment 3 to update conditions.

19.7.2. Revised Attachment 4 to update conditions.

19.7.3. Editorial revisions.

19.8. WS-MSA-0015, Revision 0, Effective 01/18/2013

19.8.1. This is the first version of this SOP.

Attachment 1: Standard Mix Analytes and Characteristic Ions

Analytes	CAS #	Quant	Confirmation	RL (ppb v/v)
1,1,1,2-Tetrafluoroethane	811-97-2	86	69, 51	1.0
1,1,1-Trichloroethane	71-55-6	97	99, 61	0.30
1,1,2,2-Tetrachloroethane	79-34-5	83	85, 131	0.40
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	101	151, 103	0.40
1,1,2-Trichloroethane	79-00-5	97	83, 85	0.40
1,1-Dichloro-1-fluuoroethane	1717-00-6	81	61, 45	0.40
1,1-Dichloroethane	75-34-3	63	65, 83	0.30
1,1-Dichloroethene	75-35-4	61	96, 98	0.80
1,1-Dichloropropene	563-58-6	75	110, 39	0.40
1,1-Difluoroethane	75-37-6	51	65	0.40
1,2,3-Trichlorobenzene	87-61-6	180	182, 145	2.0
1,2,3-Trichloropropane	96-18-4	110	97, 75	0.40
1,2,4-Trichlorobenzene	120-82-1	180	182, 145	2.0
1,2,4-Trimethylbenzene	95-63-6	120	105, 77	0.80
1,2-Dibromo-3-chloropropane	90-12-8	157	155, 75	2.0
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	135	85, 87	0.40
1,2-Dichlorobenzene	95-50-1	146	148, 111	0.40
1,2-Dichloroethane	107-06-2	62	49, 64	0.80
1,2-Dichloropropane	78-87-5	76	63, 62	0.40
1,3,5-Trimethylbenzene	108-67-8	120	105, 77	0.40
1,3-Dichlorobenzene	541-73-1	146	148, 111	0.80
1,3-Dichloropropane	142-28-9	76	41,78	0.80
1,4-Dichlorobenzene	106-46-7	146	148, 111	0.40
1,4-Difluorobenzene	540-36-3	114	88, 63	
1,4-Dioxane	123-91-1	88	58	0.80
1.1.1.2-Tetrachloroehtane	630-20-6	131	133, 95	0.40
2,2-Dichloropropane	78-87-5	77	97, 41	0.80
2-Butanone (MEK)	78-93-3	72	43, 57	0.80
2-Chlorotoluene	95-49-8	126	91, 89	0.40
2-Hexanone	591-78-6	58	43, 85100	0.40
2-Methyl-2-propanol	75-65-0	59	57, 41	2.0
3-Chloro-1-propene (Allyl Chloride)	107-05-1	41	76, 78	0.80
4-Ethyltoluene	622-96-8	120	105, 77	0.40
4-Isopropyltoluene	99-87-6	119	134, 91	0.80
4-Methyl-2-pentanone (MIBK)	108-10-1	43	58, 100	0.40
Acetone	67-64-1	43	58	5.0
Acrolein	107-02-8	56	55, 53	2.0
Alpha Methyl Styrene	98-83-9	118	103, 117	0.40
Benzene	71-43-2	78	77, 52	0.40

Analytes	CAS #	Quant	Confirmation	RL (ppb v/v)
Benzyl chloride	100-44-7	91	126, 63	0.80
Bromobenzene	108-86-1	156	77, 158	0.80
Bromoform	75-25-2	173	175, 171	0.40
Bromomethane	74-83-9	94	96, 79	0.80
Butadiene (1,3-Butadiene)	106-99-0	39	54, 53	0.80
Butane	106-97-8	43	41, 58	0.40
Carbon disulfide	75-15-0	76	78, 44	0.80
Carbon tetrachloride	56-23-5	117	119, 121	0.80
Chlorobenzene	108-90-7	112	114, 77	0.30
Chlorodibromomethane	124-48-1	129	127, 131	0.40
Chlorodifluoromethane	75-45-6	51	67, 50	0.80
Chloroethane	75-00-3	64	49, 66	0.80
Chloroform	67-66-3	83	85, 47	0.30
Chloromethane	74-87-3	50	52	0.80
cis-1,2-Dichloroethene	156-59-2	96	61, 98	0.40
cis-1,3-Dichloropropene	10061-01-5	75	77, 39	0.40
Cyclohexane	110-82-7	84	56, 69	0.40
Cyclohexanone	108-94-1	55	42, 98	0.80
Dibromomethane	74-95-3	174	93, 95	0.40
Dichlorobromomethane	75-27-4	85	83, 129	0.30
Dichlorodifluoromethane	75-71-8	85	87, 50	0.40
Dichlorofluoromethane	75-43-4	67	69	0.40
Ethanol	64-17-5	46	45	1.0
Ethyl acetate	141-78-6	43	61, 70	0.30
Ethylbenzene	100-41-4	91	106, 65	0.40
Ethylene Dibromide (EDB)	106-93-4	107	109, 81	0.80
Hexachlorobutadiene	87-68-3	225	223, 227	2.0
Hexane	110-54-3	43	57, 86	0.80
Iodomethane	74-88-4	142	127	0.40
Isooctane (2,2,4-Trimethylpentane)	540-84-1	57	41, 56	0.40
Isopropyl alcohol	67-63-0	45	4359	0.80
Isopropyl ether	108-20-3	45	87, 59	0.80
Isopropylbenzene	98-82-8	120	105, 79	0.80
Methyl methacrylate	80-62-6	69	41, 100	0.80
Methyl tert-butyl ether	1634-04-4	73	57, 41	0.80
Methylene Chloride	75-09-2	49	84, 86	0.40
m-Xylene & p-Xylene	179601-23-1	91	106, 77	0.80
Naphthalene	91-20-3	128	102, 129	0.80
n-Butanol	71-36-3	56	41, 43	2.0
n-Butylbenzene	104-51-8	92	91, 134	0.40
n-Heptane	142-82-5	43	41, 100	0.80

Analytes	CAS #	Quant	Confirmation	RL (ppb v/v)
n-Nonane	111-84-2	43	57, 85	0.80
n-Octane	111-65-9	43	41, 85	0.40
N-Propylbenzene	103-65-1	91	120, 65	0.40
o-Xylene	95-47-6	91	106, 105, 78	0.40
Pentane	109-66-0	43	57, 72	0.80
Propane	74-98-6	43	42, 39	0.40
Propane, 2-methyl	75-28-5	43	41	0.40
Propene	115-07-1	41	39, 42	0.40
sec-Butylbenzene	135-98-8	105	134, 91	0.40
Styrene	100-42-5	104	78, 103	0.40
Tert-amyl methyl ether (TAME)	994-05-8	73	55, 87	0.80
Tert-butyl ethyl ether	637-92-3	59	87, 57	0.80
tert-Butylbenzene	98-06-6	91	119, 134	0.80
Tetrachloroethene	127-18-4	166	164, 131	0.40
Tetrahydrofuran	109-99-9	42	71, 72	0.80
Toluene	108-88-3	91	92, 65	0.40
trans-1,2-Dichloroethene	156-60-5	61	96, 98	0.40
trans-1,3-Dichloropropene	10061-02-6	75	77, 39	0.40
Trichloroethene	79-01-6	130	95, 132	0.40
Trichlorofluoromethane	75-69-4	101	103, 66	0.40
Vinyl acetate	108-05-4	43	86, 42	0.80
Vinyl bromide	593-60-2	106	108, 81	0.80
Vinyl chloride	75-01-4	62	64, 61	0.40
Xylenes, Total	1330-20-7	NA	NA	1.20
Internal Standards				
Chlorobromoethane	107-04-0	130	49.128	
1,4-Difluoroethane	540-36-3	114	88, 63	
Chlorobenzene-d5	3114-55-4	117	82. 54	
Surrogates				
1,2-Dichloroethane-d4	3855-82-4	85	87, 102	
Toluene-d8	2037-26-5	100	98	
4-Bromofluorobenzene	460-00-4	95	174, 176	

Attachment 2: GC Operating Conditions, BFB and EPA TO-15

Method file: TO15				
Method File List				
GC Type: 6890				
Run type: Scan, GC, E1				
Column: Cap				
Split Ratio: 40.5:1				
	Inj.P	**Intfc**	**Source**	
Temp, °C	**150°C**	**220°C**	**NA**	
GC/DIP				
Initial Column Temperature / Hold time	**35°C for 5 minutes**			
Temperature Profile	**35°C - 110°C at 6.0°C /minute, hold for 0.10 minute, 110°C - 220°C at 8.0°C, hold for 1 minute**			
Post Time	**5.0 minutes**			
Oven equilibration Time: 0 min				
Run time: 37.35 min				
Scan Start time: 3.95 min				
Scan Parameters:				
Mass Range: 35 to 300				
Multiplier voltage: varies				
Number of samples: 2				
Threshold: 200 counts				

Attachment 3: BFB Acceptance Criteria, EPA TO-14A

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% 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% of mass 95
175	5.0 to 9.0% of mass 174
176	>95% but <101% of mass 174
177	5.0 to 9.0% of mass 176

Attachment 4: BFB Acceptance Criteria, EPA TO-15

Mass	Ion Abundance Criteria
50	8.0 to 40% of mass 95
75	30 to 66% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	<2% of mass 174
174	50 to 120% of mass 95
175	4.0 to 9.0% of mass 174
176	93% to 101% of mass 174
177	5.0 to 9.0% of mass 176

Attachment 5: Internal Standards

Bromochloromethane
1,4-Difluorobenzene
Chlorobenzene-d5

Attachment 6: Surrogate Standards

1,2-Dichloroethane-d4
Toluene-d8
4-Bromofluorobenzene

Attachment 7: Example GC/MS Initial Calibration Curve Review Checklist

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Sacramento

ICAL Review Checklist
Volatile Air GCMS

Method & Instrument ID: _____ **ICAL Date:** _____
Chrom Worklist #: _____ **Calibration ID:** _____ **TALS Analysis Batch:** _____
Method (Check the applicable box)
☐ TO-14A / TO-15 Modified SCAN ☐ TO-15 Scan ☐ TO-15 SIM ☐ TO-15 SIM (Special Project)

Acceptance criteria are found in the applicable laboratory SOP or in TALS. If item is No, create an NCM.

Review Items	Level 1		NA	Level 2
General Criteria	Yes	No		
Standards analyzed within 24 hour tune time.				
Chrom Peak Review calibration reports (F6) reviewed for error flags				
Retention time correct for isomers/coeluters and all other analytes • Dichlorodifluoromethane / 1,2-Ddichlorofluoroethane • Trichlorofluoromethane / 1,1,2-Trichlorofluoroethane • Hexane / Vinyl acetate • 2-Methyl butane / Acrolein • 1,1-Dichloroethene / cis-1,2-Dichloroethene / trans-1,2-Dichloroethene • 1,2-Dichloroethane / Benzene • cis-1,3-Dichloropropane / trans-1,3-Dichloropropene • Ethylbenzene / m/p-Xylene / o-Xylene • 4-Ethyl toluene / 1,3,5-Trimethylbenzene / 1,2,4-Trimethylbenzene • tert-Butylbenzene / 4-Isopropyltoluene • 1,3-Dichlorobenzene / 1,4-Dichlorobenzene / 1,2-Dichlorobenzene • 1,2,4,5-tetramethylbenzne / 1,2,3,4-tetramethylbenzene • 1,2,4-Trichlorobenzene / 1,2,3-Trichlorobenzene				
Curve reviewed in Chrom for all analytes and all appropriate leves are included/excluded				
Uploaded all calibration levels to TALS after any changes were made in Peak Review				
Method for this worklist set as Most Recent Method				
ICAL locked in Chrom and in TALS				
ICV passes -See criteria below				
Manual Integrations clearly identified, initialed, dated and reason given.				
SIM Criteria				
PFTBA tune documentation present in Chrom & TALS and meets criteria				
RSD < 30% (≤ 40% for up to 2 analytes.				
Linear regressions >0.995 and intercept <+/- ½ RL/IS amount, (minimum of 5 points curve).				
All internal standards are within 60-140% of ICAL midpoint.				
ICV (Second Source) meets 70-130% criteria (60-140%) for up to 2 analytes.				
Scan Criteria				
BFB Tune documentation present in Chrom & TALS and meets criteria				
For GRO: WS-GRO report (F8) reviewed to ensure IS/Surrogate subtraction from area sum.				
RSD < 30% (≤ 40% for up to 2 analytes, and ≤ 55% for up to 4 poor performers defined in SOP)				
Linear regressions >0.995 and intercept <+/- ½ RL/IS amount, (minimum of 6 points curve).				
All internal standards are within 60-140% of ICAL midpoint (for TO-15 / TO-15 Modified) or within 50-200% of ICAL midpoint (for TO-14A)				
ICV (Second Source) meets 70-130% criteria (60-140% for up to 2 analytes, and 45-155% for up to 4 poor performers defined in SOP)				

	Repeat Failure from Prev ICAL	
ICAL compounds outside RSD criteria (write compound and %RSD	Yes	No
_____		

ICV (second source) compounds outside RSD criteria (write compound and %D	Yes	No
_____		

1st Level Reviewer: _____ Date: _____
2nd Level Reviewer: _____ Date: _____
Comments: _____

Attachment 8: Example GC/MS Technical Data Review Checklist

TestAmerica
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Sacramento

MS VOA AIR Data Review Checklist

Job(s):_____ Instrument ID: _____
☐TO-14A ☐TO-14A MOD ☐TO-15 ☐TO-15 MOD ☐TO-15 SIM
ICAL Batch: _____ TALS Batch:_____

Review Items	Level 1 Yes	Level 1 No	Level 2	NA
Initial Calibration				
1. Is ICAL locked in Chrom & TALS?				
2. Is ICV properly linked in TALS for L3/L4 jobs?				
Continuing Calibration				
1. BFB tune documentation meets criteria? The PFTBA Tune Documentation is attached in TALS (SIM methods only)?				
2. All Internal Standards within 50-200% of ICAL mid-point (TO-14A/TO-14A MOD) or 60-140% (TO-15/ TO-15 MOD)?				
3. Does the %D meet:30%D for standard program, and 20%D for DoD?				
4. Isomeric pairs checked for correct peak assignment? • Dichlorodifluoromethane / 1,2-Dichlorotetrafluoroethane • Trichlorofluoromethane / 1,1,2-Trichlorotrifluoroethane • 2-Methyl butane / Acrolein • 1,1-Dichloroethene / cis-1,2-Dichloroethene / trans-1,2-Dichloroethene • Hexane / Vinyl acetate • 1,2-Dichloroethane / Benzene • cis-1,3-Dichloropropene / trans-1,3-Dichloropropene • Ethylbenzene / m/p-Xylene / o-Xylene • 4-Ethyl toluene / 1,3,5-Trimethylbenzene / 1,2,4-Trimethylbenzene • tert-Butylbenzene / 4-Isopropyltoluene • 1,3-Dichlorobenzene / 1,4-Dichlorobenzene / 1,2-Dichlorobenzene • 1,2,4,5-tetramethylbenzene / 1,2,3,4-tetramethylbenzene • 1,2,4-Trichlorobenzene / 1,2,3-Trichlorobenzene				
Client Samples & QC Sample Results				
1. Was analysis done within holding times?				
2. Are Chromatograms reviewed and spectra verified?				
3. Are positive results within calibration range?				
4. Are all positive results within RT windows?				
5. All target cpds in MB < RL (< ½ RL for DoD)? Requires NCM if "no."				
6. Are surrogate recoveries for MB and LCS/LCSD within control limits?				
7. Are target constituents in LCS/LCSD within control limits?				
8. Are all Chrom graphics uploaded?				
9. Are all QC samples properly linked in TALS?				
10. Do the Internal Standards meet criteria +/-50% (o +/-200% (TO-14A / TO-14A MOD), +/-60% to +/- 140% (TO-15/TO-15 MOD)?				
11. Do results (e.g., dilutions) make sense?				
12. Are MS/MSD recoveries and RPDs within control limits?				
13. Have all flags been reviewed for appropriateness?				
14. All manual integrations appropriate and documented?				
15. Are non-conformances documented as NCMs?				
16. Are client sample surrogate failures flagged and NCM written?				
17. Dilutions due to target cpds? ____ Dilutions due to non-targets? ____				
18. Has QC checker been run?				

1st Level Reviewer / Date:_____ 2nd Level Reviewer / Date: _____

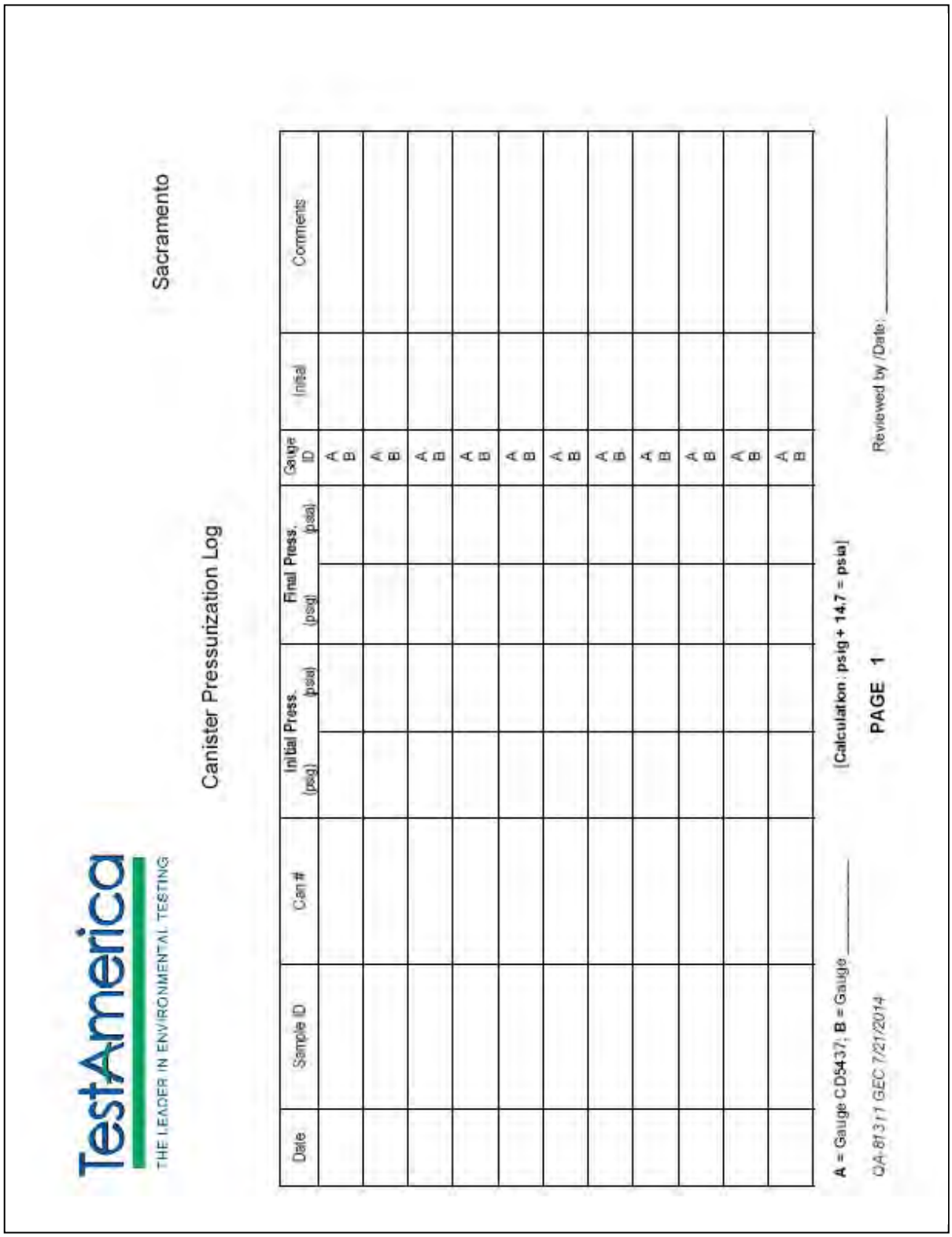
Comments: _____

c:\forms\checklists\msa-002 air gcms data review 2014-02-24.doc MSA-002 RKE 07/16/2014

Attachment 9: Schedule for Routine Maintenance of Analytical Instrument

Frequency	Maintenance Item
Daily	Check baseline level with analysis of blanks. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks. Autosampler: Check for proper operation. Leak check system.
As needed	Replace septum. Clean injector port. Break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add or change oil, if needed. Replace the exhaust filters on the mechanical rough pump every 1-3 years, or as needed. Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels. Check ion source and analyzer (clean, replace parts as needed). Clean source, including all ceramics and lenses - as is indicated by a variety of symptoms including inability to properly tune, poor response, and high background contamination. Repair/replace jet separator. Replace filaments when burnt out or performance indicates need for replacement.

Attachment 10: Canister Pressurization Logbook (Example Page)



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento

Canister Pressurization Log

Date	Sample ID	Can #	Initial Press. (psig)	Initial Press. (psia)	Final Press. (psig)	Final Press. (psia)	Gauge ID	Initial	Comments
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		

A = Gauge CD5437; B = Gauge _____ [Calculation: psig + 14.7 = psia]

PAGE 1

Reviewed by /Date: _____

QA 81311 GEC 7/21/2014

1. SCOPE AND APPLICATTION

1.1. This method describes the analysis of volatile petroleum hydrocarbons. The procedure used for the petroleum hydrocarbons is based on SW-846. Refer to Table A1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 218°C. Classes of compounds best suited for evaluation by this analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

2. SUMMARY OF METHOD

Refer to Section 2 of the main body of this SOP for a summary of the method.

3. DEFINITIONS

Refer to Section 3 of the main body of this SOP for a summary of the method.

4. INTERFERENCES

Refer to Section 4 of the main body of this SOP for interferences.

5. SAFETY

Refer to Section 5 of the main body of this SOP for Safety Information.

6. EQUIPMENT AND SUPPLIES

Refer to Section 6 of the main body of this SOP for equipment and supplies.

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Reagents

7.1.1. UHP Helium for carrier gas

7.1.2. UHP H_2 for detector combustion

7.1.3. Zero-grade air for detector combustion

7.1.4. UHP N_2 for sample preparation, standard preparation, and method blanks

7.1.5. Liquid N_2

7.2. Standards – Neat standards are good for 1 year from date purchased or if a vendor supplied standard has an earlier expiration date then that date is used.

7.2.1. Certified BTEX, MTBE, and alkane standards in pressurized gas cylinders and neat liquids, obtained from select NIST-approved vendors.

7.2.2. Certified Hexane standards in pressurized gas cylinder and neat liquid, obtained from select NIST-approved vendors.

7.2.3. Certified neat unleaded gasoline standards, obtained from select NIST-approved vendors.

7.3. Standard Preparation

Standard preparation activities are performed accordance with TestAmerica SOP WS-QA-0017.

7.3.1. Dilutions of standards are made on a volume/volume basis using serial dilution methodologies. UHP N_2 is used as the diluent gas.

7.3.2. Primary gas standards are either purchased from manufacturers in pressurized, pre-mixed gas cylinders, or prepared in passivated canisters or air sample bags from neat liquid standards.

7.3.2.1. Primary Gas Standard Prepared in Canisters - To prepare a primary gas standard from neat liquids, an aliquot of the compound is injected into a clean and evacuated (-30 inches of mercury) passivated canister. The density of the compound is used to determine the mass injected. The canister is pressurized using UHP N_2 and is then allowed to equilibrate.

7.3.2.2. Primary Gas Standard Prepared in Air Sample Bags - To prepare a primary gas standard from neat liquids, an aliquot of the compound is injected with a volumetric gas-tight syringe into a new and clean air sample bag. The air sample bag is filled with UHP N_2 to the appropriate volume using a volumetric gas-tight syringe. The air sample bag standard is then allowed to equilibrate.

To calculate the concentration of the standard prepared:

$$ppmv = \frac{\left(D \times V_1 \times 24.45 \times 10^6\right)}{\left(MW \times V_2\right)}$$

Where:

V_1 = aliquot of neat liquid (mL)

D = density of neat liquid (g/mL)

V_2 = final volume in passivated canister or air sample bag (L)

24.45 = molar volume (L/mole) at STP

10^6 = conversion factor

MW = molecular weight (g/mole)

7.3.3. Working gas standards are good for 6 months from date of preparation. They are prepared in passivated canisters or air sample bags by making dilutions of the primary pre-mixed gas cylinders (from vendors) or by making dilutions of the prepared primary gas standard prepared from the neat liquid standards (see Section 7.3.2).

7.3.3.1. Working Gas Standard Prepared in Canisters - To prepare a working gas standard, an aliquot of the prepared primary gas standard or of the pre-mixed primary gas standard is transferred to a clean and evacuated passivated canister. The aliquot is metered in by measuring the vacuum of the canister as the aliquot is being transferred. The ratio of the final canister pressure and the pressure transferred is the dilution factor. The canister is pressurized with UHP N_2 and is then allowed to equilibrate.

7.3.3.2. Working Gas Standard Prepared in Air Sample Bags - To prepare a working gas standard, an aliquot of the prepared primary gas standard or of the pre-mixed primary gas standard is transferred to a new and clean air sample bag. The aliquot is transferred by direct injection with a volumetric gas-tight syringe. The air sample bag is filled with UHP N_2 to the appropriate volume using a volumetric gas-tight syringe. The air sample bag standard is then allowed to equilibrate.

7.3.3.3. To calculate the concentration of the standard prepared:

$$ppmv = C_{ps} \times {P_i}/{P_f}$$

Where:

C_{ps} = concentration of compound in primary gas standard, ppmv

P_i = aliquot of primary gas standard used in psia or in mL if using air sample bag

P_f = final pressure of canister in psia or final volume in mL if using air sample bag

7.3.4. Gasoline Calibration Standards

7.3.4.1. The primary standards are prepared in passivated canisters from neat unleaded gasoline. Gasoline from one vendor will be used to prepare two or more gasoline calibration standards. The second

source LCS is prepared from unleaded gasoline obtained from a different lot number.

7.3.4.2. A 10-, 25-, 50-, or 100-µL gas-tight syringe is used to transfer the neat gasoline into the septum-capped evacuated canister. The total (barrel + needle) syringe volume is accounted for.

8. SAMPLE PRESERVATION AND STORAGE

Refer to Section 8 of the main body of this SOP for information regarding sample preservation and storage.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the main body of this SOP for general quality control requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the % RSD of the response factor or calibration factor of each analyte is ≤ 30%. If an analyte exceeds the criteria, then a regression line (linear or curved) may be attempted.

10.1.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the **normal** concentration/response behavior of the detector.

10.1.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is ≤ 30%. The equation for average response factor is:

Equation A1

$$\text{Average response factor} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.1.3. Linear regression

The linear fit uses the following function:

Equation A2

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response

x = Concentration

a = Slope

b = Intercept

10.1.4. Quadratic curve

The quadratic curve uses the following function:

Equation A3

$$x = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where: x = concentration

y = instrument response

a = 2nd order coefficient (curvature)

b = 1st order coefficient (slope)

c = constant (intercept)

10.2. Evaluation of calibration curves

The %RSD from the calibration curve is used to evaluate the initial calibration. If a regression line is used, the coefficient of determination (r2) shall be greater than or equal to 0.990, or the correlation coefficient (r) shall be greater than or equal to 0.995, whichever is appropriate to the regression fit used.

10.3. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response should be less than ± ½ the reporting limit for the analyte.
- The RSD for average response factors for each component must be ≤ 20%.
- When RSD is inappropriate, either the correlation coefficient (r) must be ≥ 0.995, or the coefficient of determination (r^2) must be ≥ 0.990.

10.4. Initial Calibration (ICAL)

10.4.1. At the beginning of each initial calibration curve for gasoline or other hydrocarbon fuel mixtures, the carbon range is established from the n-alkanes in the calibration standard. The start and end points for the peak summing range is the retention time at the apex of the beginning and ending carbon

range of the fuel mixture (e.g RT of C6 to RT of C12 measured at the beginning of the start peak and the end of the end peak.

10.4.2. The initial calibration for the GRO curve consists of 6 calibration standards (concentrations listed in Table A3)

10.5. Quantitating hydrocarbon mixtures:

Starting and ending retention times for quantitation are determined for each fuel as per 10.4.1. The peak areas between the starting and ending times are summed and 1,4-Difluorobenzene is used to generate a response factor. This factor is used to quantitate sample results, and depending on the client requirements, this factor may be applied to the same retention range as the standard, or to a different range.

10.6. Integration

10.6.1. When evaluating the initial calibration, also evaluate hydrocarbon pattern integration.

10.6.1.1. The integrated peaks should run from the point in the chromatogram prior to peaks to a region following the mixture's peaks in a fairly straight line.

10.6.2. The default integration parameters generated as a result of evaluating the initial calibration should remain in effect until the initial calibration is reanalyzed.

10.7. Evaluate the initial calibration as per 10.1 and 10.3.

10.8. A second source standard is analyzed immediately following the initial calibration. The acceptance criteria for the second source standard is ± 30% from the expected value.

10.9. Continuing Calibration Verification (CCV)

10.9.1. A CCV (5000ppbv or another mid-level standard) is analyzed at the beginning of each 24 hour analytical sequence. The integration for the GRO CCV follows the same technique as stated in 10.6.

10.9.2. The acceptance criteria for the CCV is ± 30% D.

11. PROCEDURE

11.1. Refer to Section 11 of the main body of this SOP for information regarding sample preparation and other procedures.

12. CALCULATIONS/DATA REDUCTION

12.1. Refer to Section 12 of the main body of this SOP for general information regarding data analysis and calculations.

12.2. Gasoline is quantitated using the area between two marker peaks as specified in Table A2.

12.3. Calculate results in samples as in Section 12 of the main portion of this SOP.

13. METHOD PERFORMANCE

Refer to Section 13 of the main body of this SOP for method performance criteria.

14. POLLUTION CONTROL

Refer to Section 14 of the main body of this SOP for information relating to pollution control.

15. WASTE MANAGEMENT

Refer to Section 15 of the main body of this SOP for information relating to waste management.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035, 8000B, and 8015B.

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, March 2003, Section 8000C.

16.3. Leaking Underground Fuel Tank Field Manual, State of California, October, 1989

17. METHOD MODIFICATIONS

17.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2. This method is performed on a GC/MS, with surrogate values calculated using Method TO-14A/TO-15, and the TPH values calculated using the total area chromatogram. All of the reference methods are based on GC-FID as the detector and the limitations thereof.

18. ATTACHMENTS

18.1. Table A1 – Standard Analyte List

18.2. Table A2 – Volatile Petroleum Hydrocarbon Quantitation Ranges.

18.3. Table A3 - Calibration Standard Concentrations

18.4. Table A4 - LCS Spiking

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Table A1		
Standard Analyte List		
Compound	CAS Number	Reporting Limit (ppbv/v)
Gasoline Range Organics (GRO)	NA	100

Table A2		
Volatile Petroleum Hydrocarbon Quantitation Ranges.		
Regulatory Method GRO	Quantitation Range	
	Start	End
GRO	n-C3	n-C12
nC4-nC12	nC4	nC12
C4-C12	n-C4	n-C12

[1] *The method specifies that the peak summing window must start at the start of the n-C6 peak and finish at the start of the n-C10 peak.*

Table A3						
Calibration Standard Concentrations (ppbv/v)						
Component	1	2	3	4	5	6
Gasoline	100	500	1000[1]	5,000	10,000	20,000

[1]*Designates the mid-level standard concentration used for the ICV and CCVs following the initial CCV of a sequence.*

Refer to the main body of this SOP for surrogate details.

Table A4	
LCS Spiking	
Component	Concentration (ppbv)
GRO	5000

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Percent Moisture, Solids, Ash, Organic Matter in Soil Samples

Methods: SM 2540G and ASTM D2974-07a

Approvals (Signature/Date):

8/27/2014	8/28/2014
Roseann Ruyechan Date	Steve Jackson Date
Inorganics Department Manager	Regional Safety Coordinator
8/27/2014	8/27/2014
Virginia Zusman Date	Deborah L. Lowe Date
Quality Assurance Manager	Laboratory Director

Controlled Source: Intranet

Company Confidential & Proprietary

1.0 Scope and Application

1.1 This method is applicable to the determination of the moisture content of soil, rock and soil-aggregate mixtures, and products. This method is also used to determine the ash content in oil, liquid (non-aqueous, i.e. sludges) and petroleum samples.

1.2 On occasion clients may request slight modifications to this SOP. These modifications are handled as indicated in PT-QA-M-001, Quality Assurance Manual.

2.0 Summary of Method

2.1 A homogenous sample is dried at 103℃ to 105℃ (to determine moisture), and 500 ± 50°C (to determine Ash). The weight loss of the sample at 103-105°C represents the moisture content and the residue the total solids.

2.2 The Ash content is the percentage of the total solids that remain after burning at 450-550°C.

2.3 Percent organic matter, or volatile solids, is equal to 100% (of the total solids) - the % ash. Therefore the % organic matter is defined as the percentage of combustible material found in the sample based on dry weight.

3.0 Definitions

3.1 Please refer to the glossary in the Quality Assurance Manual- PT-QA-M-001 for additional definitions.

3.2 TALS – TestAmerica LIMS

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Non-homogeneous samples may give erratic results.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 After heating, sample containers will present a burn hazard. Tongs or heat resistant gloves must be used when handling samples after heating in the oven or muffle furnace.

5.2 Primary Materials Used

5.2.1 There are no materials used in this method that have a serious or significant hazard rating.

5.3 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor and/or the EHSC.

6.0 Equipment and Supplies

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this sop can be met.

6.1 Analytical balance: capable of accurately weighing ± 0.0001 g

6.2 Drying Oven, 103°C ± 2°C

6.3 Muffle furnace, 550°C ± 50°C

6.4 Desiccators

6.5 Evaporating dishes, various sizes, capable of being heated above 550°C

6.6 Wooden Spatula

6.7 Aluminum pans

7.0 Reagents and Standards

7.1 Not Applicable

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples are not chemically preserved.

8.2 Samples are stored in plastic or glass containers at ≥0.0°C but ≤ 6.0°C.

8.3 There is no method recommended holding time for solid samples for these tests. The lab will assign a 28 day holding time internally.

9.0 Quality Control

9.1 A duplicate sample is analyzed with every set of 10 or fewer samples.

9.1.1 The acceptable range between the sample and sample duplicate is %RPD less than or equal to 20 percent.

9.1.2 If the %RPD is outside criteria, check calculations and verify balance calibration. Reanalyze the samples once and evaluate results vs the initial sample/duplicate. If the failure repeats, report data with an NCM and narrative description of the initial and second data sets.

10.0 Procedure

10.1 Calibration and Standardization

10.1.1 Balances are calibrated as needed, and proper balance operation is verified daily, prior to sample analysis, bracketing the range of weights used. See SOP PT-QA-012 for balance calibration and verification details.

10.1.2 Oven temperature must be checked daily and recorded in the oven temperature log. Oven and muffle furnace temperatures at the time of analysis are documented in the batch information in TALS.

10.2 Total Solids – 2540G

10.2.1 Preparation of evaporating dish: If volatile solids or ash content are to be measured, ignite a clean evaporating dish at 500± 50℃ for one hour in a muffle furnace. If only total solids is being measured, an aluminum pan baked at 103-105°C may be used. Cool dishes to room temperature in desiccator before use. Dishes may be store in desiccator until ready for use.

10.2.2 Preparation of sample: Rocks, stones, twigs, leaves, or other foreign matter which interfere with homogenizing the sample must be carefully removed and the remaining sample mixed thoroughly so that a representative sample will be obtained. Where it has been necessary to remove artifacts, the action taken and artifacts removed from the sample must be adequately described by the analyst in a narrative provided with the sample data. If there is doubt concerning the proper handling of sample artifacts (due to the nature of the particular sample or project), the laboratory supervisor and project manager must determine the procedure to be followed, and the resulting actions must be documented in a narrative provided with the sample data. For volatile solid samples and samples analyzed for percent dry solids, supernatant liquids are mixed into the sample.

10.2.3 Place weighing dish plus 5 - 10g sample in a drying oven maintained at 103℃ ± 2°C. If samples contain enough liquid to flow readily, use 25 to 50 g of sample for analysis. Sample handling and drying must be conducted in a well ventilated area.

10.2.4 Dry the sample for a minimum of 12 hours. Remove the sample from the oven and cool in a dessicator at least 30 minutes. If dried less than 12 hours, it must be documented that constant weight was attained by repeating the dry/desiccate/weight cycle, with a minimum of one hour drying time in each cycle, until a change in weight of no greater than 0.05g or 4% (whichever is greater), is seen between start weight and final weight of last cycle.

10.3 Ash (Fixed) and Volatile Solids:

10.3.1 Transfer the dried residue from Section 10.2.4 to a muffle furnace at 500± 50℃ and ignite for one hour. (If the residue contains large amounts of organic matter, first ignite it over a gas burner and under a fume hood in the presence of adequate air to lessen losses due to reducing conditions and to avoid odors in the laboratory.) Cool in desiccator to room temperature and weigh.

10.3.2 Return the sample to the muffle furnace for an additional 30 minutes. Cool in a desiccator to room temperature and weigh. Repeat burning, cooling and weighing until a constant weight is obtained. Constant weight is defined as a change in weight of no greater than 0.05g or 4% (whichever is greater), between start weight and final weight of last cycle.

10.4 Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

10.5 Organic matter is determined by subtracting percent ash content from one hundred.

11.0 Calculations / Data Reduction

11.1 Percent Total Solids:

$$\frac{(A - B)}{(C - B)} \times 100$$

Percent Volatile Solids or Organic Matter:

$$\frac{(A - D)}{(A - B)} \times 100$$

Percent Ash (Fixed) Solids:

$$\frac{(D - B)}{(A - B)} \times 100$$

Where:

A = Weight of dried residue and dish, mg

B = Weight of dish

C = Weight of wet sample and dish, mg

D = Weight of residue and dish after ignition, mg

11.2 Percent Moisture

%Moisture = 100% - % Total Solids

11.3 Report all results to the nearest 0.1 percent. Report furnace temperature used for ash content determinations.

11.4 Duplicate Sample, Relative Percent Difference (RPD):

$$RPD = \frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2}\right)} \times 100$$

Where:

X_1 = Original Result

X_2 = Duplicate Result

12.0 Method Performance

12.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

13.2 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention.

13.3 This method does not contain any specific modifications that serve to minimize or prevent pollution.

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.1.1 Used Soil Samples – This waste is collected in containers identified as "Lab Trash", Waste #12.

15.0 References / Cross-References

15.1 Standard Methods for the Examination of Water and Waste Water, Method 2540G, 2011

15.2 ASTM Standard D 2974-07a, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat Materials, 2008

15.3 PT-QA-012, Selection and Calibration of Balances and Weights

15.4 PT-QA-016, Nonconformance & Corrective Action System

15.5 PT-QA-021, Quality Assurance Program

15.6 PT-QA-024, Subsampling

15.7 PT-QA-M-001, Pittsburgh Laboratory Quality Assurance Manual

15.8 PT-HS-001, Pittsburgh Facility Addendum EH&S Manual

16.0 Method Modifications

16.1 The ash/organic content temperature (550$\pm$50^{o}C) requirement from SM2540G is used in place of either method C (440^{o}C) or D (750^{o}C) from ASTM D 2974-07a.

17.0 Attachments

17.1 All sample preparation and analysis information will be documented electronically in TALS LIMS. All the documents associated with an analysis will be electronically available for inclusion in the final report.

17.2 Organic Matter Worksheet

18.0 Revision History

18.1 Revision 4, 11/7/08

18.2 Revision 5, 10/14/2009

18.3 Revision 6, 1/21/2012

18.4 Revision 7, 8/28/2014

SOP section	Change from	Change to	Reason
Cover	Technical Analyst – Mike Wesoloski Steve Jackson – Health & Safety Manager/ Coordinator QAM – Nasreen DeRubeis	Inorganics Department Manager – Roseann Ruyechan Regional Safety Coordinator QAM –Virginia Zusman	Change in personnel
Entire SOP	Removed Updated	DoD references PT-LQAM to PT-QA-M-001	Voluntarily withdrew from the program SOP numbering change
1.1	Added	Added “and products” to the end of the first sentence	Clarification
1.2	Added	SOP Checklist text on modifications	SOP Review Sheet format
3.1	Sample Duplicate definition	Reference for glossary in QA Manual	SOP Review Sheet format
3.2	QC Batch definition	TALS – TestAmerica LIMS	Clarification
5.1	Removed	Radiation Safety Manual	Does not pertain to this facility
5.2	Changed	MSDS to SDS	Due to change in industry standard language
5.2.1	Removed statement for VITON gloves		Clarification
6	Added	Text from SOP Review Checklist for this section	SOP Review Sheet format
6.6	Removed Top-loader balance		Clarification
8	Updated	≤6˚C to ≥0.0˚C but ≤6.0 ˚C	Correction

9.1.2	Added	Verify balance calibration…. If failure repeats, NCM and narrative	Clarification
10.1.1	Added	Balances are calibrated as needed…verified bracketing the range of weights used. See SOP PT-QA-012.	Clarification
10.2.3	Added	If samples contain enough liquid to flow readily, use 25 to 50g of sample for analysis	To match method weights (SM2540G)
10.2.4	change in weight of no greater than 0.01g	change in weight of no greater than 0.05g, or a change of less than 4% of the sample weight	To match method requirement (SM2540G)
10.3.2	Added	Requirement for repeated heating, cooling and weighing to constant weight	To match method requirement (SM2540G)
12.1	Added	Supervisor responsibility text from SOP Checklist	SOP Review Sheet format
15.2	Removed	Reference to ILM04.0	No longer perform CLP analysis
15.5, 6, 7, 9, 13, 14	Removed references to methods/SOP not used for this procedure		Clarification
15.13 (now 15.7)	PT-LQAM	PT-QA-M-001	QA Manual ID change
5 and 15.8	added	Reference to Pittsburgh EH&S Manual PT-HS-001	Clarification

General Chemistry Worksheet

Batch Number: 160-25593
Method: SM 2540G
Analyst: Cox, Chrissy M

Date Open: Jan 10 2012 10:27AM
Batch End:

Lab ID	Client ID	Method Chain	Basis	CrucibleID	Empty Dish Weight	Wt of Dish and sample prior to drying	Final weight/volume of sample	First Weighing	Second Weighing
160-7343-A-2	1311B Free Sand I12742	2540G	T	11	27.75 g	37.08 g	9.33 g	36.54 g	36.54 g
160-7343-A-2-DU		2540G	T	Z15	29.51 g	38.94 g	9.43 g	38.40 g	38.40 g

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General Chemistry Worksheet

Batch Number: 180-25593
Method: SM 2540G
Analyst: Cox, Chrissy M

Date Open: Jan 10 2012 10:27AM
Batch End:

Lab ID	Client ID	Method Chain	Basis	Third Weighing	Weight of Residue and Dish	Weight after Ignition 1	Weight after Ignition 2	Weight after Ignition 3	Weight at 550 C
180-7343-A-2	1311B Free Sand I12742	2540G	T	36.54 g	36.54 g	36.55 g	36.54 g	36.55 g	36.55 g
180-7343-A-2-DU		2540G	T	38.40 g	38.4 g	38.40 g	38.39 g	38.40 g	38.4 g

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General Chemistry Worksheet

Batch Number: 160-25593
Method: SM 2540G
Analyst: Cox, Chrissy M

Date Open: Jan 10 2012 10:27AM
Batch End:

Lab ID	Client ID	Method Chain	Basis	Calculation Message
160-7343-A-2	1311B Free Sand I12742	2540G	T	OK
160-7343-A-2-DU		2540G	T	OK

Oven ID: 2/3
Oven, Bath or Block Temperature 1: 104/550 Celsius
Perform Calculation (0=No, 1=Yes): 1

General Chemistry Worksheet

Batch Number: 160-25593
Method: SM 2540G
Analyst: Cox, Chrissy M

Date Open: Jan 10 2012 10:27AM
Batch End:

Comments

Lab ID	Client ID	Method Chain	Basis	Analysis comment
160-7343-A-2	1311B Free Sand I12742	2540G	T	
160-7343-A-2-DU		2540G	T	

Batch Comment: oven 2- 10:32 1/10/12 - 08:44 1/11, oven 3- 08:55-09:55 0 1/11/12

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CRDS Water Isotope Analyzer Operation

Created 2014 by Crystal Tulley-Cordova

Revised 3/10/2015 by Kali Blevins

Part 1 – Sample Preparation

1. Fill standard and sample vials
    a. Pipette samples
        i. Pipette 0.5 mL from sample container into 2mL crimp-top vial. Use a separate, clean pipette tip for each sample. Discard used tips into a beaker for washing and re-use.
        ii. Use crimper to seal lid. Check to make sure the lid is tight (i.e. does not rotate on the top of the vial) and cleanly crimped. If the crimper is not creating a tight seal, you can adjust it with an Allen wrench.
        iii. Pipette samples 8 at a time, writing each sample name or a unique abbreviation of each sample name on each 2 mL vial with a sharpie.
        iv. Place samples in the sample tray leaving the first four plate locations empty and leaving one plate location blank between each group of eight samples. Empty locations will be used for reference waters. A maximum of 56 samples should be run at a time.
    b. Pipette reference water
        i. In order to limit evaporation, minimize the amount of time the reference waters are out of the refrigerator and the amount of time the cap is off of the reference water bottles. After pipetting reference waters, immediately replace the cap securely on the bottle and parafilm tightly around the cap.
        ii. Pipette 0.5 mL from container into 2mL crimp-top vial. Use a separate, clean pipette tip for each reference water. Discard used tips into a beaker for washing and re-use.
        iii. Use crimper to seal lid. Check to make sure the lid is tight (i.e. does not rotate on the top of the vial) and cleanly crimped.
        iv. Fill 1 vial with reference water PZ and 1 with reference water UT. These primary references are used to calibrate each run.
        v. Fill 1 vial with PT to put at the beginning of the run, 1 additional vial to run before every 8 samples, and 1 to be run in the last plate location. This secondary reference water is used to quality check the accuracy of the calibration regression and to generate a drift correction in each run. An example plate map is shown below.

Table 1. Example plate map with maximum plate locations filled

64	Sample54	65	Sample55	66	Sample56	67	PT	68		69		70	
57	Sample48	58	PT	59	Sample49	60	Sample50	61	Sample51	62	Sample52	63	Sample53
50	Sample41	51	Sample42	52	Sample43	53	Sample44	54	Sample45	55	Sample46	56	Sample47
43	Sample35	44	Sample36	45	Sample37	46	Sample38	47	Sample39	48	Sample40	49	PT
36	Sample29	37	Sample30	38	Sample31	39	Sample32	40	PT	41	Sample33	42	Sample34
29	Sample23	30	Sample24	31	PT	32	Sample25	33	Sample26	34	Sample27	35	Sample28
22	PT	23	Sample17	24	Sample18	25	Sample19	26	Sample20	27	Sample21	28	Sample22
15	Sample10	16	Sample11	17	Sample12	18	Sample13	19	Sample14	20	Sample15	21	Sample16
8	Sample4	9	Sample5	10	Sample6	11	Sample7	12	Sample8	13	PT	14	Sample9
1	PT	2	PZ	3	UT	4	PT	5	Sample1	6	Sample2	7	Sample3

Part 2 – Instrument Preparation

2. Clean syringe
    a. Unplug Autosampler and carefully remove syringe
    b. Don gloves (latex or nitrile), a lab coat, and safety goggles
    c. Open chemical fume hood sash, turn on light, and ensure that it is functioning properly with adequate air flow
    d. Lay down 2 Kimwipes; one to lay syringe on and another for ejecting solvent onto
    e. Use solvent (1-methyl-2-pyrrolidinone) from cabinet below hood to clean the syringe
        i. Insert syringe through the septum on solvent bottle, preferably targeting a previously punctured location. Be careful not to bend the syringe tip.
        ii. Extract solvent with syringe to capacity by moving plunger slowly until it comes out of the syringe. Rotate the plunger as you remove it to thoroughly clean the syringe. Rotating may also help to free the plunger if it becomes stuck.
        iii. Remove syringe from solvent bottle and eject solvent from syringe onto Kimwipe by replacing the plunger in the syringe and fully depressing it.
        iv. Repeat process at least three times or as many times as necessary until the plunger moves smoothly and easily in the syringe. If you cannot get the plunger to move freely it might need to be replaced. Note that broken syringes must be discarded in the sharps container.
        v. Once complete, re-cap the solvent bottle and return to storage under hood. Leave the Kimwipe with the ejected solvent in the fume hood until the solvent has completely evaporated before discarding.
    f. Use deionized water to rinse the syringe three times, fully filling and emptying the syringe each time
    g. Dry syringe
        i. Remove the plunger from the syringe, wiping it with a Kimwipe
        ii. Insert tip of syringe into the piece of blue tubing near the compressed air station, preferably locating a pre-existing hole and being careful not to bend the syringe
        iii. Place the opening of the tubing loosely against the air nozzle (do not try to force tubing over the nozzle)
        iv. Turn the air on and let it run until any water has been blown out of the syringe

v. Check the syringe to ensure that no water droplets remain and then replace the plunger. This eliminates the possibility of air bubbles that might affect the fill volume of the syringe.

h. Replace Syringe
   i. Plug the power supply to the autosampler back in
   ii. On the CRDS computer desktop, open the Autosampler Controller and press Chg Syringe button and wait for the syringe holder to move into position
   iii. Place syringe in syringe holder, making sure body and plunger are in holding locations
   iv. Press Swap Done button on Autosampler Controller

3. Fill out instrument log
   a. Open the sample log file on the desktop of the CRDS instrument computer
   b. Input the following information: date, time, initials, project name, number of samples, number of injections, N_2 pressure from the primary regulator on the N_2 tank
      i. N_2 pressure on the primary regulator should be > 150 psi. If the value is lower do not start the run. Wait for all instruments to finish their runs and then replace the tank will a full one. If no full spare tanks remain on the rack alert the SPATIAL facility manager who will order replacements.
      ii. N_2 pressure on the red, secondary regulator (on the bench top near the tanks) should be 2 (+/- 0.3) psi. If outside this range alert the SPATIAL facility manger. This value does not need to be recorded in the log.
   c. Look at the previous row to determine whether the septum should be changed – the septum should be changed every 300 injections. See section 5 for instructions on changing the septum. Indicate with an "X" if you have changed the septum or syringe.
4. Fill out sample ID file
   a. Open "Sample_list_template.csv" from desktop
   b. In the runfiles folder, save as "YYMMDD_project_HIDS20XX"
   c. Fill out template with sample IDs and project identifiers
5. Change septum
   a. The septum needs to be changed every 300 injections. Check run log to see if it needs to be replaced.
   b. Unscrew the injection port of the vaporizer, using fingers or tool that sits on top of the injection port to grasp and turn the ceramic top of the port. Caution: ceramic surface is warm, but metal parts at base of injection port will be hot.
   c. Remove old septum, replace with new septum. Check to see that new septum is centered.
   d. Replace injection port and hand-tighten
6. Check glass wool
   a. Make sure the glass wool container remains dry and no condensation is apparent in the vial. Check to make sure that the aquarium pump is working correctly and air is being forced through the opening at the top of the vial.

7. Check that background H_2O concentration reported in the data viewer window is below 100 ppm. If not, check that the N_2 tank regulator valve is open and the primary and secondary regulator pressures are correct (see #3).
8. Open Coordinator Launcher
    a. Don't press launch yet
    b. Make sure High Precision program ("AI High Precision" on some instruments) is selected
9. Open Autosampler Control
    a. Check that the following settings are correct:
        i. Row 1 is set for samples 1-4 and ten injections
        ii. Row 2 is set for samples 5-67 and four injections
        iii. If running less than 67 vials, update number in row 2 to match the position of the final sample in the autosampler tray
        iv. Method should be set as 'GJB rinse' (see Appendix)
    b. Click the Run button
10. After clicking Run in Autosampler Control, click the Launch button in Coordinator Launcher
11. Watch first few samples before leaving lab to ensure no problems at start of analysis
12. **Do not open OpenOffice files while run in progress, otherwise raw data will be lost and not saved to .csv file**

Part 3 – Data Processing

13. Run ChemCorrect
    a. After analysis has completed, close the Coordinator Launcher and Autosampler Control and open ChemCorrect from the desktop of the Picarro computer
    b. Click the Source button and navigate to the file with the correct date
    c. Click the 'OK' button to run ChemCorrect
    d. Examine the results to ensure that no samples show evidence for contamination, as indicated by red highlighting in the results window. Any samples flagged for contamination should be treated with activated charcoal and re-run.
        i. Uncap sample vial, add 0.5 mg activated charcoal, recap, and invert 3 times.
        ii. Allow treated sample to rest for 12 or more hours before re-analyzing.
        iii. If the same sample is flagged for contamination by ChemCorrect a second time, the sample should be transferred to the SIRFER lab for analysis by IRMS.
14. Process data
    a. Ensure that the data has been synced to the Dropbox folder and can be accessed from a computer with R installed on it.
    b. Open R and open the R script 'CRDS_liquid_3.R'
    c. Following the instructions in the R script, process the data and write an excel workbook with the data and quality assurance information. Also see the information in CRDS Data Processing R Scripts protocol.
    d. Examine the quality control and final sample data by running the appropriate functions in R and/or examining the information written to the excel workbook.

i. Quality control metrics for the run are compared against the criteria stored in the file "CRDS_liquid_parameters.csv". If any of the quality metrics have been violated, as indicated by the 'Ignore_Run' flag, troubleshooting will be necessary to determine why and all of the samples should be rerun. Examine the memory correction terms, the raw and memory-corrected values for the primary reference waters, the raw, memory-corrected, and drift-corrected values for the secondary reference water to determine what may have caused the quality parameter to be violated and correct for this before running samples again.

ii. Raw data for any individual samples that have violated the quality parameters, as indicated by an 'Ignore' flag, but was not flagged by ChemCorrect, should be reviewed manually.

1. Open the .csv instrument output file for the run and identify the analysis results for the four injections of the problem sample.
2. If one of the four injections is a clear outlier (> 3 standard deviations different from the other 3 in terms of H_2O concentration or either isotope ratio) the results for this injection can be deleted from the file. Highlight column "F" and all columns to the right for the row representing the outlier injection and delete the contents. Save the results file as a copy, appending "toAnalyze" at the end of the file name. No more than injection can be deleted for any given sample. Re-process the data file using the R scripts before exporting the results to the database and excel file.
3. If there is no clear outlier or if more than one injection appears to be an outlier the sample should be re-analyzed. If the sample has already been re-run twice, it should be transferred to SIRFER to be analyzed by IRMS.

e. After examining the data, run the appropriate functions in R to upload the data to the database and export an Excel results file.

Part 4 – Sample Disposal

15. Keep 2 mL sample vials in fridge for two weeks after returning data in case some need to be re-run. After this window, discard used sample vials in Broken Glass box.

Appendix – GJB_rinse autosampler method