

FINAL

# Quality Assurance Project Plan for BNSF ROW R2R, Coeur d'Alene, Idaho

Revision No. 1

Prepared for:



**Idaho Department of Environmental Quality**

**Waste and Remediation Division**

**State Office: 1410 N. Hilton, Boise, Idaho 83706**

**Coeur d'Alene Regional Office: 2110 Ironwood Parkway, Coeur d'Alene, ID 83814**

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September 23, 2016

IDEQ TRIM Record 2016BBD147

## Approval Page

### Quality Assurance Project Plan for

### BNSF ROW R2R, Coeur d'Alene, ID

IDEQ State Office: 1410 N. Hilton, Boise, Idaho 83706

Date: September 19, 2016

Note: This QAPP becomes effective on the date of the last approval signature.

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Date

\*Note: At the time of QAPP signature, the project QAO is required to update the IDEQ QAO project document tracker, found at 2012AEB8.

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## Acronyms and Abbreviations

bgs	below ground surface
BNSF	Burlington Northern Santa Fe Railway Company
CDA	Coeur d'Alene
CFR	Code of Federal Regulations
COC	chemical of concern
DQI	data quality indicator
DU	decision unit
EADA	asphalt emulsion dust suppressant
ESA	Environmental Site Assessment
ESC	ESC Laboratory Sciences
HAZWOPER	Hazardous Waste Operations Health and Emergency Response
IDAPA	Idaho Administrative Procedures Act
IDEQ	Idaho Department of Environmental Quality
IDTL	Initial Default Target Level
IDW	Investigation-derived Waste
ISM	Incremental Sampling Methodology
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
MS	matrix spike
MSD	matrix spike duplicate
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbon
PPE	personal protective equipment
QA	quality assurance
QAPP	Quality Assurance Project Plan
QAO	Quality Assurance Officer
QC	quality control
QMP	Quality Management Plan
RCRA	Resource Conservation and Recovery Act
ROW	Right of Way
RSL	Regional Screening Level
RUSL	Residential Use Screening Level
SIM	selected ion monitoring
SVL	SVL Analytical, Inc.
SVOC	semi-volatile organic compound
TerraGraphics	TerraGraphics Environmental Engineering, Inc.

TPH	Total Petroleum Hydrocarbon
TRC	TRC Environmental Solutions
USEPA	U.S. Environmental Protection Agency

## Units

g	gram
gal	gallon
mg/kg	milligram per kilogram
mm	millimeter
oz	ounce

## Section 1.0 Distribution List

The TerraGraphics Environmental Engineering, Inc. (TerraGraphics) Project Manager is responsible for distributing the final signed version of this document to the appropriate personnel. Table 1 shows the personnel to receive an electronic copy of the final signed Quality Assurance Project Plan (QAPP) and data validation reports.

**Table 1. Project Document Distribution List**

<b>Name</b>	<b>Address</b>	<b>Document Distribution</b>
<b>Title / Project Role</b>	<b>Phone</b>	
<b>Organization/Affiliation</b>	<b>Email</b>	
Don Zaroban, Ph.D IDEQ Quality Assurance Manager Director's Office	1410 N. Hilton Street Boise, ID 83706 (208) 373 - 0405 <a href="mailto:Don.Zaroban@deq.idaho.gov">Don.Zaroban@deq.idaho.gov</a>	QAPP (e-copy) Data (validation reports)
Eric Traynor IDEQ Program Manager State Office	1410 N. Hilton Street Boise, ID 83706 (208) 373 - 0565 <a href="mailto:Eric.Traynor@deq.idaho.gov">Eric.Traynor@deq.idaho.gov</a>	QAPP (e-copy) Data (validation reports)
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Melody Studer Project Manager TerraGraphics	988 S. Longmont Ave., Suite 200 Boise, ID 83706 (208) 336 - 7080 <a href="mailto:Melody.Studer@terragraphics.com">Melody.Studer@terragraphics.com</a>	QAPP (e-copy) Data (validation reports)
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Jon Munkers Principal in Charge TerraGraphics	988 S. Longmont Ave., Suite 200 Boise, ID 83706 (208) 336 - 7080 <a href="mailto:Jon.Munkers@terragraphics.com">Jon.Munkers@terragraphics.com</a>	QAPP (e-copy) Data (validation reports)



**Table 1. Project Document Distribution List**

Name	Address	
Title / Project Role	Phone	Document Distribution
Organization/Affiliation	Email	
Christine Meyer Laboratory Contact SVL Analytical, Inc.	One Government Gulch PO Box 929 Kellogg, ID 83837 (208) 784 - 1258 <a href="mailto:chris@svl.net">chris@svl.net</a>	QAPP (e-copy with highlighted areas pertaining to SVL)
Shane Gambill Laboratory Contact ESC Laboratory Sciences	12065 Lebanon Rd Mt Juliet, TN 37122 (615) 773 - 9747 <a href="mailto:sgambill@esclabsciences.com">sgambill@esclabsciences.com</a>	QAPP (e-copy with highlighted areas pertaining to ESC)

## Section 2.0 Project/Task Organization

Figure 1 provides an organizational chart. Table 2 defines the key project personnel and their responsibilities.

Figure 1. Project Organizational Chart

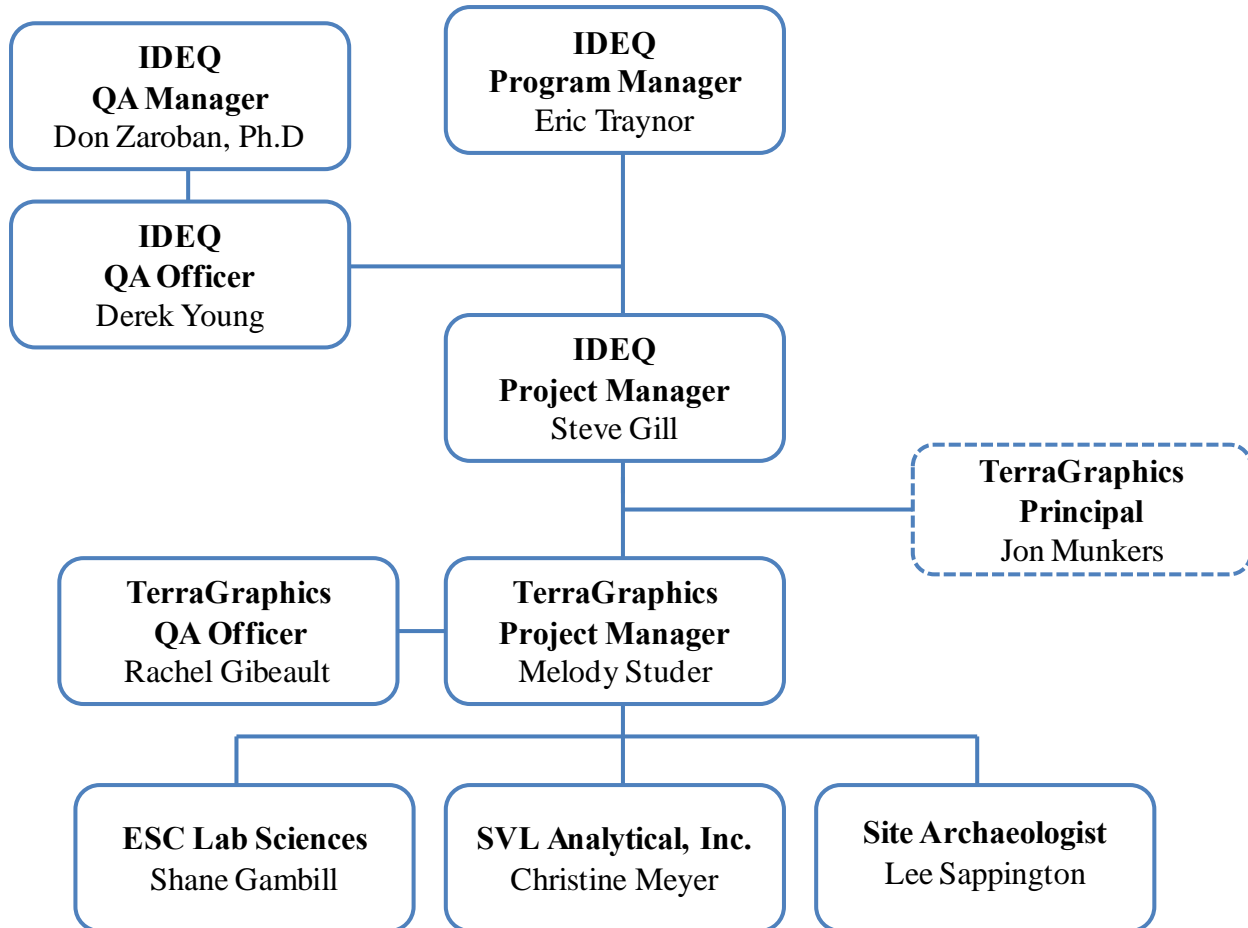


Table 2 is a summary of the project staff duties and responsibilities; refer to sections 1.2.5 through 1.2.7 of the Idaho Department of Environmental Quality (IDEQ) Quality Management Plan (QMP) (IDEQ 2012) for a more detailed description.

**Table 2. Key Project Personnel and Associated Responsibilities**

Name	Project Title/Responsibility
Eric Traynor	<p><b>IDEQ Program Manager:</b> Note: The following description is <i>not all inclusive</i>; see section 1.2.7 of the IDEQ QMP for a more detailed description. This person is the regional manager or State Office program manager for the project. Duties and responsibilities include:</p> <ul style="list-style-type: none"> <li>• Assists in the review of the QAPP and signs the final QAPP as an approver.</li> <li>• Confirms the project QAPP meets the needs of the program/region.</li> <li>• Ensures the QAPP is approved prior to the start of project work.</li> <li>• Ensures the program/regional procedures and policies referenced in the QAPP are current and approved for use.</li> <li>• Performs all duties and responsibilities as assigned in the project QAPP.</li> <li>• Selects and assigns a project quality assurance officer (QAO), who meets the criteria for independence defined in the IDEQ QMP (see QAO duties below), and obtains approval for this selection from the IDEQ quality manager.</li> </ul>
Derek Young	<p><b>IDEQ Project QAO:</b> Note: The following description is <i>not all inclusive</i>; see section 1.2.5 of the IDEQ QMP for a more detailed description. Each project has an assigned QAO, whose duties and responsibilities include:</p> <ul style="list-style-type: none"> <li>• Assists in the review of the QAPP, verifies the QAPP meets the requirements of the IDEQ QMP, and signs the QAPP as an approver.</li> <li>• All assigned IDEQ QAOs are required to contact the IDEQ quality manager to discuss the project prior to signing any project QAPP for approval. When the IDEQ project QAO signs the QAPP for approval, the IDEQ QAO is required to update the IDEQ QAO project document tracker found at TRIM record #2015BBD142.</li> <li>• Performs an annual audit on all assigned projects to evaluate project compliance with the approved project QAPP. Files the completed audit checklist in TRIM to document the audit.</li> <li>• Participates in final project report review.</li> <li>• Documents all audit and data validation activities in the IDEQ TRIM system, per the IDEQ QMP and the approved QAPP.</li> <li>• In matters of project quality, this individual has a direct line of communication to the IDEQ quality manager and will communicate issues to the project manager.</li> <li>• Must meet the following independence criteria: The QAO shall not be the project manager or program manager, or be otherwise assigned to the project data generation efforts. Neither the project manager nor the QAO may directly report to the other within the IDEQ organizational structure, and both of these individuals may not be directly supervised by the same person.</li> <li>• Performs all other duties and responsibilities as assigned in the project QAPP.</li> </ul>

**Table 2. Key Project Personnel and Associated Responsibilities**

Name	Project Title/Responsibility
Rachel Gibeault	<p><b>TerraGraphics QAO:</b></p> <ul style="list-style-type: none"> <li>• Reports to TerraGraphics' Project Manager, Melody Studer.</li> <li>• Responsible for reviewing and approving the project QAPP and amendments, and completing data review, validation, and verification.</li> <li>• Will also be part of the review team for project final reports.</li> <li>• Shall not be the Project Technical Lead, the Project Manager, or the Program Manager, or be otherwise assigned to the project data generation efforts.</li> </ul>
Steve Gill	<p><b>IDEQ Project Manager:</b> Note: The following description is <i>not all inclusive</i>; see section 1.2.6 of the IDEQ QMP for a more detailed description. Each project has an assigned project manager, whose duties and responsibilities include:</p> <ul style="list-style-type: none"> <li>• Assists with preparation of the project QAPP, and signs the final QAPP as an approver.</li> <li>• Performs overall project planning, document development and approval, sample planning and coordination, laboratory coordination, reporting functions, project report/summary development, and project file maintenance in TRIM.</li> <li>• Enters the approved and current project QAPP in the TRIM system, including a copy of the signed approval page.</li> <li>• Ensures all project work is conducted in accordance with the IDEQ QMP, the approved QAPP, and the applicable project operating procedures.</li> <li>• Ensures that personnel assigned to this project are appropriately trained and qualified, with the corresponding training records on file in human resources.</li> <li>• Reviews the project QAPP annually to determine if revision is necessary. If the project QAPP requires revision, the project manager initiates such action. All such documents will be revised, reviewed, and approved in accordance with the IDEQ QMP.</li> <li>• Documents all audit and data review/verification activities in the IDEQ TRIM system, per the IDEQ QMP and approved QAPP.</li> <li>• Performs all other duties and responsibilities as assigned in the project QAPP.</li> </ul>
Melody Studer	<p><b>TerraGraphics Project Manager:</b></p> <ul style="list-style-type: none"> <li>• Reports to IDEQ Program Manager and IDEQ Project Manager and TerraGraphics' Principal in Charge, Jon Munkers.</li> <li>• Responsible for overall project planning, document development and approval, sample planning and coordination, laboratory coordination, data review and verification, reporting functions, and project report/summary development as required by the project documents.</li> <li>• Responsible for ensuring all project work is conducted in accordance with the approved project QAPP.</li> <li>• Responsible for ensuring that personnel assigned to this project are appropriately trained and qualified.</li> <li>• Will also be part of the review team for project final reports.</li> </ul>
Shane Gambill (ESC) Chris Meyer (SVL)	<p><b>Laboratory Contact/Manager:</b> Primary laboratory contacts for TerraGraphics' project staff.</p> <ul style="list-style-type: none"> <li>• Responsible for ensuring all laboratory work is conducted in accordance with the approved project QAPP.</li> </ul>

## **Section 3.0 Problem Definition / Background**

A Phase I Environmental Site Assessment (ESA) completed by TerraGraphics in 2015 uncovered several recognized environmental conditions at the Burlington Northern Santa Fe Railway Company (BNSF) Right of Way (ROW), Riverstone to Huetter section (TerraGraphics 2015). The Phase I ESA divided the ROW into three Zones based upon historical industrial uses (Figure 2). All Zones have a history of railroad use for more than 100 years. In the early 1900s there were hourly electric train services along this corridor linking Spokane, Washington, to the City of Coeur d'Alene (CDA), Idaho. The region also has a long history of heavy metal mining and rail distribution.

TRC Environmental Solutions (TRC) conducted previous limited assessment of the ROW for BNSF, included sampling of shallow sub-surface soils for Resource Conservation and Recovery Act (RCRA) 8 metals, Total Petroleum Hydrocarbons (TPHs), and limited polycyclic aromatic hydrocarbons (PAHs) from the 18- to 24-inch depth interval. Metals concentrations from soil samples collected from the 18- to 24-inch depth interval were above Initial Default Target Levels (IDTLs).

In Zones 1, 2 and 3, the Phase I ESA identified recognized environmental conditions for heavy metals and PAHs from historical rail activity. As a result of the past railroad transportation, loading, and unloading operations, the surface soils within the Zones have potential to be contaminated with elevated amounts of PAHs, semi-volatile organic compounds (SVOCs), and heavy metals. TerraGraphics recommended additional characterization for surface soils for RCRA 8 metals, PAHs, and SVOCs to evaluate the extent of contamination and evaluate threats to human health and the environment.

### **3.1 State the Problem**

On May 28, 2015, the City and Ignite CDA, the City's urban renewal agency, purchased the BNSF ROW property. IDEQ requested TerraGraphics perform additional characterization activities on the ROW Site for use in a human health risk assessment focused on currently identified future redevelopment goals and land use. The City envisions the conversion of the former rail ROW to a pedestrian-bicycle path along the CDA river frontage. The City has strong interest in, and public support for, redeveloping this property for a public pedestrian and/or bike trail, green space and public waterfront access. The corridor contains some of the last remaining opportunities for public access to the Spokane River in CDA.

### **3.2 Intended Usage of Data**

The purpose of this assessment is to delineate the extent of RCRA 8 metals, PAHs, and SVOCs within the three Zones of the BNSF ROW that may have been released from former rail and heavy industrial operations and deposited into the shallow subsurface (less than 12 inches below ground surface [bgs]). TerraGraphics will only evaluate SVOCs within Zone 2 based on specific historical locations of a trestle and bone yard noted in the Phase I ESA (TerraGraphics 2015). TerraGraphics will further sub-divide each zone into 17 separate parallel decision units (DUs) based on historical grade elevations and evaluate them based on their historical use (Figures 2 through 5).

The DUs are as follows:

<b>Zone 1</b>	<b>Zone 2</b>	<b>Zone 3</b>
DU 1.1	DU 2.1 (A, B, C)	DU 3.1 (A, B, C)
DU 1.2	DU 2.2 (A, B, C)	DU 3.2 (A, B, C)
DU 1.3 (A, B, C)		

Based on historical Site investigations, the chemicals of concern (COCs) at the Site are as listed as follows:

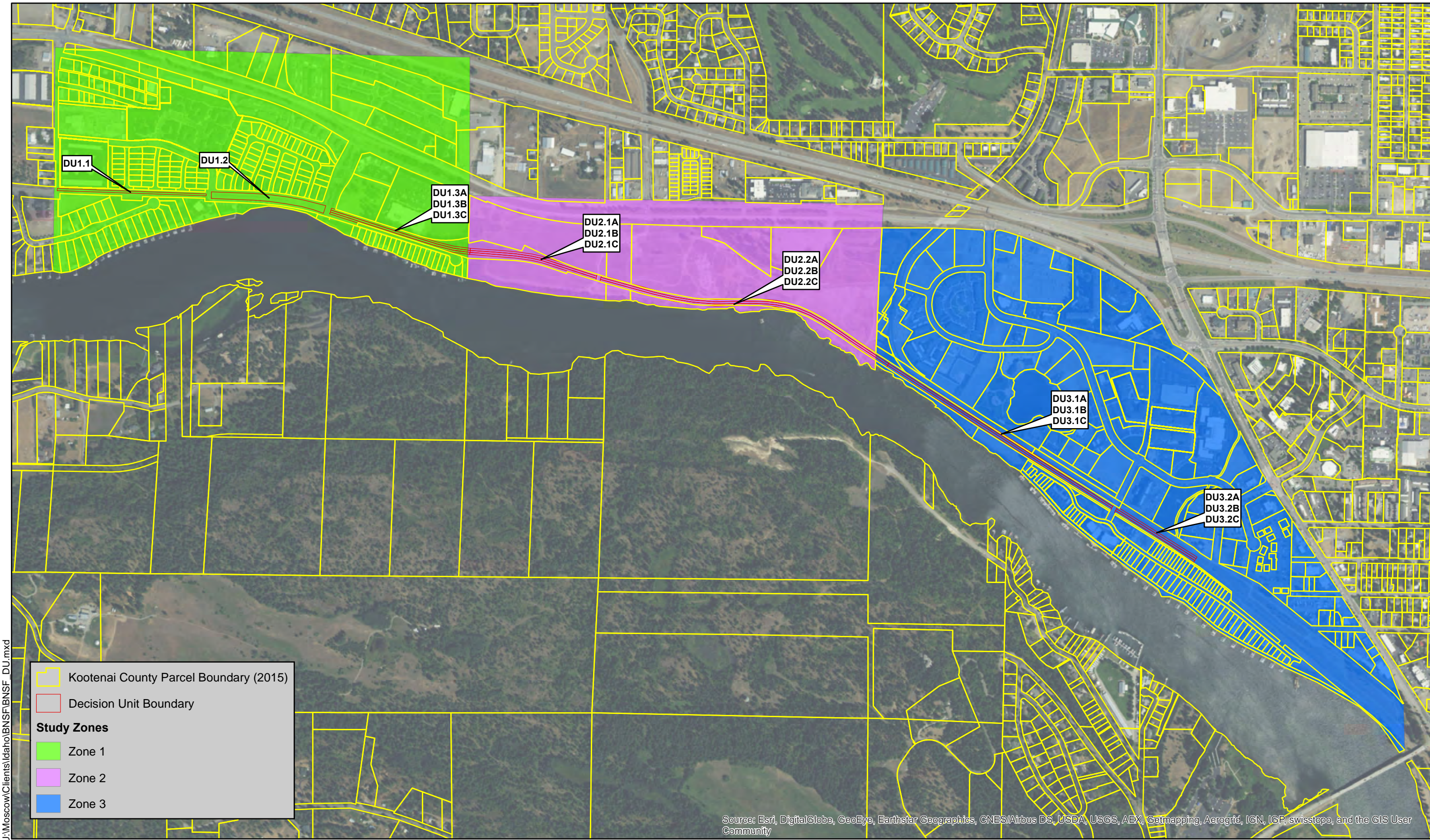
- PAHs, which include: Anthracene, Acenaphthene, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Chrysene, Fluoranthene, Fluorene, and Pyrene.
- RCRA 8 metals, which include: Arsenic, Barium, Cadmium, Chromium, Lead, Selenium, Silver, and Mercury.
- SVOCs (only within DU 2.2A, 2.2B, and 2.2C), which include: Acenaphthylene, Benzidine, Benzo(g,h,i)perylene, Bis(2-chlorethoxy)methane, Bis(2-chloroethyl)ether, Bis(2-chloroisopropyl)ether, 4-Bromophenyl-phenylether, 2-Chloronaphthalene, 4-Chlorophenyl-phenylether, Dibenz(a,h)anthracene, 3,3-Dichlorobenzidine, 2,4-Dinitrotoluene, 2,6-Dinitrotoluene, Hexachlorobenzene, Hexachloro-1,3-butadiene, Hexachlorocyclopentadiene, Hexachloroethane, Indeno(1,2,3-cd)pyrene, Isophorone, Naphthalene, Nitrobenzene, n-Nitrosodimethylamine, n-Nitrosodiphenylamine, n-Nitrosodi-n-propylamine, Phenanthrene, Benzylbutyl phthalate, Bis(2-ethylhexyl)phthalate, Di-n-butyl phthalate, Diethyl phthalate, Dimethyl phthalate, Di-n-octyl phthalate, 1,2,4-Trichlorobenzene, 4-Chloro-3-methylphenol, 2-Chlorophenol, 2,4-Dichlorophenol, 2,4-Dimethylphenol, 4,6-Dinitro-2-methylphenol, 2,4-Dinitrophenol, 2-Nitrophenol, 4-Nitrophenol, Pentachlorophenol, Phenol, and 2,4,6-Trichlorophenol.

TerraGraphics will compare the PAH results to Residential Use Screening Levels (RUSLs) taken from the Idaho Administrative Procedures Act (IDAPA) Application of Risk Based Corrective Action at Petroleum Release Sites (IDAPA 58.01.24) rules.

TerraGraphics will compare RCRA 8 metal and SVOC concentrations in Site soil to IDEQ's Risk Evaluation Manual IDTLs (IDEQ 2004). The U.S. Environmental Protection Agency (USEPA) Regional Screening Levels (RSLs) for Resident Soil or Resident Soil to Groundwater (USEPA 2015, 2016, respectively) may be used for comparison depending on the constituent and risk pathway evaluated. For example, if the IDTL screening level is based on a critical pathway that is incomplete, then the most appropriate RSL with a potentially complete pathway will be compared to concentration data.

TerraGraphics will also compare metal concentrations to Washington Statewide metal background levels (Ecology 1994) and Oregon Statewide metal background levels (ODEQ 2013) where available. Background levels are considered due to previous site assessments showing elevated metal concentrations that are not necessarily associated with Site use and possibly more related to naturally occurring element concentrations.

Appendix A provides a Site-specific summary of IDTLs and Washington and Oregon state background metal values for constituents analyzed during this assessment.



U:\Moscow\Clients\idaho\BNSF\BNSF\_DU.mxd

Kootenai County Parcel Boundary (2015)  
 Decision Unit Boundary  
**Study Zones**  
 Zone 1  
 Zone 2  
 Zone 3

Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AEX, Getmapping, Aerogrid, IGN, IGP, swisstopo, and the GIS User Community

**TerraGraphics**  
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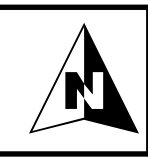
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August 18, 2016  
PROJECT NUMBER  
15029-01

REQUESTOR  
M. Studer  
PROJECT MANAGER  
M. Studer  
CARTOGRAPHER  
B. Bailey

PROJECT NAME  
**BNSF ROW R2R  
Coeur d'Alene, Idaho**

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1:10,000  
1 inch = 833 feet  
0 500 1,000 Feet



**Figure 2**

**Site Layout  
Showing Decision Units and Zones**

U:\Moscow\Clients\idaho\BNSF\BNSF\_Zone1.mxd



- Study Zone 1 Boundary
- Kootenai County Parcel Boundary (2015)
- Decision Unit Boundary

Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AEX, Getmapping, Aerogrid, IGN, IGP, swisstopo, and the GIS User Community



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August 18, 2016  
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15029-01

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CARTOGRAPHER  
B. Bailey

PROJECT NAME  
**BNSF ROW R2R  
Coeur d'Alene, Idaho**

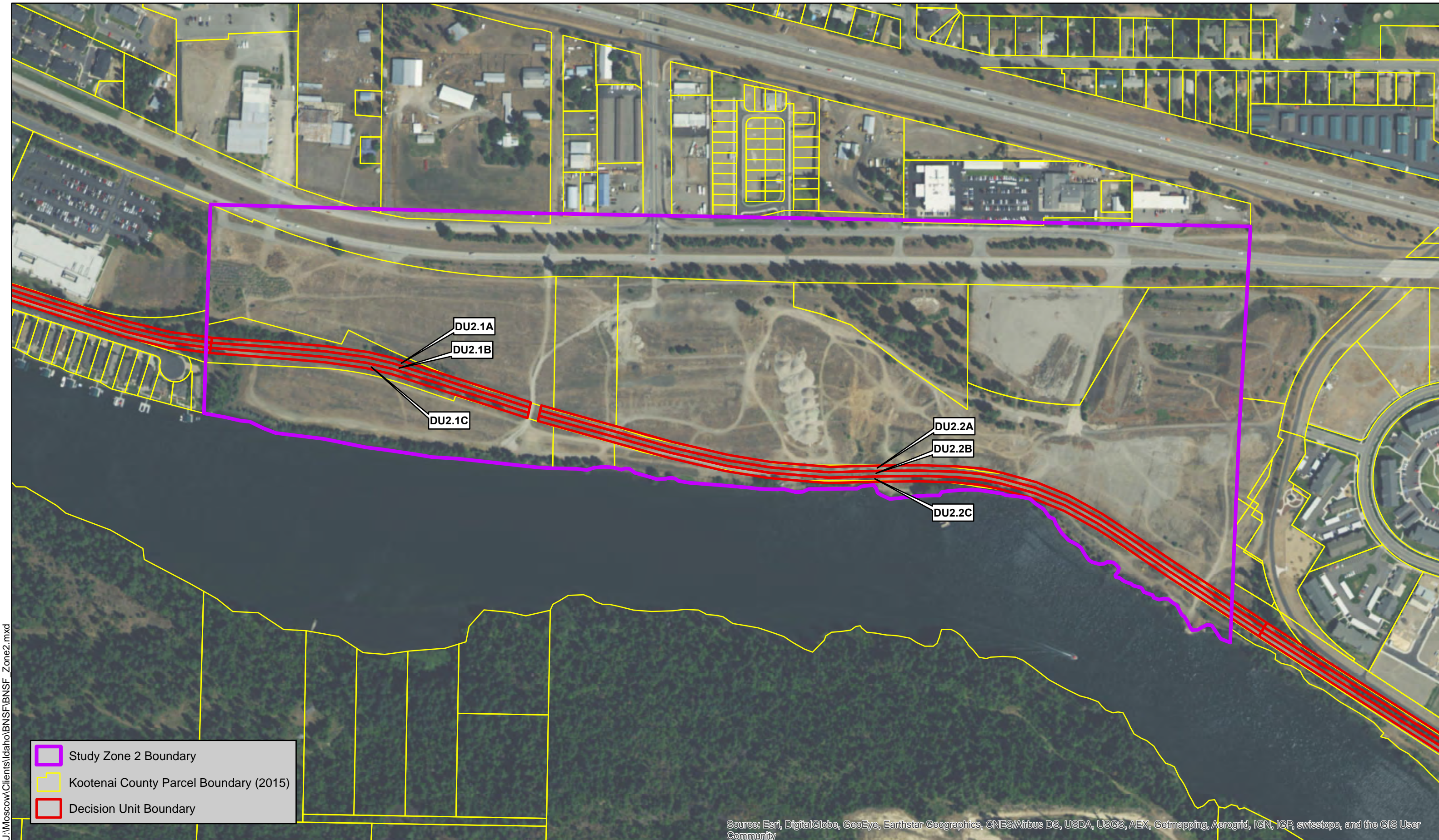
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1 inch = 333 feet  
0 200 400 Feet



**Zone 1 Decision Unit** Figure 3





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- Study Zone 2 Boundary
- Kootenai County Parcel Boundary (2015)
- Decision Unit Boundary

Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AEX, Getmapping, Aerogrid, IGN, IGP, swisstopo, and the GIS User Community

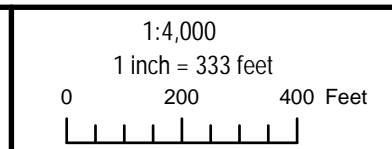


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M. Studer  
CARTOGRAPHER  
B. Bailey

PROJECT NAME  
**ROW R2R  
Coeur d'Alene, Idaho**




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**Figure 4**  
**Zone 2 Decision Unit**

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-  Study Zone 3 Boundary
-  Kootenai County Parcel Boundary (2015)
-  Decision Unit Boundary

Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AEX, Getmapping, Aerogrid, IGN, IGP, swisstopo, and the GIS User Community



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PROJECT NUMBER  
15029-01

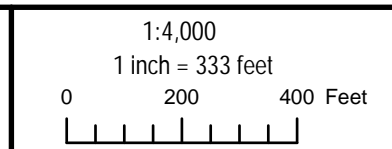
REQUESTOR  
M. Studer

PROJECT MANAGER  
M. Studer

CARTOGRAPHER  
B. Bailey

PROJECT NAME  
**ROW R2R  
Coeur d'Alene, Idaho**

This map was produced using information obtained from several different sources that have not been independently verified. These sources have also not provided information on the precision and accuracy of the data. Information on this map is not a substitute for survey data.



**Zone 3 Decision Unit** Figure 5

## Section 4.0 Project / Task Description

### 4.1 General Overview of the Project

TerraGraphics will collect soil samples for COC analysis. Sample data will be used to evaluate the extent of contamination remaining from historical use. TerraGraphics will collect and handle samples in accordance with the soil sampling and handling methodologies described in Sections 9.0 and 10.0. TerraGraphics will submit the samples to ESC Lab Sciences (ESC) and SVL Analytical, Inc. (SVL) for analyses. All onsite activities will comply with the Site-specific Health and Safety Plan, included in Appendix B.

Project objectives are as follows:

1. Determine the status of RCRA 8 metal, SVOC, and PAH COC concentrations in the surface soil (0-12 inches bgs) of the 17 DUs in an attempt to locate potential sources of contamination (from historical use and residual contamination).

#### 4.1.1 Archaeological Site Observations

TerraGraphics will use direct push technology to assess Site soil. Based on the location of the Site and its extensive use throughout history, TerraGraphics may encounter historic and prehistoric cultural material during the Site activities. Therefore, an archaeologist will be onsite during sampling to observe direct push technology activities, record the location of sampling locations, and examine the soil by screening intermittent samples through a ¼-inch mesh hardware cloth. All historic and prehistoric cultural material encountered during subsurface exploration will be noted, recorded, photographed (if considered diagnostic), and then returned to original units prior to backfilling. If Site activities encounter concentrations of artifacts, sampling in that area will cease until it can be properly recorded. If human remains are encountered, all sampling will immediately cease in that area and no further sampling will occur there; State Historic Preservation Office, Tribal, law enforcement, and other appropriate parties will be notified by TerraGraphics and IDEQ.

### 4.2 Project Timetable

Table 3 presents the approximate schedule for the major project activities, such as field sampling, data review, and report generation.

**Table 3. Project Schedule**

Task	Dates	Comments
QAPP Review and Approval	September 2016	Approved prior to field work.
Field Work	October 2016	Scheduled October 3 <sup>rd</sup> through October 7 <sup>th</sup> .
Shipment to Laboratories	Immediately following field work	Immediately following field work activities.
ESA Report	Anticipated November 2016	Delivered no later than 4 weeks following receipt of laboratory data.

## Section 5.0 Quality Objectives and Criteria for Measurement Data

This project's data collection efforts and intended use of the data are based on the systematic planning efforts of IDEQ and TerraGraphics. A primary goal of this assessment is to obtain analytical results representative of the physical and chemical compositions of the surface soil at the Site. To assure that data quality is sufficient to achieve this goal, sampling, analyses, and data review will follow strict protocols. TerraGraphics' Project Manager and QAO will guide all stages of the project to ensure proper methodology has been followed. In addition, TerraGraphics will assess the data quality using the following data quality indicators (DQIs), defined in Table 4.

The analytical laboratories, ESC and SVL, provided estimated reporting limits and method detection limits for COCs (Tables 6 and 7 and Appendix A). Actual reporting limits may differ slightly and will depend on sample dilution, sample concentrations, sample matrix effects, and analytical procedures.

**Table 4. Data Quality Indicators (2 pages)**

<b>DQI</b>	<b>Definition</b>	<b>Evaluation Procedure</b>
Precision	A measure of data variation quantified by repeating measurements of a characteristic on a single sample or co-located sample set.	Evaluated based on field duplicate samples, laboratory duplicates, laboratory control sample duplicates (LCSD), and matrix spike duplicates (MSD). Appendix C lists analyte-specific precision criteria and goals in terms of relative percent difference (RPD).
Accuracy / Bias	A measure of the closeness of the agreement between a true, or reference, value and the associated measured value.	Quantified by testing field and laboratory samples spiked with a known concentration of a known analyte and will be evaluated based on laboratory control sample (LCS), matrix spike (MS), and surrogate recoveries. Appendix C lists analyte-specific accuracy criteria
Sensitivity	The capability of a method or instrument to discriminate between measurement responses representing different level (e.g., concentrations) of a variable of interest.	Comparing the reporting limits or the method detection limits to demonstrate that where possible, they are at or below the regulatory standards: IDTLs, RUSLs, and USEPA RSLs (Appendix A).
Representativeness	The degree to which the sample data accurately and precisely represent Site conditions. It is best satisfied by confirming that sample locations are appropriately defined, sample collection procedures are appropriate and consistently followed, a sufficient number of samples are collected, and analytical results meet the DQIs.	Representativeness is evaluated based on data review, verification, validation, and reconciliation efforts by comparing the combination of data accuracy, precision, appropriate measurement range, and methods, and assessing other potential sources of bias including sample holding times, results of blank samples, and laboratory quality assurance (QA) review.

**Table 4. Data Quality Indicators (2 pages)**

Reproducibility / Comparability	<p>Comparability is a qualitative expression of the measure of confidence that two or more data sets may contribute to a common analysis. Using standard USEPA accepted protocols, TerraGraphics will collect the samples and measure field parameters for comparison with previous project data, if available.</p>	<p>The laboratories will process and analyze all samples according to this QAPP at sufficient detection limits, precision, accuracy, and reproducibility (the closeness of agreement between independent results obtained with the same method on identical test material but under different conditions). Previous data that are comparable are shown in Table 12 of this QAPP.</p>
Completeness	<p>The percentage of valid data relative to the total possible data points. A measure of whether enough samples were obtained to support the decision to be made.</p>	<p>Evaluated for each analyte based on the total number of project samples, excluding QA/quality control (QC) samples. TerraGraphics' goal for completeness is <math>\geq 90\%</math>.</p>

## Section 6.0 Special Training Requirements / Certification

TerraGraphics' field personnel performing sampling activities at the Site will have completed a minimum of an Occupational Safety and Health Administration (OSHA) 24- or 40-hour Hazardous Materials Technician course, in compliance with Code of Federal Regulations (CFR) Title 29 Part 1910.120: OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER), and have current certification. Documentation of necessary training and certifications is available upon request.

Laboratories performing the analyses will be part of the National Environmental Laboratory Accreditation Program.

## Section 7.0 Documentation and Records

### 7.1 Field Documentation

The TerraGraphics Project Manager is responsible for ensuring that the most current approved revision of the project QAPP is available to IDEQ and the field personnel.

Sampling personnel will document each day's activities in field notebooks. Field staff will record information as follows:

- Record project data directly, promptly, and legibly.
- Make field logbook or field sheet entries in permanent ink and sign/initial and date by the data entrant.
- Indicate changes or corrections to data with a single line through the original entry.
- Initial, date, and explain changes.
- Include a reference to the sampling procedures in the field notes.

TerraGraphics developed a sample numbering scheme to allow each sample to be uniquely identified and to provide a means of tracking the sample from collection through analysis. The numbering scheme indicates the Site location and DU number. The unique sample number will be entered in the field notebook, chain-of-custody forms, and other records documenting sampling activities.

## **7.2 Laboratory Data Management and Storage**

TerraGraphics will store the data in electronic form and retain the data on a local server. The laboratories will email analytical data to TerraGraphics as Microsoft® Excel® and PDF files. TerraGraphics will provide laboratory result reports to IDEQ in an appendix to the ESA Report (Sections 17.0 and 18.0). IDEQ will store all project files, including this QAPP, the laboratory result reports, and data summary report, in its TRIM document management system.

## **Section 8.0 Sampling Process Design (Experimental Design)**

This section describes the project data collection activities, assumptions, sampling Site selection, the number of samples to be obtained and the number of sampling locations, whether samples are to be individually handled or composited, and any other relevant project-specific information. TerraGraphics will present soil sample results in an ESA Report.

### **8.1 Rationale for Selection of Sampling Sites**

Sample planning requires understanding the layout and possible contaminants associated with the CDA BNSF ROW Site. The primary use of this Site has been historical railroad use since the early 1900s. Three parallel railroad beds run the length of the site and vary in elevation, with potential differing historical impacts. Based on this conceptual understanding and the purposes of performing a Site investigation/characterization, the sampling process design will include dividing the area into 17 DUs based on specified areas within each of the three Zones. Table 5 describes the DU location, the number of samples collected, and COCs targeted. Figure 2 shows DU locations across the Site, with Figures 3 through 5 focusing on each Zone.

DU1.1 runs from Huetter Avenue to W. Shoreview Lane and is 1,400 feet long by 20 feet wide. North of DU1.1 will remain an active BNSF line. In the future, this DU will be paved for public access and limited parking for Johnson Mill Park.

DU1.2 is 1,100 feet in length by 60 feet wide and runs parallel to Johnson Mill Park. Future land use plans remove the southern one-way road and create a two-way road by joining with the northern one-way (west) road. Once removed, the area previously occupied by the southern road would be redeveloped as a public park and green space.

DUs 1.3A, 1.3B, and 1.3C are each 1,400 feet long and 20 feet wide.

DUs 2.1A, 2.1B, and 2.1C are each 1,250 feet long and 20 feet wide.

DUs 2.2A, 2.2B, and 2.2C as well as DUs 3.1A, 3.1B, and 3.1C are each 2,950 feet long and 20 feet wide.

DUs 3.2A, 3.2B, and 3.2C are each 900 feet long and 20 feet wide.

Reasoning for creating the specific DUs (1.3[A,B,C], 2.1[A,B,C], 2.2[A,B,C], 3.1[A,B,C], and 3.2[A,B,C]) is based on differing elevation topography and past uses. The southern 20 feet was an historical old vehicle spur road, and possibly rail bed, (C- labels within the DUs); the middle 20 feet is a former rail bed track elevated with ballast (B-labels within the DUs); and the northern 20 feet currently consists of possible former rail bed that is now vegetated (A-labels within the DUs). Future land use plans include trails, possibly mix of paved surfaces and vegetation.

## **8.2 Sample Design Logistics**

### **8.2.1 Utility Marking**

TerraGraphics will notify the one-call agency (Utility Notification Center) at least five days prior to field activities. The appropriate utility companies will mark the utilities at locations where the underground lines enter the Site.

### **8.2.2 Random Sample Location Generator**

As described in the Incremental Sampling Methodology (ISM) guidance Section 5.3, Field Collection (ITRC 2012), TerraGraphics will randomly generate subsample locations. TerraGraphics will generate the random points in ArcGIS using a pre-programmed procedure named "Create Random Points," which places points based on a pre-determined area, the number of points desired, and a minimum distance of 5 feet between the points (with an error of 5-10 feet) to prevent overlapping subsample locations. Once TerraGraphics separately generates each set of points for a DU, then they combine all DUs into one GIS layer with numbered points, and calculated coordinates in both feet and decimal degrees. They then export the completed file to an Excel table or import them into a Garmin GPS unit for use in the field. If these pre-determined subsample locations cannot be collected due to safety or obstruction (i.e., the presence of trees, concrete, or surface debris), TerraGraphics will determine a new subsample location from a second set of random locations generated as a backup for these specific cases (Appendix F). The field crew will have access to data sheets noting the GPS coordinates of the subsample locations for each DU (Appendix F). The field crew will mark with pin flags each subsample location in the field using the GPS unit at the beginning of each ISM sampling round.

### **8.2.3 General Incremental Sampling Methodology Logistics**

The sampling design will consist of the ISM in all DUs generally described below, shown in Table 5, and provided in more detail in Section 9.1:

- TerraGraphics' field crew will collect one sample (created from 30 subsamples within each DU) from each of the 17 DUs. Tables 9 and 10 discuss additional samples collected for QA purposes.
  - The field crew will have access to data sheets noting the GPS coordinates of the subsample locations for each DU.
  - The field crew will collect subsamples (sample increments) from the surface (0 inches to a maximum depth of 12 inches bgs) using direct push methods. The field crew will collect an equal volume of soil from each direct push soil core.
  - The field crew will first work the core through a 1/4-inch (6.35 millimeter

- [mm]) sieve to break up the soil core and remove larger debris, then through a #10 sieve (2.00 mm) in the field to better filter and homogenize the samples.
- Field crews will place the soil from each DU into a dedicated, new, clean, and disposable plastic bucket for homogenization. Section 9.1 describes details of homogenization and sampling techniques the field crew will use.
  - TerraGraphics' field crew will collect a DU sample from the homogenized soil (for a total of 17 samples plus additional samples collected for QA purposes) and send them to SVL where the samples will be dried, sieved, and analyzed for RCRA 8 metals by USEPA Method 6010C (USEPA 1996b). Mercury will be analyzed by USEPA Method 7471B (USEPA 2007).
  - TerraGraphics' field crew will collect a DU sample from the homogenized soil (for a total of 17 samples plus additional samples collected for QA purposes) and send them to ESC where the samples will be analyzed for PAHs by USEPA Method 8270D-selected ion monitoring (SIM) (USEPA 1996a).
  - In DUs 2.2A, 2.2B, and 2.2C, TerraGraphics' field crew will collect a DU sample from the homogenized soil (for a total of three samples plus additional samples collected for QA purposes) and send them to ESC where they will be analyzed for the full suite of SVOCs by USEPA Method 8270D (USEPA 1996a).

#### **8.2.4 Special Case Sample Logistics**

TerraGraphics will also collect one discrete surficial soil sample from DU3.1B to assess potential COCs present due to an asphalt emulsion dust suppressant (known as EADA) recently sprayed by the City of CDA street department. Sample numbers are shown in Table 5, and sample methods are provided in more detail in Section 9.1:

- TerraGraphics' field crew will fill one 1-gallon (gal) plastic, resealable (Ziploc) bag with surficial soil (from 0 inches to a maximum depth of 12 inches bgs) and send it to SVL where it will be analyzed for RCRA 8 metals by USEPA Method 6010C (USEPA 1996b). Mercury will be analyzed at SVL by USEPA Method 7471B (USEPA 2007). This discrete sample will provide information regarding RCRA 8 COC concentrations in the EADA-affected surface soil.
- TerraGraphics' field crew will fill one 4-ounce (oz) glass jar with surficial soil (from 0 inches to a maximum depth of 12 inches bgs) and send it to ESC where it will be analyzed for PAHs by USEPA Method 8270D-SIM (USEPA 1996a). This discrete sample will provide information regarding PAH COC concentrations in the EADA-affected surface soil.

TerraGraphics will also collect one discrete surficial soil sample within either DU3.1A or DU3.1C (known to not have EADA sprayed on the surface) for comparison purposes to the sample known to contain EADA. The field crew will determine and record the sample location in the field.

- TerraGraphics' field crew will fill one 4-oz glass jar and ship to SVL where it will be analyzed for RCRA 8 metals by USEPA Method 6010C (USEPA 1996b). Mercury will be analyzed at SVL by USEPA Method 7471B (USEPA 2007).
- TerraGraphics' field crew will fill one 4-oz glass jar and ship to ESC where it will be analyzed for PAHs by USEPA Method 8270D-SIM (USEPA 1996a).



## 8.2.5 Sample Identification

The following field identification for sample numbering will have two components as follows:

- ID-DU#
- Where:
  - ID = CDA BNSF ROW
  - DU# = Decision Unit (decision unit 1 = DU1.1, decision unit 2 = DU2.1A, etc.)  
(discrete samples will note ID-DU#-EADA)

**Table 5. Sample Type, Amount, and Location Information**

Sample Matrix and Sample Depth	Number of Samples	Site Area	DU within each Zone	Target COC	Subsample
Soil 12 in. bgs	Up to 17 total samples (1 sample from each DU, plus QA samples as shown in Table 9)	Zone 1	DU1.1	PAHs, RCRA 8 metals	30
			DU1.2		30
			DU1.3A		30
			DU1.3B		30
			DU1.3C		30
		Zone 2	DU2.1A	PAHs, RCRA 8 metals	30
			DU2.1B		30
			DU2.1C		30
			DU2.2A	PAHs, RCRA 8 metals, SVOCs	30
			DU2.2B		30
			DU2.2C		30
		Zone 3	DU3.1A	PAHs, RCRA 8 metals	30
			DU3.1B		30
			DU3.1C		30
			DU3.2A		30
DU3.2B	30				
DU3.2C	30				
Soil Within 12 in. bgs	1 discrete sample	Zone 3	DU3.1B (known EADA spray location)	PAHs, RCRA 8 metals	N/A
Soil Within 12 in. bgs	1 discrete sample	Zone 3	DU3.1 A or DU3.1C (non-EADA spray location)	PAHs, RCRA 8 metals	N/A

**Table 6. Analytes, Screening Levels, and Reporting Limits of Site Soil Samples (PAHs and SVOCs) (2 pages)**

Method	Analyte	IDEQ IDTL <sup>a</sup> /RUSL <sup>b</sup> (mg/kg)	ESC Method Detection Limit (mg/kg)	ESC Reporting Limit (mg/kg)
PAHs using USEPA Method 8270D-SIM	Acenaphthene	200 <sup>b</sup>	0.0006	0.006
	Anthracene	3,200 <sup>b</sup>	0.0006	0.006
	Benz(a)anthracene	0.09 <sup>b</sup>	0.0006	0.006
	Benzo(a)pyrene	0.02 <sup>b</sup>	0.0006	0.006
	Benzo(b)fluoranthene	0.19 <sup>b</sup>	0.0006	0.006
	Benzo(k)fluoranthene	1.9 <sup>b</sup>	0.0006	0.006
	Chrysene	9.5 <sup>b</sup>	0.0006	0.006
	Fluoranthene	1,400 <sup>b</sup>	0.0006	0.006
	Fluorene	240 <sup>b</sup>	0.0006	0.006
	Pyrene	1,000 <sup>b</sup>	0.0006	0.006
	Naphthalene	0.12 <sup>b</sup>	0.002	0.02
	Acenaphthylene	1,000 <sup>b</sup>	0.0006	0.006
	Benzo(g,h,i)perylene	1,177 <sup>a</sup>	0.0006	0.006
	2-Chloronaphthalene	127 <sup>a</sup>	0.002	0.02
	Dibenz(a,h)anthracene	0.04 <sup>a</sup>	0.0006	0.006
	Indeno(1,2,3-cd)pyrene	0.42 <sup>a</sup>	0.0006	0.006
	Phenanthrene	79.04 <sup>a</sup>	0.0006	0.006
SVOCs using USEPA Method 8270D	Benzidine	0.000001 <sup>a</sup>	0.333	0.0637
	Bis(2-chloroethoxy)methane	-	0.333	0.0077
	Bis(2-chloroethyl)ether	0.00011 <sup>a</sup>	0.333	0.00896
	Bis(2-chloroisopropyl)ether	3.11 <sup>a</sup>	0.333	0.0076
	4-Bromophenyl-phenylether	0.005 <sup>a</sup>	0.333	0.0114
	4-Chlorophenyl-phenylether	-	0.333	0.00627
	3,3-Dichlorobenzidine	0.0018 <sup>a</sup>	0.333	0.0794
	2,4-Dinitrotoluene	0.0003 <sup>a</sup>	0.333	0.00607
	2,6-Dinitrotoluene	0.0002 <sup>a</sup>	0.333	0.00737
	Hexachlorobenzene	0.04 <sup>a</sup>	0.333	0.00856
	Hexachloro-1,3-butadiene	0.04 <sup>a</sup>	0.333	0.01
	Hexachlorocyclopentadiene	0.01 <sup>a</sup>	0.333	0.0587
	Hexachloroethane	0.14 <sup>a</sup>	0.333	0.0134

**Table 6. Analytes, Screening Levels, and Reporting Limits of Site Soil Samples (PAHs and SVOCs) (2 pages)**

Method	Analyte	IDEQ IDTL <sup>a</sup> /RUSL <sup>b</sup> (mg/kg)	ESC Method Detection Limit (mg/kg)	ESC Reporting Limit (mg/kg)
SVOCs using USEPA Method 8270D	Isophorone	0.14 <sup>a</sup>	0.333	0.00522
	Nitrobenzene	0.02 <sup>a</sup>	0.333	0.00695
	n-Nitrosodimethylamine	0.000002 <sup>a</sup>	0.333	0.00906
	n-Nitrosodiphenylamine	0.09 <sup>a</sup>	0.333	0.0647
	n-Nitrosodi-n-propylamine	0.00002 <sup>a</sup>	0.333	0.00594
	Benzylbutyl phthalate	511 <sup>a</sup>	0.333	0.0103
	Bis(2-ethylhexyl)phthalate	11.84 <sup>a</sup>	0.333	0.012
	Di-n-butyl phthalate	30.99 <sup>a</sup>	0.333	0.0109
	Diethyl phthalate	27.53 <sup>a</sup>	0.333	0.00691
	Dimethyl phthalate	271 <sup>a</sup>	0.333	0.0054
	Di-n-octyl phthalate	1,828 <sup>a</sup>	0.333	0.00907
	1,2,4-Trichlorobenzene	0.69 <sup>a</sup>	0.333	0.00876
	4-Chloro-3-methylphenol	-	0.333	0.00477
	2-Chlorophenol	0.36 <sup>a</sup>	0.333	0.00831
	2,4-Dichlorophenol	0.10 <sup>a</sup>	0.333	0.00746
	2,4-Dimethylphenol	0.82 <sup>a</sup>	0.333	0.0471
	4,6-Dinitro-2-methylphenol	-	0.333	0.124
	2,4-Dinitrophenol	0.04 <sup>a</sup>	0.333	0.098
	2-Nitrophenol	-	0.333	0.013
	4-Nitrophenol	0.23 <sup>a</sup>	0.333	0.0525
Pentachlorophenol	0.01 <sup>a</sup>	0.333	0.048	
Phenol	7.36 <sup>a</sup>	0.333	0.00695	
2,4,6-Trichlorophenol	0.0044 <sup>a</sup>	0.333	0.00779	

Notes:

<sup>a</sup> Initial Default Target Levels (IDTL) from Idaho Risk Evaluation Manual (IDEQ 2004).

<sup>b</sup> Residential Use Screening Levels (RUSL) from IDAPA 58.01.24.

Shaded areas identify constituents where the reporting limit exceeds the screening level.

mg/kg = milligram per kilogram

**Table 7. Analytes, Screening Levels, and Reporting Limits of Site Soil Samples (RCRA 8 Metals)**

Method	Analyte	Statewide WA Background <sup>a</sup> (mg/kg)	Statewide OR Background <sup>b</sup> (mg/kg)	IDEQ IDTL <sup>c</sup> (mg/kg)	SVL Method Detection Limit (mg/kg)	SVL Reporting Limit (mg/kg)
RCRA 8 Metals using USEPA Method 6010C and 7471B (Mercury)	Arsenic	7.00	17.0	0.39	0.81	2.5
	Barium	-	970	895	0.66	0.20
	Cadmium	1.00	-	1.35	0.69	0.20
	Total Chromium	42.0	120	2,134	0.16	0.60
	Lead	17.1	30.0	49.6	0.32	0.75
	Selenium	-	0.490	2.03	1.50	4.0
	Silver	-	2.20	0.189	0.22	0.50
	Mercury	0.0100	0.750	0.0051	0.0053	0.33

Notes:

<sup>a</sup> Natural Background Soil Metals Concentrations in Washington State: Table 6 (Ecology 1994).

<sup>b</sup> Background Levels of Metals in Soils for Cleanups: Table 4; Owyhee Uplands (ODEQ 2013).

<sup>c</sup> Initial Default Target Levels from Idaho Risk Evaluation Manual (IDEQ 2004).

Shaded areas identify constituents where the reporting limit exceeds the screening level.

mg/kg = milligram per kilogram

## Section 9.0 Sampling Methods

This section describes the procedures the TerraGraphics field crew will use to obtain project samples. TerraGraphics' field crew will send samples to ESC and SVL with an accompanying complete chain-of-custody form upon completion of sampling all DUs (see Section 10.0). Section 11.0 and Table 8 lists the equipment necessary to perform the sampling activities, and describes the actions TerraGraphics will take if problems arise in the field.

Personal protective equipment (PPE) necessary to perform the field work for this project will be consistent with the requirements of the *Idaho General Safety and Health Standards* (Division of Building Safety 2006) and all project-specific health and safety plans associated with the project.

In addition to these PPE requirements, the following specific PPE is required for field work associated with this project:

- Nitrile gloves
- Safety glasses with side shields
- Hearing protection (when working near the direct push drill rig--PowerProbe™)
- Steel toe boots (when working near the direct push drill rig--PowerProbe™)
- Safety helmet (when working near the direct push drill rig--PowerProbe™)

QA/QC procedures as specified for sample collection will be followed by sampling personnel. The QA/QC procedures will be fulfilled by adhering to all requirements detailed in this QAPP and the soil sampling procedures described below. Such adherence will be demonstrated through

appropriate documentation of sampling procedures within the field logbook or field sheets as described herein. Field audits by the project QAO may also be part of QA/QC procedures.

## 9.1 Incremental Sampling Methodology

The sampling design consists of a multi-incremental sampling approach, consistent with the ITRC's ISM guidance document (ITRC 2012). The ISM strategy is a method to collect soil samples that are representative of a whole DU area. TerraGraphics divided the Site into 17 individual DUs (Table 5 and Figure 2). The field crew will collect 30 subsamples to complete one multi-increment sample for each DU (for a total of 17 samples with additional samples for QA purposes as discussed in Section 12.0). The following bullet list describes RCRA 8 metals and PAH/SVOC sampling methods:

- The field crew will collect the ISM samples from the surface to a maximum depth of 12 inches bgs.
- The field crew will collect an equal volume of sample from each subsample location using direct push methods (ASTM 2014).
- The field crew will first work the core through a ¼-inch (6.35 mm) sieve to break up the soil core and remove larger debris, then through a #10 sieve (2.00 mm) in the field to better filter and homogenize the samples.
- Field crews will place the soil from each DU into a dedicated, new, clean, and disposable plastic bucket for homogenization.
- The field crew will contain all subsample soil temporarily into a dedicated 5-gal bucket until all subsamples have been obtained.
- To homogenize the material, the field crew will lay out all contents of the 5-gal bucket onto dedicated visqueen material (polyethylene sheeting) and mix thoroughly by hand using single use, nitrile gloves.
- The field crew will use a cone and quarter technique to further composite and collect an equal volume of soil from each DU.
  - The field crew will form the soil into a cone shape on a dedicated clean flat visqueen surface before further flattening the cone with a spatula (or similar).
  - The field crew will divide the volume into four equal volumes.
  - The field crew will collect a sample and place it into a 1-gal Ziploc bag for RCRA 8 Metals analysis. Sample will be sent to SVL labs for preparation and analysis as follows:
    - SVL will air dry, sieve using a size 80-mesh on the split portion of the sample, and homogenize the samples following ITRC guidance (ITRC 2012). *They will not pulverize the samples.*
    - SVL will use the 2-D Japanese Slab Cake method to create the analytical 10-gram (g) aliquots which will be weighed to within  $\pm 0.1$  g.
    - SVL will use a riffle splitter to split the sample to a size that will fit on the 2-D Japanese Slab Cake tray.
    - SVL will dry the samples to 5-10% moisture content, and determine percent solids to correct results for dry weight.
    - SVL will use 1 g of the 10-g aliquot for their analysis

- The field crew will collect a sample and place it into a 4-oz jar for SVOC and/or PAH analysis. Sample will be sent to ESC for analysis. ESC will not dry, sieve, or pulverize these soil samples.

## 9.2 Discrete Sampling Method

The field crew will implement a discrete/grab sampling approach within the EADA-affected area of DU3.1B (on the old railroad ballast which is an approximately 20-foot-wide section that stretches 2,650 feet along the DU). TerraGraphics' field crew will collect up to one surface soil grab sample to evaluate the potential COCs present in the surface soil due to EADA recently sprayed in the area. TerraGraphics' field crew will also collect up to one surface soil grab sample within DU3.1A or DU3.1C (where no EADA has been sprayed) to compare to the sample collected with known EADA.

Using a clean, single use, nitrile glove, the field crew will collect the surficial soil sample and place it into: 1) a 4-oz glass jar for PAH analysis, and 2) a 4-oz glass jar for RCRA 8 metal analysis (see Table 8). The field crew will place these soil samples in a cooler containing double-bagged ice (refrigerated) immediately after collection and hold the samples under chain-of-custody for shipment to ESC (PAH analysis) and SVL (RCRA 8 metals analysis; not subject to drying/sieving as ISM-prep samples).

## 9.3 DU 3.1B Excavation

TerraGraphics will oversee an excavation subcontractor who will remove the top layer (approximately 6 inches) of EADA-affected soils in DU3.1B at all subsample locations prior to conducting ISM sampling. Since the underlying soils likely represent site conditions prior to EADA application, excavation efforts are being conducted to minimize the potential effect of cross contamination from the overlying 6 inches of EADA-affected soils. Once each subsample has been collected, the small pits will be backfilled with the same excavated soil and compacted using the excavator tracks.

## 9.4 Decontamination

The field crew will decontaminate the sampling equipment between DUs and collect a rinsate at the end of each sampling day. Field work is estimated to take 5 days.

To the greatest extent possible, the field crew will use disposable and/or dedicated PPE and sampling equipment to avoid cross-contamination. When required, the field crew will conduct decontamination in a central location, upwind, and away from suspected contaminant sources. The field crew will use the following procedures for all non-dedicated sampling equipment:

1. Clean with tap water and non-phosphate detergent (Alcotabs ®), using a brush, if necessary, to remove particulate matter and surface films.
2. Rinse thoroughly with tap water.
3. Rinse thoroughly with de-ionized/distilled water.
4. Air dry the equipment completely.
5. Remove the equipment from the decontamination area and cover with plastic.
6. Wrap equipment stored overnight in aluminum foil and cover with clean, unused plastic.

## 9.5 Investigation-derived Waste

Investigation-derived Waste (IDW) consists of any remaining soil cuttings and water from decontamination procedures. The following steps summarize the approach to managing and documenting the management of wastes:

1. Containerize the waste,
2. On-site treatment and disposal *or* transportation and off-site disposal, and
3. Documentation of waste determination, transportation, and disposal.

### 9.5.1 General responsibilities

The field crew manager as identified in the flow chart of this QAPP is responsible for ensuring that the field crew conducts field activities in accordance with TerraGraphics' IDW standard operating procedure. The field crew is responsible for implementing the IDW standard operating procedure and communicating any unusual or unplanned condition to the field crew manager's attention.

### 9.5.2 IDW Labeling

TerraGraphics will use Department of Transportation-approved containers to store IDW, typically 55-gal drums.

#### 9.5.2.1 Label Information

Two general IDW conditions exist and containers must be properly labeled: i) from previous studies or on-site data, waste characteristics are known to be either hazardous or nonhazardous; or ii) waste characteristics are unknown until additional data are obtained. The waste characteristics are unknown for this Site. For situations where the waste characteristics are known, the waste containers should be packaged and labeled in accordance with any state and federal regulations that may govern the labeling of waste.

**The following information shall be placed on all waste labels:**

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Category	Example of Information
Description of waste	i.e., soil cuttings, decontamination water
Contact information (i.e., client requesting sampling)	i.e., contact name and telephone number
Date when the waste was first accumulated	9/30/2016

#### 9.5.2.2 Label Placement

TerraGraphics will use weatherproof waste labels and fill each out with a permanent marker to prevent being washed off or becoming faded by sunlight. Apply each label or marking to the upper one-third of the container at least twice, on opposite sides. However, when multiple containers are accumulated together, it also may be helpful to include labels on the top of the containers to facilitate organization and disposal. The label must not be affixed across container bungs, seams, ridges, or dents.

### **9.5.3 Waste Accumulation on Site**

TerraGraphics will predetermine staging areas for IDW containers that are easily accessible to the field crew and potential waste haulers. Determine the methods and personnel required to safely transport IDW containers to the staging area before field mobilization. Handling and transport equipment will be consistent with the associated weight for both lifting and transporting. Store the IDW containers in a secured storage area and so that the field crew or potential waste haulers can easily read the labels.

IDW that is classified as nonhazardous or “characterization pending analysis” should be disposed of as soon as possible. Until disposal, such containers should be inventoried, stored as securely as possible, and inspected regularly, as a general good practice. IDW that is classified as hazardous shall not be accumulated on Site longer than 30 days after receipt of analytical results.

### **9.5.4 IDW Container Movement**

TerraGraphics will contact a state-certified hazardous waste hauler to transport all wastes classified as hazardous. All waste manifests will be signed either by the client or the client’s designee, which can in special circumstances be the project manager if acting as an authorized agent for the client.

### **9.5.5 Documentation**

Documentation requirements apply to all IDW during project activities. TerraGraphics’ field crew will keep field records of all waste generation activities in the field notebook including, but not limited to:

- Description of waste generating activities
- Location of waste generation (including depth, if applicable)
- Type and volume of waste
- Date and time of generation
- Description of any waste sampling
- Name of person recording information
- Name of field manager at time of generation

The field crew will record each container of IDW in the field notebook and make a note of IDW placement on the Site figure. After the waste is disposed of, either by transportation offsite or disposal on Site in an approved disposal area, the field crew will document proper disposition of IDW in the same field notebook. Throughout the project, the field crew manager will maintain an inventory itemizing the type and quantity of the IDW. TerraGraphics will keep all manifests with the project file and will attach a copy of the manifest as an appendix in the ESA Report.

## **Section 10.0 Sample Handling and Custody**

The TerraGraphics field crew will collect samples into laboratory-supplied sampling containers (i.e., from analytical laboratory, laboratory supplier, or laboratory equipment provider), or from the store (i.e., Ziploc bags), label them, place them in a bag (to isolate the sample in case the



container breaks), place them in an ice-chilled cooler, and transport them directly to the shipment location.

TerraGraphics' field crew will oversee proper storage and handling of all collected samples until they are transferred to the appropriate analytical facility or until they are properly discarded. TerraGraphics will hold samples under chain-of custody per ASTM D-4840-99, Standard Guide for Sampling Chain-of-Custody Procedures (ASTM 2010). The field crew will use chain-of-custody forms to document sample custody and transfer. Chain-of-custody forms will accompany the samples from sample collection throughout the shipping process and will be filed in the project TRIM system files by the IDEQ project manager.

## Section 11.0 Analytical Methods

Table 8 lists the analytical method(s), container type(s), preservative(s), and holding time(s) applicable to all samples obtained under this project. All sample containers, labels, and preservatives will be obtained from ESC and SVL, as appropriate. Samples must be preserved as directed and analyzed within the holding times. The project manager will notify the laboratories prior to sample shipment to ensure a holding time is not exceeded. The TerraGraphics field crew will follow all sample collection and preparation instructions provided by the analytical laboratories for the duration of this project.

**Table 8. Soil Analytical Methods, Container Types, Preservation Methods, and Sample Holding Times**

Analyte Group	Analytical Method	Lab	Sample Container	Preservative(s)	Holding Time
Arsenic, Barium, Cadmium, Total Chromium, Lead, Selenium, Silver	USEPA 6010C	SVL	1-gal, double plastic, resealable bag for ISM samples;  (1) 4-oz glass jar for grab samples	None	180 days
Mercury	USEPA 7471B	SVL	1-gal, double plastic, resealable bag for ISM samples;  (1) 4-oz glass jar for grab samples	None	28 days
PAHs <i>(as listed in Table 6)</i>	USEPA 8270D-SIM	ESC	(1) 4-oz glass jar for both ISM and grab samples	Ice to 4°C ± 2°C	14 days
SVOCs <i>(as listed in Table 6; only for DU2.2A, DU2.2B, and DU2.2C)</i>	USEPA 8270D	ESC	(1) 4-oz glass jar for ISM samples	Ice to 4°C ± 2°C	14 days

## Section 12.0 Quality Control Requirements

TerraGraphics will use QC samples to evaluate data quality. QC samples are controlled samples introduced into the analysis stream and whose results TerraGraphics will use to review data quality and evaluate the accuracy, precision, and representativeness of the data. This section describes the purposes of each type of QC sample.

### 12.1 Field Quality Control Requirements

TerraGraphics will accomplish field QC checks through the analysis of controlled samples that are introduced to the laboratory from the field. The TerraGraphics field crew will collect field replicates and field duplicates and submit them to the laboratory to provide a means of assessing the quality of data. Table 9 shows the frequency for each type of field QC sample used for this project and Table 10 lists the analytical method(s), container type(s), preservative(s), and holding time(s) applicable to the QC samples for this project.

**Table 9. Soil Field Quality Control Samples**

QC Check	Typical Frequency	Total Number for this Project	QA Purpose
Rinsate Blank	1:sample day	5*	Identifies contamination in sample collection and/or preparation.
Field Replicate	1:20 samples	2 total (1 triplicate from DU1.1 and 1 triplicate from DU1.2)	Evaluates error associated with the collection of samples in the field and evaluates the error associated with the processing and analysis of those samples.
Field Duplicate	1:20 samples	2 total (1 from DU2.2 for SVOCs and 1 from a field replicate sample)	Evaluates sample precision and ensures reliable estimates represented by a relative percent difference.

\* TerraGraphics expects the sampling to last 5 days.

**Table 10. Analytical Methods, Container Types, Preservation Methods, and QC Sample Holding Times**

Analyte Group	Analytical Method	Lab	Sample Container	Preservative(s)	Holding Time
<b>Soil</b>					
Arsenic, Barium, Cadmium, Total Chromium, Lead, Selenium, Silver	USEPA 6010C	SVL	1-gal, double plastic, resealable bag	None	180 days
Mercury	USEPA 7471B	SVL	1-gal, double plastic, resealable bag	None	28 days
PAHs (as listed in Table 6)	USEPA 8270D-SIM	ESC	(1) 4-oz glass jar	Ice to 4°C ± 2°C	14 days
SVOCs (as listed in Table 6; only for DU2.2A, DU2.2B, and DU2.2C)	USEPA 8270D	ESC	(1) 4-oz glass jar	Ice to 4°C ± 2°C	14 days

### 12.1.1 Rinsate Blank

The objective of the rinsate blank is to provide a laboratory analytical check on possible sources of contamination of a sample that may be related to equipment decontamination and sample handling procedures. The field crew will collect all rinsate blank samples in the field by pouring the same de-ionized/distilled water used in decontamination through/over the sampling equipment (after it has been decontaminated) and collect the rinsate in the appropriate sample containers and adding appropriate preservatives.

### 12.1.2 Field Replicate Sampling Procedure for RCRA 8 Metals and PAHs

The field crew will collect field replicates (three rounds of sampling) in the predetermined DUs 1.1 and 1.2. This sampling approach is also referred to as “replicates in triplicate” and could also be considered “one sample plus two replicates” or “three samples.” The purpose of collecting multiple samples is to quantify uncertainty in the estimate of the mean concentration. Each replicate will consist of 30 subsamples randomly selected and marked.

### 12.1.3 Field Duplicate Sampling Procedure for RCRA 8 Metals and PAHs

During the cone and quarter technique, the field crew will collect an additional equal volume of soil from one of the DUs where a replicate sample is collected, either DU1.1 or DU1.2. The field crew will follow the same techniques listed in Sections 9.1 and 9.2 but will place an additional portion of soil into another 1-gal Ziploc bag and another 4-oz glass jar that will be marked as duplicate samples.

### 12.1.4 Field Duplicate Sampling Procedure for SVOCs

During the cone and quarter technique, the field crew will collect an additional equal volume of soil from within DU2.2A, or DU2.2B, or DU2.2C. The field crew will follow the same technique as listed in Section 9.1 and 9.2 but will place an additional portion of soil into another 4-oz glass jar that will be marked as a duplicate sample.

## **12.2 Laboratory Quality Control Requirements**

ESC and SVL will use the required QC procedures for each laboratory's analyses as described in their individual standard operating procedures and QA manuals (included in Appendices D and E). The reported laboratory QC will include the types of samples shown in Table 11 to assess the project's DQIs (see Table 4 of this QAPP). The reported laboratory QC will also include the actual reporting limits, holding times, dilutions, etc. Appendix C includes calculations for precision and accuracy.

Laboratory QC checks are routinely performed as part of the analysis process. The frequency and type of QC samples are often analysis method-dependent and include method blanks, laboratory spikes, and internal laboratory splits. Analyzing laboratories will report any variance from QC limits impacting the quality of sample results and may report details of internal laboratory QC if requested. The analytical laboratory may provide appropriate sample containers, chain-of-custody forms, sample labels, and any necessary container seals. Laboratory QA/QC and data reports will be filed in TRIM following applicable filing protocols.

### ***12.2.1 Laboratory or Method Blank***

A laboratory or method blank is a sample of known matrix where the specific constituents requested for analysis are known to be absent or are present at concentrations less than the laboratory minimum limit of detection. The laboratory blank is analyzed to evaluate the accuracy of the analysis.

### ***12.2.2 Laboratory Control Sample and Laboratory Control Sample Duplicate***

An LCS is a sample that contains a known concentration of analytes and is analyzed to assess the overall method performance. An LCS undergoes the same preparatory and determinative procedures as the project samples and is the primary indicator of laboratory performance. LCS percent recoveries are used to measure accuracy. The RPD for LCSD recoveries is used to measure precision.

### ***12.2.3 Laboratory Duplicate Sample***

A laboratory duplicate sample is a sample that is split by the laboratory into two separate and identical samples. The two samples are analyzed and a comparison of the results (RPD) is used to assess laboratory precision.

### ***12.2.4 Matrix Spike and Matrix Spike Duplicate***

An MS sample has a known amount of the target analyte added to project matrix before analysis to assess possible matrix interferences on the analysis. Percent recoveries on MS samples should be compared to percent recoveries of LCS samples. An MS/MSD pair can be used to assess precision. During the cone and quarter technique, the field crew will collect an additional equal volume of soil from a location determined in the field. The field crew will follow the same technique as listed in Section 9.1 and 9.2 but will place an additional portion of soil into another 4-oz glass jar that will be marked as an MS/MSD sample on the chain-of-custody form.

**Table 11. Laboratory Quality Control Samples**

QC Check		Frequency	Total Number	QA Purpose
Blanks	Lab method blank	1:analyte group	1	Identify errors or contamination in sample collection, preparation, and analysis.
Duplicates	Lab duplicate completed on the replicate sample	1:20 samples	1	Evaluates the error associated with the processing and analysis of the replicate sample.
Spikes	MS or LCS	1:20 samples	1	Evaluate accuracy and precision of the method.
	MSD or LCSD		1-2	
Surrogates		Method specific requirement	Equal to the total number of analyses per sample requiring surrogates.	Indicate bias and accuracy of monitoring compounds.

### 12.3 Data Analysis Quality Control Checks

QC data may be checked/reviewed for quality by the project manager or QAO at any time during the project and must be checked after all of the data are collected. Corrective actions, as needed, will be documented in the event that control limits are exceeded. Data qualifiers will be assigned following appropriate data verification/validation procedures. Any qualifiers added will be defined in the project summary/technical report and will be consistent with USEPA guidance (USEPA 2002, 2014b and c).

## Section 13.0 Instrument/ Equipment Testing, Inspection, Maintenance, and Calibration

Laboratory instrument calibration and maintenance frequency will follow their individual standard operating procedures and certification requirements. Laboratory instrument/equipment testing, inspection, and maintenance are performed and documented by the laboratory. Procedures and schedules for preventive maintenance of sampling equipment are the responsibility of the laboratory. Each instrument or item of laboratory equipment will be maintained periodically to ensure accuracy. These procedures and frequency of performance are designated in the individual instrument manuals (see attached Quality Assurance Manuals, Appendix D for SVL and Appendix E for ESC).

Calibrations and maintenance of relevant field instruments will be completed by TerraGraphics as required by and in accordance with the equipment user manuals.

## Section 14.0 Inspection/ Acceptance Requirements for Supplies and Consumables

TerraGraphics, ESC, and SVL will use services and supplies of adequate quality. TerraGraphics will select the supplies and consumables used for this project based on manufacturer and laboratory recommendations and/or on the standard of practice for the accomplished service. ESC and SVL will maintain a procedure for the purchase, storage, and evaluation of supplies and services, as well as records of inspections, verifications, and supplies as stated in their respective quality assurance manuals (Appendices D and E).

## Section 15.0 Data Acquisition Requirements (Non-Direct Measurements)

This project will not rely upon secondary data for this project (Table 12).

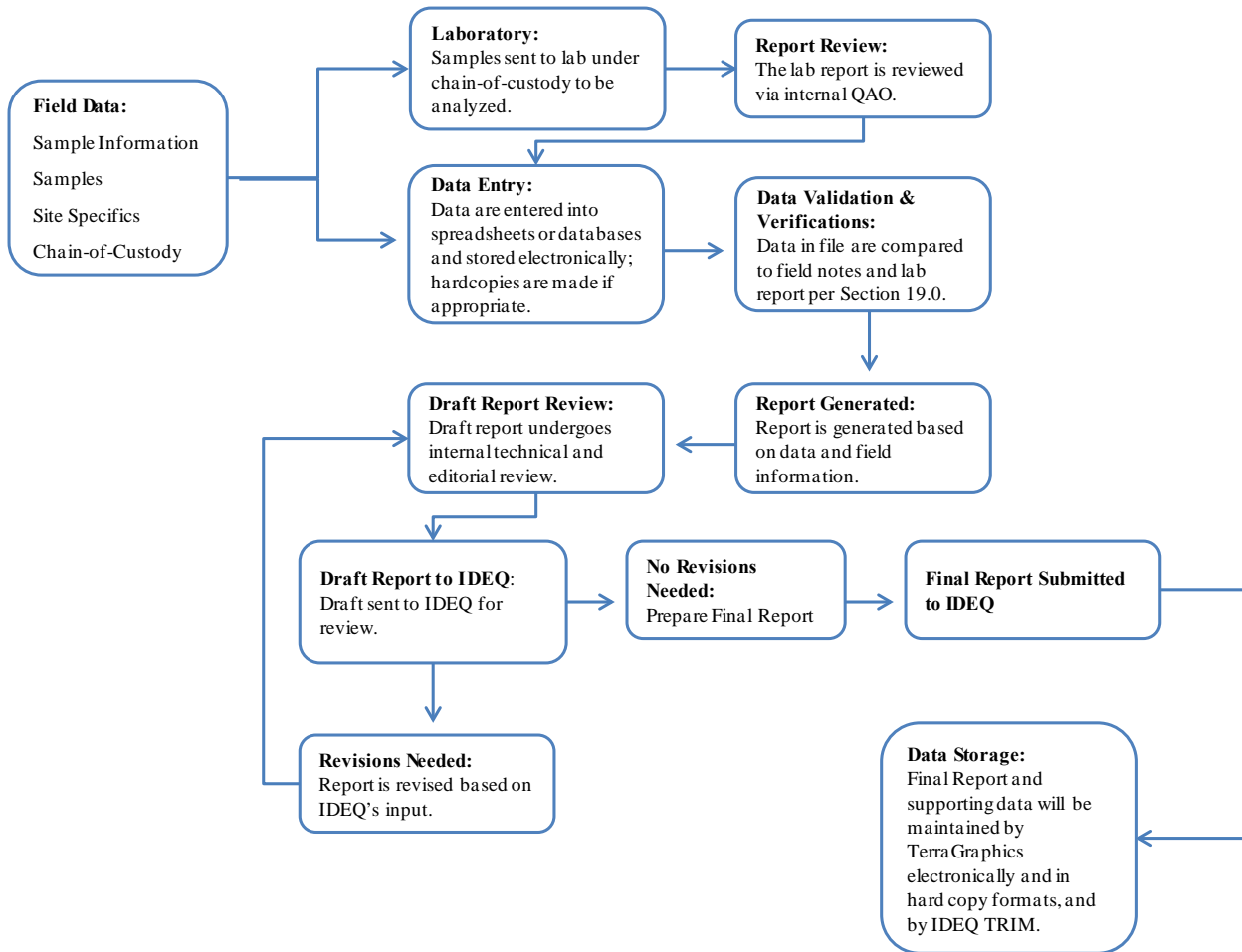
**Table 12. TerraGraphics Equipment Inspection and Maintenance**

<b>Data Sources</b>	<b>Intended Use</b>	<b>Rationale for Use</b>	<b>Acceptance Criteria</b>
Previous limited assessment of the ROW, conducted by TRC for BNSF (TRC 2014), included sampling of shallow sub-surface soils for RCRA 8 metals, TPHs, and limited PAHs from the 18- to 24-inch depth interval. Metals concentrations from soil samples collected from the 18- to 24-inch depth interval were above IDTLs.	Historical COC data.	General informational purposes only.	QA/QC procedures of the data is unknown.
TerraGraphics, 2015. Phase I Environmental Site Assessment, Coeur d'Alene BNSF Railroad Corridor, Coeur d'Alene, ID 83814. Prepared for the Idaho Department of Environmental Quality, May.	Determination of recognized environmental conditions.	Filling in data gaps and aiding in planning Phase II ESA.	Completed to ASTM E1527-13.

## Section 16.0 Data Management

Figure 6 shows the data management flow chart. Data management will follow the USEPA Region 10 Data Management Plan for Environmental Monitoring and Associated Geospatial Data (USEPA 2014a).

**Figure 6. Data Management Diagram**



TerraGraphics' Project Manager, QAO, and supporting staff will review field documents for consistency, completeness, and accuracy. They will return any issues discovered in review to the field crew for clarification or resolution. Staff will review shipping documents prior to final shipping preparations. Original field documents will be stored in the project file for the duration of the project as well as scanned and stored electronically.

TerraGraphics will pair data from the laboratory with the previously entered field information in preparation for summary and analysis. The combined field and laboratory data will be reviewed for accuracy and completeness prior to use.

On request, TerraGraphics will make available the raw or validated data for third party data transformation. In addition, TerraGraphics will maintain all data files in their originally received form and make them available upon request. IDEQ will store all data and reports in TRIM.

## **Section 17.0 Assessments and Response Actions**

The TerraGraphics QAO will assess project compliance with this QAPP by reviewing field documentation and laboratory reports. The TerraGraphics QAO will investigate and correct any errors or inconsistencies identified in the field notes to ensure the integrity of the data and conformation to the QAPP. They will take into account the review of internal laboratory QA, audits, and surveillances or other types of laboratory assessments. If unexpected analytical results are reported for any reason, TerraGraphics will contact the laboratory to perform an additional quality review of the data in question. The TerraGraphics QAO will perform additional assessment independently of the Project Manager and field crew lead. The TerraGraphics QAO will provide a written data usability memorandum as described in Sections 18.0 and 19.0 to the TerraGraphics Project Manager; TerraGraphics will include this usability memorandum as an attachment to the ESA Report.

Additionally, if the TerraGraphics Project Manager makes a decision necessary in the field that might deviate from the QAPP as a result of an unforeseen challenge or field limitation, the responses will be documented and the TerraGraphics Project Manager will immediately notify the IDEQ Project Manager.

## **Section 18.0 Reports to Management**

TerraGraphics will prepare an ESA Report for IDEQ detailing investigation results. Raw data from the lab and a QA/QC memorandum will be included in the report as appendices. TerraGraphics will summarize field and laboratory data from the Site investigation and include an assessment of investigation data quality in the report, and will submit electronic copies to appropriate IDEQ personnel.

## **Section 19.0 Data Review, Validation, and Verification Requirements**

TerraGraphics will conduct a data review to ensure that project data have been recorded, transmitted, and processed correctly. This is to ensure that QAPP requirements were met and that data produced by this project are of acceptable quality to be used for assessment in determining future remedial strategies.

The TerraGraphics QAO or designee will compare the precision, accuracy/bias, completeness, and sensitivity for all data collected for this project to the acceptance criteria listed in Table 4 and Appendix C. Precision, accuracy/bias, and sensitivity will be evaluated as data are received from the laboratories. Completeness, representativeness, and comparability will be evaluated after all data have been received. Representativeness and comparability will be evaluated by the QAO and verified by the Project Manager.



The data for this project plan will be evaluated using criteria specified in the USEPA National Functional Guidelines for Superfund Organic Methods Data Review (USEPA 2014c) and National Functional Guidelines for Inorganic Superfund Data Review (USEPA 2014b). The laboratories will analyze and report data so TerraGraphics can perform a Stage 2A data validation (USEPA 2009). TerraGraphics will produce a QA/QC memorandum documenting the evaluation of the quality objectives after all data have been received and appropriately validated, and will include it as an attachment to the ESA Report.

## **Section 20.0 Validation and Verification Methods**

### **20.1 Data Verification**

Data verification methods relate to the process of evaluating the completeness and correctness of the data and conformance of this QAPP. TerraGraphics will compare relevant field records with sample containers and labels prior to sample shipment to ensure accurate sample documentation and verify conformance to the QAPP. If inconsistencies are found, the reviewer will consult the field crew to gather additional information in an attempt to resolve the discrepancy. If the discrepancy either cannot be resolved or is found to be due to deviation from or inability to meet sampling protocol, the reviewer will inform the QAO and Project Manager, as needed, who will determine whether and how to qualify the data.

Parameter data collected for each DU are stored in Excel® and reviewed for consistency and adherence to procedures outlined in this QAPP.

Upon data receipt, TerraGraphics will verify chain-of-custody forms, sample preservation records, analytical holding times, case narratives, sample data as compared to QC sample data, requested turnaround time, and reporting requirements. The QAO will discuss problems or questions for further resolution and/or documentation with the laboratory if necessary.

### **20.2 Data Validation**

For soil data provided by ESC and SVL, TerraGraphics will conduct a stage 2A data validation of all laboratory-supplied data in accordance with the USEPA data validation guidance (USEPA 2009). Each laboratory's technical staff and QAO will review analytical data. The case narrative will identify whether any laboratory QC data are outside the laboratory's QC criteria. Data deliverables will include a memorandum noting a case narrative, analytical results, and laboratory QC sample results that will enable a stage 2A level of validation/verification to be performed.

Additionally, TerraGraphics will review the sample data and case narratives to evaluate the lab and field QC data to determine the data quality and assess data usability relative to the project's DQIs presented in Table 4 and Appendix C. This process will provide a basis for meaningful interpretation of the data quality and evaluate the need for corrective actions and/or comprehensive data validation. TerraGraphics will document data exceeding QAPP DQIs and will qualify and potentially reject data not meeting precision or accuracy requirements.

If the QAO determines the data do not meet the criteria given in Appendix C and Table 4, the data will be qualified appropriately based on applicable USEPA guidance documents (USEPA

2002, 2009, and 2014b and c) and/or the professional opinion of the reviewer; any limitations will be detailed in the QA/QC memorandum.

## **Section 21.0 Reconciliation with User Requirements**

The TerraGraphics Project Manager and QAO will perform the data quality assessment to determine if the project data set is of the right type, quality, and quantity to achieve the objectives of the project and can confidently be used to make informed decisions. Information and findings associated with the project data review, verification, and validation efforts shall be considered during the data assessment process.

If the TerraGraphics Project Manager or QAO decide the project data do not meet the project needs or the QAPP objectives and/or if the conclusions drawn from the data do not appear to be reasonable, the Project Manager or QAO will report this to IDEQ and determine and document the necessary corrective actions. If failure to meet project specifications occurs, the cause of the failure will be evaluated and corrected.

## Section 22.0 References and Resources Used

- ASTM, 2010. D-4840-99, Standard Guide for Sampling Chain-of-Custody Procedures.
- ASTM, 2014. D6282/D6282M-14, Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations.
- ASTM E1527-13, Standard Practice for Environmental Site Assessments: Phase I Environmental Site Assessment Process.
- 29 CFR 1910.120, "Hazardous Waste Operations and Emergency Response," Title 29, Code of Federal Regulations, Part 1910.
- Washington State Department of Ecology (Ecology), 1994. Natural Background Soil Metals Concentrations in Washington State. Toxics Cleanup Program, Publication #94-115, October.
- IDAPA 58.01.24. Idaho Administrative Procedures Act (IDAPA) Application of Risk Based Corrective Action at Petroleum Release Sites.
- Idaho Department of Environmental Quality (IDEQ), 2004. Idaho Risk Evaluation Manual, July.
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- ITRC (Interstate Technology & Regulatory Council). 2012. Incremental Sampling Methodology. ISM-1. Washington, D.C.: Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team. [www.itrcweb.org](http://www.itrcweb.org).
- Oregon Department of Environmental Quality (ODEQ), 2013. Background Levels of Metals in Soils for Cleanups, Fact Sheet. Available at <http://www.deq.state.or.us/lq/pubs/docs/cu/FSbackgroundmetals.pdf>. Accessed on June 8, 2016.
- TerraGraphics Environmental Engineering, Inc. (TerraGraphics), 2015. Phase I Environmental Site Assessment Report: Coeur d'Alene BNSF Railroad Corridor, Coeur d'Alene, ID 83814. Prepared for the Idaho Department of Environmental Quality, May.
- USEPA, 1996a. Method 8270D/8270D-SIM: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). Revision 3, December.
- USEPA, 1996b. Method 6010C: Inductively Coupled Plasma-Atomic Emission Spectrometry. Revision 2. December.
- USEPA, 2002. Guidance on Environmental Data Verification and Data Validation, EPA QA/G-8, EPA/240/R-02/004.
- USEPA, 2007. Method 7471B: Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique). Revision 2. February.
- USEPA, 2009. Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use. EPA 540-R-08-005. January.

USEPA, 2014a. Region 10 Data Management Plan for Environmental Monitoring and Associated Geospatial Data.

USEPA, 2014b. USEPA National Functional Guidelines for Inorganic Superfund Data Review (ISM02.2) OSWER 9355.0-131, EPA 540-R-013-001. August.

USEPA, 2014c. USEPA National Functional Guidelines for Superfund Organic Methods Data Review (SOM02.2) OSWER 9355.0-132, EPA 540-R-014-002. August.

USEPA, 2015. Regional Screening Level (RSL) Resident Soil Table. November.  
<https://semspub.epa.gov/work/03/2229085.pdf>

USEPA, 2016. RSL Resident Soil to Groundwater. May.  
<https://semspub.epa.gov/work/03/2229097.pdf>

## FINAL TECHNICAL MEMORANDUM

### Office Locations:

Kellogg, Idaho  
Boise, Idaho  
Las Vegas, Nevada  
Pasco, Washington  
Richland, Washington

**To:** Steve Gill, IDEQ Coeur d'Alene, ID: Regional Office  
Eric Traynor, IDEQ Boise, ID: State Office

**From:** Tom Jenkins, Moscow  
Jon Munkers, Boise

**Date:** August 25, 2017

**Project Code:** 17085

**Subject:** **Amendment to the Quality Assurance Project Plan for the BNSF ROW R2R, Coeur d'Alene, Idaho**

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The purpose of this Technical Memorandum is to describe additional Incremental Sampling Methodology (ISM) activities in Decision Unit (DU) 2.2B of the Burlington Northern Santa Fe Railway Company (BNSF) Right of Way (ROW) Site Assessment. This amendment covers the additional work and amends the existing Final Quality Assurance Project Plan (QAPP) (TerraGraphics 2016).

The purpose of this additional sampling is to identify recognized environmental conditions associated with DU 2.2B within the 2.2-mile section of the BNSF Railroad ROW. Recent removal of the railroad lines within this area have provided an opportunity to sample at depth within this decision unit. The Phase II Additional Assessment will provide information to evaluate potential risk from identified COCs that can then be used to inform the potential purchase and subsequent redevelopment of the property.

## 1 ISM Sampling

TerraGraphics will utilize procedures and protocols consistent with the previously drafted QAPP for the BNSF ROW R2R project (TerraGraphics 2016) except where noted in this amendment. Field personnel will use a multi-incremental sampling approach, consistent with the Interstate Technology and Regulatory Council (ITRC) ISM guidance document (ITRC 2012) to collect ISM samples for a portion of DU2.2B that has been excavated as shown in the site layout map (Figure 1). Field personnel will send 4 (8-oz) soil samples to SVL Analytical Inc. (SVL) to prepare and analyze them for arsenic using USEPA Method 6010 (USEPA 1996a). Field crews will perform field sieving following protocols outlined in the previous QAPP (TerraGraphics 2016).. Personnel will collect soil samples from the homogenized replicates within the DU2.2B and send a total of three replicates to ESC Laboratory Sciences (ESC) to analyze each of them for polycyclic aromatic hydrocarbons (PAHs) using USEPA Method 8270D-selective ion

monitoring (SIM; USEPA 1996b). Beryllium, cadmium, chromium, copper, nickel, thallium, lead, antimony, selenium, silver, and zinc are not being analyzed as part of this assessment since the results of the previous assessment showed that they are not COCs at this location.

The field crew will have access to data sheets noting the GPS coordinates of the subsample locations for each DU (Appendix F in QAPP, TerraGraphics 2016). The field crew will generate random subsample locations in ArcGIS using a pre-programmed procedure named “Create Random Points,” which places points based on a pre-determined area, the number of points desired, and a minimum distance of 5 feet between the points (with an error of 5–10 feet) to prevent overlapping subsample locations.

Three rounds of 30 subsamples will be collected at the predetermined locations for triplicate sampling. Subsample locations will be located using the coordinates generated in ArcGIS and using handheld GPS. Samples will be collected by the use of a hand auger and dedicated 5-gallon buckets. At each subsample location the field crew will remove approximately 2 inches of overburden surface soil then collect soil from approximately 2 inches to 6 inches below ground surface. The 30 subsamples will be composited and sampled according to ITRC’s ISM guidance document (ITRC 2012).

## 2 Timeline of Activities

The following shows a projected timetable of sampling and reporting activities. The reporting will include a summary of field activities, sample results, a data validation summary, and memorandum.

<b>Activity</b>	<b>Anticipated Time of Initiation</b>	<b>Anticipated Time of Completion</b>
ISM Sampling	August/September 2017	August/September 2017
Data Summary Memo	September 2017	September 2017

## 3 References

- Interstate Technology and Regulatory Council (ITRC). 2012. Incremental Sampling Methodology. ISM-1. Washington, D.C.: Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team. [www.itrcweb.org](http://www.itrcweb.org).
- TerraGraphics Environmental Engineering, Inc. (TerraGraphics). 2016. Quality Assurance Project Plan for BNSF ROW R2R, Coeur d’Alene, ID 83814. Prepared for the Idaho Department of Environmental Quality, September.
- U.S. Environmental Protection Agency (USEPA). 1994. Method 7470A: Mercury in Liquid Waste (Manual Cold-Vapor Technique). Revision 1, September.
- USEPA. 1996a. Method 6010C: Inductively coupled Plasma-Atomic Emission Spectrometry. Revision 2. December.

USEPA, 1996b. Method 8270D/8270D-SIM: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). Revision 3, December.

**Attachment A**  
**(Figure 1)**



U:\Moscow\Clients\Idaho\BNSF\DU2B\_a\_points.mxd



- Round 1 Sampling Location
- Round 2 Sampling Location
- Round 3 Sampling Location

Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AeroGRID, IGN, and the GIS User Community



PRINT DATE  
August 23, 2017

PROJECT NUMBER  
15029-01

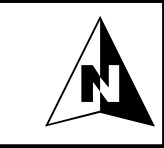
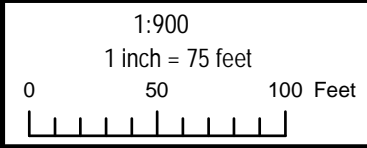
REQUESTOR  
M. Studer

PROJECT MANAGER  
M. Studer

CARTOGRAPHER  
B. Bailey

PROJECT NAME  
**ROW R2R  
Coeur d'Alene, Idaho**

This map was produced using information obtained from several different sources that have not been independently verified. These sources have also not provided information on the precision and accuracy of the data. Information on this map is not a substitute for survey data.



**Figure 1**

**Decision Unit 2.2B**  
Predetermined ISM Sampling Locations

**Appendix A Site-Specific Analytical Summary Tables with Method, Reporting Limits,  
Method Detection Limits, IDTLs, RUSLs, Critical Pathways, and WA/OR Background  
Metals**

**Sampling Method, Reporting Limit, and Method Detection Limit for Soil  
(ESC Lab Sciences and SVL Analytical, Inc.)**

Analyte	Sample Method	Reporting Limit (mg/kg)	Method Detection Limit (mg/kg)	Screening Level (mg/kg)	Screening Level Pathway	WA Statewide Background Level (mg/kg)	OR Statewide Background Level (mg/kg)
<b>PAHs</b>		<b>(ESC)</b>	<b>(ESC)</b>	<b>RUSL</b>	<b>RUSL Pathway</b>		
acenaphthene	USEPA 8270D-SIM	0.006	0.0006	200	GWP	-	-
anthracene	USEPA 8270D-SIM	0.006	0.0006	3,200	GWP	-	-
benzo(a)anthracene	USEPA 8270D-SIM	0.006	0.0006	0.09	GWP	-	-
benzo(a)pyrene	USEPA 8270D-SIM	0.006	0.0006	0.02	Direct Contact	-	-
benzo(b)fluoranthene	USEPA 8270D-SIM	0.006	0.0006	0.19	Direct Contact	-	-
benzo(k)fluoranthene	USEPA 8270D-SIM	0.006	0.0006	1.9	Direct Contact	-	-
chrysene	USEPA 8270D-SIM	0.006	0.0006	9.5	GWP	-	-
fluoranthene	USEPA 8270D-SIM	0.006	0.0006	1,400	GWP	-	-
fluorene	USEPA 8270D-SIM	0.006	0.0006	240	GWP	-	-
pyrene	USEPA 8270D-SIM	0.006	0.0006	0.12	GWP	-	-
<b>SVOCs</b>		<b>(ESC)</b>	<b>(ESC)</b>	<b>IDEQ IDTL</b>	<b>IDTL Critical Pathway</b>		
Acenaphthylene	USEPA 8270D	0.033	0.00671	78.02	GWP	-	-
Benzidine	USEPA 8270D	0.333	0.0637	0.000001	GWP	-	-
Benzo(g,h,i)perylene	USEPA 8270D	0.033	0.00721	1177.98	Surficial Soil	-	-
Bis(2-chloroethoxy)methane	USEPA 8270D	0.333	0.0077	-	GWP	-	-
Bis(2-chloroethyl)ether	USEPA 8270D	0.333	0.00896	0.00011	GWP	-	-
Bis(2-chloroisopropyl)ether	USEPA 8270D	0.333	0.0076	3.11	GWP	-	-
4-Bromophenyl-phenylether	USEPA 8270D	0.333	0.0114	0.005	GWP	-	-
2-Chloronaphthalene	USEPA 8270D	0.033	0.00639	127.66	GWP	-	-
4-Chlorophenyl-phenylether	USEPA 8270D	0.333	0.00627	-	GWP	-	-
Dibenz(a,h)anthracene	USEPA 8270D	0.033	0.00821	0.04	GWP	-	-
3,3-Dichlorobenzidine	USEPA 8270D	0.333	0.0794	0.0018	GWP	-	-
2,4-Dinitrotoluene	USEPA 8270D	0.333	0.00607	0.0003	GWP	-	-
2,6-Dinitrotoluene	USEPA 8270D	0.333	0.00737	0.0002	GWP	-	-
Hexachlorobenzene	USEPA 8270D	0.333	0.00856	0.04	Subsurface Soil	-	-
Hexachloro-1,3-butadiene	USEPA 8270D	0.333	0.01	0.04	Subsurface Soil	-	-
Hexachlorocyclopentadiene	USEPA 8270D	0.333	0.0587	0.01	GWP	-	-
Hexachloroethane	USEPA 8270D	0.333	0.0134	0.14	GWP	-	-
Indeno(1,2,3-cd)pyrene	USEPA 8270D	0.033	0.00772	0.42	Surficial Soil	-	-
Isophorone	USEPA 8270D	0.333	0.00522	0.14	GWP	-	-
Naphthalene	USEPA 8270D	0.033	0.00889	1.14	Subsurface Soil	-	-
Nitrobenzene	USEPA 8270D	0.333	0.00695	0.02	GWP	-	-
n-Nitrosodimethylamine	USEPA 8270D	0.333	0.00906	0.000002	GWP	-	-
n-Nitrosodiphenylamine	USEPA 8270D	0.333	0.0647	0.09	GWP	-	-
n-Nitrosodi-n-propylamine	USEPA 8270D	0.333	0.00594	0.00002	GWP	-	-
Phenanthrene	USEPA 8270D	0.033	0.00528	79.04	GWP	-	-
Benzylbutyl phthalate	USEPA 8270D	0.333	0.0103	511.17	GWP	-	-
Bis(2-ethylhexyl)phthalate	USEPA 8270D	0.333	0.012	11.84	GWP	-	-
Di-n-butyl phthalate	USEPA 8270D	0.333	0.0109	30.99	GWP	-	-
Diethyl phthalate	USEPA 8270D	0.333	0.00691	27.53	GWP	-	-
Dimethyl phthalate	USEPA 8270D	0.333	0.0054	270.81	GWP	-	-
Di-n-octyl phthalate	USEPA 8270D	0.333	0.00907	1828.81	Surficial Soil	-	-
1,2,4-Trichlorobenzene	USEPA 8270D	0.333	0.00876	0.69	GWP	-	-
4-Chloro-3-methylphenol	USEPA 8270D	0.333	0.00477	-	GWP	-	-
2-Chlorophenol	USEPA 8270D	0.333	0.00831	0.36	GWP	-	-
2,4-Dichlorophenol	USEPA 8270D	0.333	0.00746	0.1	GWP	-	-
2,4-Dimethylphenol	USEPA 8270D	0.333	0.0471	0.82	GWP	-	-
4,6-Dinitro-2-methylphenol	USEPA 8270D	0.333	0.124	-	GWP	-	-
2,4-Dinitrophenol	USEPA 8270D	0.333	0.098	0.04	GWP	-	-
2-Nitrophenol	USEPA 8270D	0.333	0.013	-	GWP	-	-
4-Nitrophenol	USEPA 8270D	0.333	0.0525	0.23	GWP	-	-
Pentachlorophenol	USEPA 8270D	0.333	0.048	0.01	GWP	-	-
Phenol	USEPA 8270D	0.333	0.00695	7.36	GWP	-	-
2,4,6-Trichlorophenol	USEPA 8270D	0.333	0.00779	0.0044	GWP	-	-
<b>RCRA 8 Metals</b>		<b>(SVL)</b>	<b>(SVL)</b>				
Arsenic	USEPA 6010C	2.5	0.81	0.39	Surficial Soil	7	17
Barium	USEPA 6010C	0.2	0.66	895.64	GWP	-	970
Cadmium	USEPA 6010C	0.2	0.69	1.35	GWP	1	-
Total Chromium	USEPA 6010C	0.6	0.16	2,134.77	GWP	42	120
Lead	USEPA 6010C	0.75	0.32	49.62	GWP	17.1	30
Selenium	USEPA 6010C	4	1.5	2.03	GWP	-	0.49
Silver	USEPA 6010C	0.5	0.22	0.189	GWP	-	2.2
Mercury	USEPA 7471A	0.33	0.0053	0.0051	GWP	0.01	0.75

Natural Background Soil Metals Concentrations in Washington State: Table 6 (Ecology 1994).

Background Levels of Metals in Soils for Cleanups: Table 4; Owyhee Uplands (ODEQ 2013).

Residential Use Screening Levels (RUSL) from IDAPA 58.01.24.

Initial Default Target Levels (IDTL) from Idaho Risk Evaluation Manual (IDEQ 2004).

Shaded areas identify constituents where the report limit exceeds the screening level.

## **Appendix B Site Health and Safety Plan**

**TerraGraphics**  
**Site Health and Safety Plan**

**GENERAL INFORMATION**

**CLIENT:** Idaho Department of Environmental Quality  
**PROJECT MANAGER:** Melody Studer  
**SITE NAME:** Coeur d'Alene Burlington Northern Santa Fe Right of Way  
**SITE LOCATION:** Riverstone to Huetter Section, Coeur d'Alene, ID  
**PURPOSE OF FIELD VISIT(S):** Collect soil samples  
**DATE OF VISIT(S):** September 2016

**Article I. Site Characteristics**

**AREA DESCRIPTION**

According to an Environmental Assessment regarding the Burlington Northern Santa Fe (BNSF) Railway Company, BNSF sought to abandon 6.23 miles of rail line from milepost 6.10 at Post Falls, Idaho, to milepost 12.33 at Coeur d'Alene, in Kootenai County, Idaho. BNSF proposed to salvage the rails, ties, and bridge (located at milepost 7.61) and would leave the ballast and culverts in place. The abandonment was in preparation for the City to purchase the land.

The line was used primarily to provide rail service to the various lumber mills along the Spokane River in the vicinity of Coeur d'Alene. The railway was abandoned in November 2009 and most of the tracks and ties were removed. Additionally, previous mills, burners, etc., have been removed. According to BNSF, there was a single bridge on the Line that was constructed in 1955 and was located at milepost 7.61. The bridge was a wood structure, 16 feet long and 3 feet high, and crossed a dry ditch. BNSF has no record of any significant alterations made to the bridge prior to its removal.

Based on the recognized environmental conditions revealed during the Phase I ESA completed by TerraGraphics Environmental Engineering, Inc. (TerraGraphics) in 2015, the Idaho Department of Environmental Quality (IDEQ) requested TerraGraphics to perform additional characterization activities on the right of way (ROW) Site for use in a human health risk assessment focused on currently identified future redevelopment goals and land use. The City's plan for future use of the Site includes the conversion of the former rail ROW to a pedestrian-bicycle path along the Coeur d'Alene River frontage.

The ROW was divided into three Zones based upon historic industrial uses. All Zones have a history of railroad use for more than 100 years. In the early 1900s there were hourly electric train services along this corridor linking Spokane, Washington, to Coeur d'Alene. The region also has a long history of heavy metal mining and rail distribution.

**POSSIBLE CONTAMINANT CHARACTERISTICS**

**a) Waste Type(s)**

Liquid\_\_\_ Solid X Sludge\_\_\_ Gas\_\_\_ Dust X

**b) Characteristics**

Corrosive\_\_\_ Ignitable\_\_\_ Radioactive\_\_\_ Volatile X  
Toxic X Reactive\_\_\_ Unknown\_\_\_ Other \_\_\_

## Article II. Hazard Evaluation

### CHEMICAL HAZARDS

Based upon review of the historical site location and use, potential chemical hazards on the site include: semi-volatile organic compounds (SVOCs), polycyclic aromatic hydrocarbons (PAHs), and Resource Conservation and Recovery Act (RCRA) 8 Metals. Site personnel are trained in hazard recognition and will use personal protective equipment (PPE) appropriate to the potential hazards.

### AIR MONITORING

If the project manager, or site supervisor, deems it appropriate, they may employ direct read air monitoring equipment for contaminants and toxic or flammable atmospheres prior to collecting samples.

### GENERAL SAFETY HAZARDS

Sampling at the proposed sites is unlikely to pose any unanticipated safety hazard to workers. The proposed scheme involves subsurface soil sampling. TerraGraphics site investigators are 40-hour HAZWOPER trained and can identify site hazards during site investigations.

There will be no confined space hazard. If sampling will be performed along roads and alleys, personnel will don "OSHA Orange" vests and traffic control measures will be initiated. The site supervisor will identify any site-specific hazards during pre-job safety meetings. The site supervisor will update employees if site hazards change.

The most likely hazards to be encountered are those commonly encountered on many work-sites (heat stress, working around machinery, noise, etc.). All TerraGraphics employees performing field work on this project will comply with the most current Health and Safety Manual and Health and Safety Standard Operating Procedures for TerraGraphics. Each employee has been provided access to this manual.

### DRILL RIG/SOIL BORINGS

Hazards generally associated with drilling operations are listed in the following bullet points. General work practices follow each hazard.

- Shut down drill rig and/or divert exhaust fumes (i.e., carbon monoxide) if they become a hazard.
- Overhead utility wires (i.e., electrical and telephone) can be hazardous when the drill rig mast is in the upright position. To avoid contact with any overhead lines, the drill rig mast will be lowered prior to moving the rig. Overhead utilities will be considered "live" until determined otherwise. The rig mast will not be erected within 10 feet of an overhead electrical line until the line is de-energized, grounded, or shielded and an electrician has certified that arcing cannot occur.
- Underground pipelines and utility lines can be ruptured or damaged during active drilling operations
- Moving parts (i.e., augers) on the drill rig may catch clothing. Free or falling parts from the mast head may cause head injury. Hard hats will be worn at all times when working around a drill rig. Secure loose clothing. Check the drill rig mast prior to approaching the drill rig.
- Ear protection will be worn during drill rig operations.

- Moving the drill rig over uneven terrain may cause the vehicle to roll over or get stuck in a rut or mud. Be aware of hazards associated with moving heavy machinery and other associated injury.
- All chains, lines, cables will be inspected daily for weak spots, frays, etc. High pressure hydraulic lines and air lines used on drill rigs are hazardous when they are in disrepair or incorrectly assembled. All high pressure lines will be checked prior to and during use.

### **Article III. Work Practices**

Workers will comply with all TerraGraphics Health and Safety Manual rules. Workers will comply with all state and federal regulations.

#### **PERSONAL PROTECTIVE EQUIPMENT**

Section 100.5 of the most current Health and Safety Manual and Health and Safety Standard Operating Procedures for TerraGraphics addresses PPE selection:

- A Class A, B, or C hard hat as appropriate to the site,
- Steel toe foot protection (ASTM F2413-05)
- Hearing protection, and
- Safety Glasses.

#### **DECONTAMINATION PROCEDURES**

##### ***a) Personnel***

Before leaving the sample area, thoroughly wash hands and face with soap and water before eating, drinking, or smoking. If water is not available, use pre-moistened towelettes to wash face and hands.

Do not track contaminated soils and dust off-site.

##### ***b) Samples***

After the sample containers are filled they will be sealed shut, marked with indelible marker, and any excess dirt will be wiped from the outside of the sample containers before they are stored. Sample containers will be transported in suitable sealed containers placed in stable containers that can be securely closed.

##### ***c) Disposal of Materials Generated On-Site***

Collect trash and non-hazardous waste and place it in appropriate trash receptacles for municipal trash collection. Potentially contaminated materials will be separated, sealed in chemically compatible containers, and labeled for appropriate off-site disposal.

##### ***d) Safety Equipment and Materials***

Each sampling team will have access to a first aid kit, clean water, paper cups, and pre-moistened towelettes. Site supervisors will ensure appropriate safety gear is available for site operations. The site supervisor will also be equipped with a cell phone in case of an emergency requiring outside assistance.

### **Article IV. Emergency Procedures**

If an injury occurs, take the following steps:

- Prevent further injury and notify the site supervisor.
- Initiate first aid and get medical attention for the injured person immediately.

- Depending on the type and severity of the injury, call for medical attention.
- Prepare an incident report.
- The crew chief/site safety officer will assume charge during a medical emergency.

***a) Local Emergency Phone Numbers***

Ambulance:	911
Hospital:	
Kootenai Health	(208) 625-4000 (non-emergency)
2003 Kootenai Health Way	911 (emergency department)
Coeur d'Alene, ID 83814	
Poison Control Center:	800-222-1222
Police:	911
	(208) 769-2320 (non-emergency)
Fire Department:	911
	(208) 769-2340 (non-emergency)

***b) Emergency Contacts***

8 am to 5 pm:	TerraGraphics Boise office	(208) 336-7080
After hours:	Jon Munkers (Mobile)	(208) 791-3663
	Melody Studer (Mobile)	(208) 918-7075

Article V. Site Organization

Map/Sketch Attached	<u>YES</u>	Site Secured	<u>NO</u>
Perimeter Identified	<u>YES</u>		



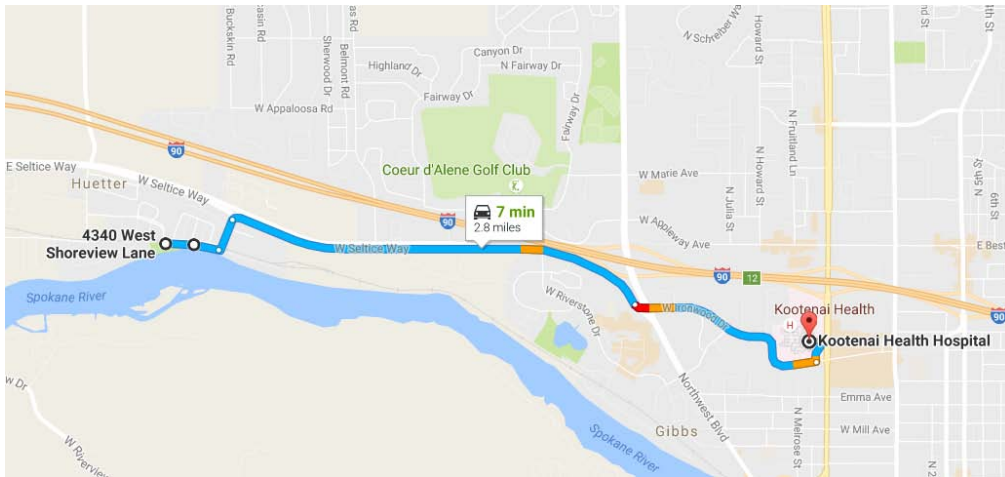
## EMERGENCY ROUTE

Driving directions to Kootenai Health

### From Zone 1

Total Travel Estimates: about 7 minutes / 2.8 miles

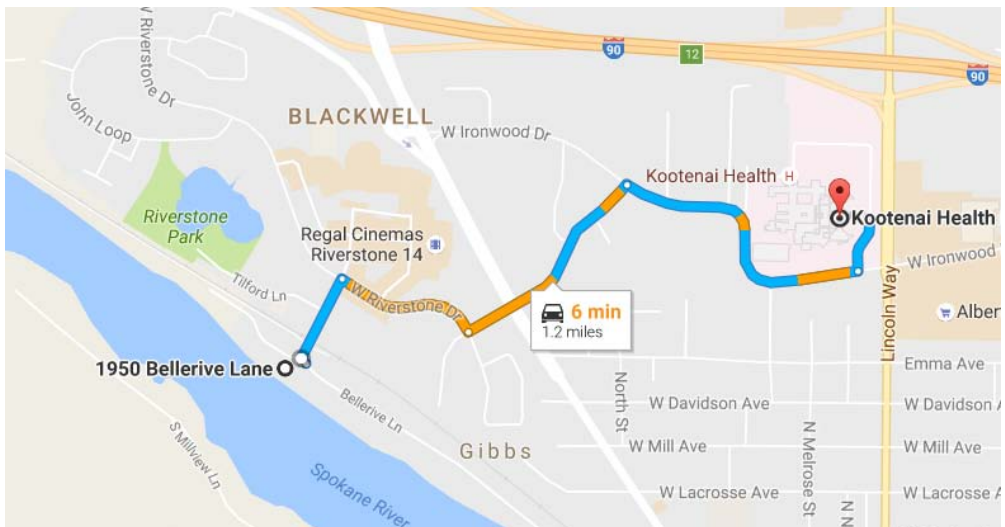
- |  |         |
|--|---------|
| 1. Head <b>EAST</b> on <b>W. Shoreview</b> toward Grand Mill                       | 0.1 mi  |
| 2. Turn <b>LEFT</b> onto <b>Grand Mill Ln.</b>                                     | 0.1 mi  |
| 3. Turn <b>RIGHT</b> onto <b>W. Seltice Way</b>                                    | 1.6 mi  |
| 4. Turn <b>LEFT</b> onto <b>W. Ironwood Dr.</b>                                    | 0.5 mi  |
| 5. Turn <b>LEFT</b> onto <b>Ironwood Ct.</b>                                       | 292 ft  |
| 6. Turn <b>RIGHT</b> onto <b>Kootenai Health Way</b> , Destination on <b>RIGHT</b> | 318 ft. |



### From Zone 2

Total Travel Estimates: about 5 minutes / 1.9 miles

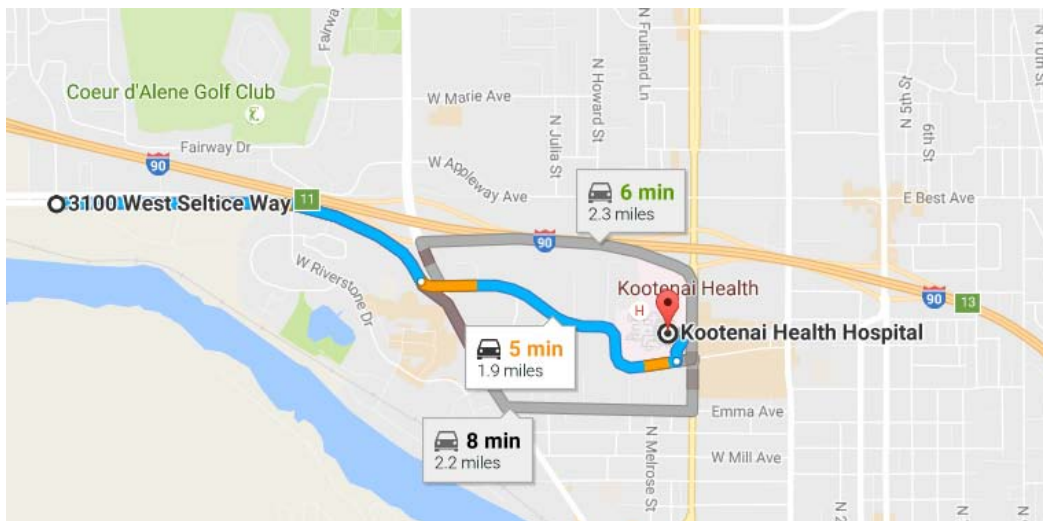
- |  |         |
|--|---------|
| 1. Head <b>EAST</b> on <b>W. Seltice Way</b>                                       | 1.0 mi  |
| 2. Turn <b>LEFT</b> onto <b>W. Ironwood Dr.</b>                                    | 0.5 mi  |
| 3. Turn <b>LEFT</b> onto <b>Ironwood Ct.</b>                                       | 292 ft  |
| 4. Turn <b>RIGHT</b> onto <b>Kootenai Health Way</b> , Destination on <b>RIGHT</b> | 318 ft. |



## From Zone 3

Total Travel Estimates: about 6 minutes / 1.2 miles

1. Head **NORTHWEST** on Bellervie Lane toward N. Beebe Blvd 52 ft
2. Turn **RIGHT** onto **N. Beebe Blvd** 0.1 mi
3. Turn **RIGHT** onto **W. Riverstone Dr.** 0.2 mi
4. Turn **LEFT** onto **N. Lakewood Dr.** 0.3 mi
5. Turn **RIGHT** onto **W. Ironwood Dr.** 0.4 mi
6. Turn **LEFT** onto **Kootenai Health Way**, Destination on **LEFT** 427 ft.



ATTACHMENT 1  
HEALTH AND SAFETY PLAN ACCEPTANCE FORM

**HEALTH AND SAFETY PLAN ACCEPTANCE FORM**  
**PROPERTY SAMPLING ACTIVITIES**

I, \_\_\_\_\_, have read, understand, and agree to abide by all requirements of the Site Health and Safety Plan (HSP) for BNSF ROW Sampling Activities.

I understand that my failure to abide by any aspect of the HSP can lead to disciplinary action, including immediate permanent removal from the project.

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date

**ATTACHMENT 2**  
**INCIDENT RESPONSE REPORT**

Today's Date:	Employee:
Incident Date:	Medical Care provided? <input type="checkbox"/> Yes <input type="checkbox"/> No
Incident Time: <input type="checkbox"/> am/ <input type="checkbox"/> pm	Filled out by:

**A. Incident/Near Miss Report**

**This side to be filled out by employee and supervisor/HR/H&S Representative**

**Name of individual(s) involved:**

**Location of incident:** (TG office building and room, or address and general description)

**Description of task being performed, related to what project?** (If applicable)

**Description of incident:** (What events occurred, etc.?)

**Part(s) of body injured:** (If applicable)

**Description of medical care or first aid received:** (List health care provider)

**Potential cause(s) of incident:** (Describe equipment or items contributing to incident.)

**Action(s) taken or proposed to reduce chance of reoccurrence:**

**Incident Category:**  Injury  Non-Injury  Near-Miss  
 Property Damage  Exposure to Hazardous Substance  
 Other: \_\_\_\_\_

**Incident Severity:**  First Aid Only  Minor medical  Serious  
 No lost time  Lost Time  Hospitalized

**Employee signature:** \_\_\_\_\_

**Supervisor printed name and signature:** \_\_\_\_\_

Employee:	Supervisor:
Incident date:	Incident time:
H&S/HR rep.:	Date this form completed:

**B. Additional Information/Documentation - Internal**

**This side to be completed by H&S or HR representative**

1. Is external report required?  Yes  No  
Has this report been made?  Yes  No.  
If so, to whom was report made? (Name and agency) \_\_\_\_\_
2. Were training requirements for the job met?  Yes  No
3. Was protective equipment used or were protective measures being taken?  Yes  No  
Describe:
4. Were safety procedures being followed?  Yes  No  
Which ones?
5. Was employee working alone?  Yes  No  
If no, who was present?
6. Witness Interviews: (name of witness; date and time; add extra sheet if needed)
7. Hazards Identified:
8. Other Comments:
9. Recommendations:
10. Recommendations Approved:  Yes  No  
By: \_\_\_\_\_ Date: \_\_\_\_\_
11. **Follow up** after  days/  weeks. Update provided to client?  Yes  No  
**New status** (if changed):

**ATTACHMENT 3**  
**TAILGATE MEETING REPORT**



## Tailgate Meeting Report

Date of Meeting: \_\_\_\_\_

Company Name: \_\_\_\_\_

Names of Attendees:

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Discussion Items:

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Problem Areas, Issues, or Concerns:

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Corrective Actions Taken:

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Signature: \_\_\_\_\_

## Appendix C Laboratory Quality Assurance/Quality Control Tables

TerraGraphics

Table 1. Precision Criteria of Field Duplicates for Soil

where: RPD = relative percent difference

C<sub>1</sub> = concentration in the first sample

C<sub>2</sub> = concentration in the second/duplicate sample

$$RPD = \left( \frac{C_1 - C_2}{\left( \frac{C_1 + C_2}{2} \right)} \right) \times 100\%$$

Where both C<sub>1</sub> and C<sub>2</sub> > 5 times the laboratory Method Detection Limit (MDL).

Where one or both C<sub>1</sub> and C<sub>2</sub> are less than 5 times the MDL, the results will be considered within control limits where C<sub>1</sub> and C<sub>2</sub> are ± MDL.

PAHs	USEPA Method	RPD
ANTHRACENE	USEPA 8270D-SIM	±50.0%
ACENAPHTHENE	USEPA 8270D-SIM	±50.0%
BENZO(A)ANTHRACENE	USEPA 8270D-SIM	±50.0%
BENZO(A)PYRENE	USEPA 8270D-SIM	±50.0%
BENZO(B)FLUORANTHENE	USEPA 8270D-SIM	±50.0%
BENZO(K)FLUORANTHENE	USEPA 8270D-SIM	±50.0%
CHRYSENE	USEPA 8270D-SIM	±50.0%
FLUORANTHENE	USEPA 8270D-SIM	±50.0%
FLUORENE	USEPA 8270D-SIM	±50.0%
PYRENE	USEPA 8270D-SIM	±50.0%

SVOCs	USEPA Method	RPD
ACENAPHTHYLENE	USEPA 8270D	±50.0%
BENZIDINE	USEPA 8270D	±50.0%
BENZO(G,H,I)PERYLENE	USEPA 8270D	±50.0%
BIS(2-CHLORETHOXY)METHANE	USEPA 8270D	±50.0%
BIS(2-CHLOROETHYL)ETHER	USEPA 8270D	±50.0%
BIS(2-CHLOROISOPROPYL)ETHER	USEPA 8270D	±50.0%
4-BROMOPHENYL-PHENYLEETHER	USEPA 8270D	±50.0%
2-CHLORONAPHTHALENE	USEPA 8270D	±50.0%
4-CHLOROPHENYL-PHENYLEETHER	USEPA 8270D	±50.0%
DIBENZ(A,H)ANTHRACENE	USEPA 8270D	±50.0%
3,3-DICHLOROBENZIDINE	USEPA 8270D	±50.0%
2,4-DINITROTOLUENE	USEPA 8270D	±50.0%
2,6-DINITROTOLUENE	USEPA 8270D	±50.0%
HEXACHLOROBENZENE	USEPA 8270D	±50.0%
HEXACHLORO-1,3-BUTADIENE	USEPA 8270D	±50.0%
HEXACHLOROCYCLOPENTADIENE	USEPA 8270D	±50.0%
HEXACHLOROETHANE	USEPA 8270D	±50.0%
INDENO(1,2,3-CD)PYRENE	USEPA 8270D	±50.0%
ISOPHORONE	USEPA 8270D	±50.0%
NAPHTHALENE	USEPA 8270D	±50.0%
NITROBENZENE	USEPA 8270D	±50.0%
N-NITROSODIMETHYLAMINE	USEPA 8270D	±50.0%
N-NITROSODIPHENYLAMINE	USEPA 8270D	±50.0%
N-NITROSODI-N-PROPYLAMINE	USEPA 8270D	±50.0%
PHENANTHRENE	USEPA 8270D	±50.0%
BENZYL BUTYL PHTHALATE	USEPA 8270D	±50.0%
BIS(2-ETHYLHEXYL)PHTHALATE	USEPA 8270D	±50.0%
DI-N-BUTYL PHTHALATE	USEPA 8270D	±50.0%
DIETHYL PHTHALATE	USEPA 8270D	±50.0%
DIMETHYL PHTHALATE	USEPA 8270D	±50.0%
DI-N-OCTYL PHTHALATE	USEPA 8270D	±50.0%
1,2,4-TRICHLOROBENZENE	USEPA 8270D	±50.0%
4-CHLORO-3-METHYLPHENOL	USEPA 8270D	±50.0%
2-CHLOROPHENOL	USEPA 8270D	±50.0%
2,4-DICHLOROPHENOL	USEPA 8270D	±50.0%
2,4-DIMETHYLPHENOL	USEPA 8270D	±50.0%
4,6-DINITRO-2-METHYLPHENOL	USEPA 8270D	±50.0%
2,4-DINITROPHENOL	USEPA 8270D	±50.0%
2-NITROPHENOL	USEPA 8270D	±50.0%
4-NITROPHENOL	USEPA 8270D	±50.0%
PENTACHLOROPHENOL	USEPA 8270D	±50.0%
PHENOL	USEPA 8270D	±50.0%
2,4,6-TRICHLOROPHENOL	USEPA 8270D	±50.0%

RCRA 8 Metals	USEPA Method	RPD
ARSENIC	USEPA 6010C	±50.0%
BARIUM	USEPA 6010C	±50.0%
CADMIUM	USEPA 6010C	±50.0%
CHROMIUM	USEPA 6010C	±50.0%
LEAD	USEPA 6010C	±50.0%
SELENIUM	USEPA 6010C	±50.0%
SILVER	USEPA 6010C	±50.0%
MERCURY	USEPA 7471B	±50.0%

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Table 2. Precision Criteria of Lab Control Sample Duplicates (LCSDs) and Matrix Spike Duplicates (MSDs) for Soil

where: RPD = relative percent difference  
 C<sub>1</sub> = concentration in the first sample  
 C<sub>2</sub> = concentration in the second/duplicate sample

$$RPD = \left| \frac{(C_1 - C_2)}{\left(\frac{C_1 + C_2}{2}\right)} \right| \times 100\%$$

Where both C<sub>1</sub> and C<sub>2</sub> > 5 times the laboratory Method Detection Limit (MDL).

Where one or both C<sub>1</sub> and C<sub>2</sub> are less than 5 times the MDL, the results will be considered within control limits where C<sub>1</sub> and C<sub>2</sub> are ± MDL.

RPD

PAHs	USEPA Method	LCSD	MSD
ANTHRACENE	USEPA 8270D-SIM	±20.0%	±20.7%
ACENAPHTHENE	USEPA 8270D-SIM	±20.0%	±20.0%
BENZO(A)ANTHRACENE	USEPA 8270D-SIM	±20.0%	±24.7%
BENZO(A)PYRENE	USEPA 8270D-SIM	±20.0%	±25.3%
BENZO(B)FLUORANTHENE	USEPA 8270D-SIM	±20.0%	±29.5%
BENZO(K)FLUORANTHENE	USEPA 8270D-SIM	±20.0%	±26.1%
CHRYSENE	USEPA 8270D-SIM	±20.0%	±25.7%
FLUORANTHENE	USEPA 8270D-SIM	±20.0%	±26.0%
FLUORENE	USEPA 8270D-SIM	±20.0%	±20.0%
PYRENE	USEPA 8270D-SIM	±20.0%	±25.1%

SVOCs	USEPA Method	LCSD	MSD
ACENAPHTHYLENE	USEPA 8270D	±20.0%	±25.9%
BENZIDINE	USEPA 8270D	±40.0%	±40.0%
BENZO(G,H,I)PERYLENE	USEPA 8270D	±20.0%	±31.9%
BIS(2-CHLOROETHOXY)METHANE	USEPA 8270D	±20.0%	±26.1%
BIS(2-CHLOROETHYL)ETHER	USEPA 8270D	±26.0%	±33.6%
BIS(2-CHLOROISOPROPYL)ETHER	USEPA 8270D	±20.7%	±31.7%
4-BROMOPHENYL-PHENYLEETHER	USEPA 8270D	±20.0%	±26.0%
2-CHLORONAPHTHALENE	USEPA 8270D	±20.0%	±26.5%
4-CHLOROPHENYL-PHENYLEETHER	USEPA 8270D	±20.0%	±25.9%
DIBENZ(A,H)ANTHRACENE	USEPA 8270D	±20.0%	±29.5%
3,3-DICHLOROBENZIDINE	USEPA 8270D	±22.0%	±40.0%
2,4-DINITROTOLUENE	USEPA 8270D	±20.0%	±29.7%
2,6-DINITROTOLUENE	USEPA 8270D	±20.0%	±29.7%
HEXACHLOROBENZENE	USEPA 8270D	±20.1%	±25.4%
HEXACHLORO-1,3-BUTADIENE	USEPA 8270D	±20.0%	±29.7%
HEXACHLOROCYCLOPENTADIENE	USEPA 8270D	±20.7%	±37.5%
HEXACHLOROETHANE	USEPA 8270D	±22.7%	±31.9%
INDENO(1,2,3-CD)PYRENE	USEPA 8270D	±20.0%	±31.5%
ISOPHORONE	USEPA 8270D	±20.0%	±27.7%
NAPHTHALENE	USEPA 8270D	±20.0%	±27.2%
NITROBENZENE	USEPA 8270D	±21.0%	±27.8%
N-NITROSODIMETHYLAMINE	USEPA 8270D	±23.5%	±32.0%
N-NITROSODIPHENYLAMINE	USEPA 8270D	±20.0%	±25.9%
N-NITROSODI-N-PROPYLAMINE	USEPA 8270D	±20.0%	±28.2%
PHENANTHRENE	USEPA 8270D	±20.0%	±26.5%
BENZYL BUTYL PHTHALATE	USEPA 8270D	±20.0%	±28.5%
BIS(2-ETHYLHEXYL)PHTHALATE	USEPA 8270D	±20.5%	±35.2%
DI-N-BUTYL PHTHALATE	USEPA 8270D	±20.0%	±25.9%
DIETHYL PHTHALATE	USEPA 8270D	±20.0%	±25.5%
DIMETHYL PHTHALATE	USEPA 8270D	±20.0%	±25.4%
DI-N-OCTYL PHTHALATE	USEPA 8270D	±22.0%	±32.5%
1,2,4-TRICHLOROBENZENE	USEPA 8270D	±20.0%	±28.4%
4-CHLORO-3-METHYLPHENOL	USEPA 8270D	±20.0%	±26.6%
2-CHLOROPHENOL	USEPA 8270D	±20.0%	±29.3%
2,4-DICHLOROPHENOL	USEPA 8270D	±20.0%	±27.3%
2,4-DIMETHYLPHENOL	USEPA 8270D	±20.0%	±32.3%
4,6-DINITRO-2-METHYLPHENOL	USEPA 8270D	±23.7%	±32.7%
2,4-DINITROPHENOL	USEPA 8270D	±36.5%	±39.4%
2-NITROPHENOL	USEPA 8270D	±20.9%	±29.9%
4-NITROPHENOL	USEPA 8270D	±20.0%	±30.2%
PENTACHLOROPHENOL	USEPA 8270D	±22.9%	±28.3%
PHENOL	USEPA 8270D	±20.0%	±29.6%
2,4,6-TRICHLOROPHENOL	USEPA 8270D	±20.0%	±28.1%

Table 3. Accuracy Criteria of Lab Matrix Spike (MS) Samples for Soil

MS and Surrogate Percent Recoveries

where: %R = percent recovery  
 $C_s$  = Measured concentration of Spiked Sample  
 $C_{us}$  = Measured concentration of Un-Spiked sample  
 $C_T$  = True concentration of Spike added

$$\%R = (C_s - C_{us}) / C_T \times 100\%$$

Percent Recovery

PAHs	USEPA Method	Low %	High %
ANTHRACENE	USEPA 8270D-SIM	36.7	144
ACENAPHTHENE	USEPA 8270D-SIM	39.4	132
BENZO(A)ANTHRACENE	USEPA 8270D-SIM	28.0	144
BENZO(A)PYRENE	USEPA 8270D-SIM	23.8	147
BENZO(B)FLUORANTHENE	USEPA 8270D-SIM	18.2	147
BENZO(K)FLUORANTHENE	USEPA 8270D-SIM	26.5	143
CHRYSENE	USEPA 8270D-SIM	27.4	150
FLUORANTHENE	USEPA 8270D-SIM	23.2	158
FLUORENE	USEPA 8270D-SIM	30.8	139
PYRENE	USEPA 8270D-SIM	22.6	151

Percent Recovery

SVOCs	USEPA Method	Low %	High %
ACENAPHTHYLENE	USEPA 8270D	38.7	129
BENZIDINE	USEPA 8270D	0.00	49.9
BENZO(G,H,I)PERYLENE	USEPA 8270D	10.0	127
BIS(2-CHLOROETHOXY)METHANE	USEPA 8270D	35	132
BIS(2-CHLOROETHYL)ETHER	USEPA 8270D	28.8	128
BIS(2-CHLOROISOPROPYL)ETHER	USEPA 8270D	31.8	118
4-BROMOPHENYL-PHENYLEETHER	USEPA 8270D	39.0	130
2-CHLORONAPHTHALENE	USEPA 8270D	37.5	123
4-CHLOROPHENYL-PHENYLEETHER	USEPA 8270D	37.9	123
DIBENZ(A,H)ANTHRACENE	USEPA 8270D	10.5	128
3,3-DICHLOROBENZIDINE	USEPA 8270D	10.0	129
2,4-DINITROTOLUENE	USEPA 8270D	27.8	147
2,6-DINITROTOLUENE	USEPA 8270D	36.5	137
HEXACHLOROBENZENE	USEPA 8270D	34.4	116
HEXACHLORO-1,3-BUTADIENE	USEPA 8270D	36.5	125
HEXACHLOROCYCLOPENTADIENE	USEPA 8270D	10.0	124
HEXACHLOROETHANE	USEPA 8270D	11.3	143
INDENO(1,2,3-CD)PYRENE	USEPA 8270D	10.0	128
ISOPHORONE	USEPA 8270D	25.7	116
NAPHTHALENE	USEPA 8270D	36.4	121
NITROBENZENE	USEPA 8270D	30.9	134
N-NITROSODIMETHYLAMINE	USEPA 8270D	19.2	127
N-NITROSODIPHENYLAMINE	USEPA 8270D	26.8	133
N-NITROSODI-N-PROPYLAMINE	USEPA 8270D	33.0	134
PHENANTHRENE	USEPA 8270D	30.8	137
BENZYL BUTYL PHTHALATE	USEPA 8270D	33.4	128
BIS(2-ETHYLHEXYL)PHTHALATE	USEPA 8270D	21.8	141
DI-N-BUTYL PHTHALATE	USEPA 8270D	32.2	133
DIETHYL PHTHALATE	USEPA 8270D	39.4	136
DIMETHYL PHTHALATE	USEPA 8270D	35.8	137
DI-N-OCTYL PHTHALATE	USEPA 8270D	28.5	128
1,2,4-TRICHLOROBENZENE	USEPA 8270D	36.5	114
4-CHLORO-3-METHYLPHENOL	USEPA 8270D	27	154
2-CHLOROPHENOL	USEPA 8270D	33.2	121
2,4-DICHLOROPHENOL	USEPA 8270D	34.8	134
2,4-DIMETHYLPHENOL	USEPA 8270D	12.3	149
4,6-DINITRO-2-METHYLPHENOL	USEPA 8270D	10.0	144
2,4-DINITROPHENOL	USEPA 8270D	10.0	121
2-NITROPHENOL	USEPA 8270D	29.5	144
4-NITROPHENOL	USEPA 8270D	20.0	133
PENTACHLOROPHENOL	USEPA 8270D	10.0	139
PHENOL	USEPA 8270D	25.1	130
2,4,6-TRICHLOROPHENOL	USEPA 8270D	33.8	133

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Table 4. Accuracy Criteria of Lab Control Samples (LCS) for Soil

LCS Percent Recovery

$$\%R = C_M / C_T \times 100\%$$

where: %R = percent recovery  
 C<sub>M</sub> = Measured spike/LCS concentration  
 C<sub>T</sub> = True spike/LCS concentration

PAHs	USEPA Method	Percent Recovery	
		Low %	High %
ANTHRACENE	USEPA 8270D-SIM	51.3	136
ACENAPHTHENE	USEPA 8270D-SIM	48.7	127
BENZO(A)ANTHRACENE	USEPA 8270D-SIM	55.0	126
BENZO(A)PYRENE	USEPA 8270D-SIM	51.9	127
BENZO(B)FLUORANTHENE	USEPA 8270D-SIM	54.0	125
BENZO(K)FLUORANTHENE	USEPA 8270D-SIM	53.9	132
CHRYSENE	USEPA 8270D-SIM	55.7	133
FLUORANTHENE	USEPA 8270D-SIM	54.0	132
FLUORENE	USEPA 8270D-SIM	48.7	127
PYRENE	USEPA 8270D-SIM	54.0	129

SVOCs	USEPA Method	Percent Recovery	
		Low %	High %
ACENAPHTHYLENE	USEPA 8270D	49.2	111
BENZIDINE	USEPA 8270D	0.00	48.0
BENZO(G,H,I)PERYLENE	USEPA 8270D	45.8	108
BIS(2-CHLOROETHOXY)METHANE	USEPA 8270D	44.9	108
BIS(2-CHLOROETHYL)ETHER	USEPA 8270D	32.5	112
BIS(2-CHLOROISOPROPYL)ETHER	USEPA 8270D	40.4	99.0
4-BROMOPHENYL-PHENYLEETHER	USEPA 8270D	51.4	110
2-CHLORONAPHTHALENE	USEPA 8270D	47.1	105
4-CHLOROPHENYL-PHENYLEETHER	USEPA 8270D	48.1	108
DIBENZ(A,H)ANTHRACENE	USEPA 8270D	45.7	111
3,3-DICHLOROBENZIDINE	USEPA 8270D	21.0	101
2,4-DINITROTOLUENE	USEPA 8270D	53.0	112
2,6-DINITROTOLUENE	USEPA 8270D	51.6	110
HEXACHLOROBENZENE	USEPA 8270D	43.2	104
HEXACHLORO-1,3-BUTADIENE	USEPA 8270D	41.5	112
HEXACHLOROCYCLOPENTADIENE	USEPA 8270D	13.5	123
HEXACHLOROETHANE	USEPA 8270D	36.2	103
INDENO(1,2,3-CD)PYRENE	USEPA 8270D	47.5	109
ISOPHORONE	USEPA 8270D	28.8	104
NAPHTHALENE	USEPA 8270D	43.4	103
NITROBENZENE	USEPA 8270D	40.7	109
N-NITROSODIMETHYLAMINE	USEPA 8270D	18.1	122
N-NITROSODIPHENYLAMINE	USEPA 8270D	48.8	107
N-NITROSODI-N-PROPYLAMINE	USEPA 8270D	43.3	109
PHENANTHRENE	USEPA 8270D	51.6	107
BENZYL BUTYL PHTHALATE	USEPA 8270D	47.5	115
BIS(2-ETHYLHEXYL)PHTHALATE	USEPA 8270D	48.1	116
DI-N-BUTYL PHTHALATE	USEPA 8270D	49.7	113
DIETHYL PHTHALATE	USEPA 8270D	52.0	112
DIMETHYL PHTHALATE	USEPA 8270D	51.4	108
DI-N-OCTYL PHTHALATE	USEPA 8270D	49.6	112
1,2,4-TRICHLOROBENZENE	USEPA 8270D	39.8	100
4-CHLORO-3-METHYLPHENOL	USEPA 8270D	51.1	113
2-CHLOROPHENOL	USEPA 8270D	40.8	103
2,4-DICHLOROPHENOL	USEPA 8270D	46.2	109
2,4-DIMETHYLPHENOL	USEPA 8270D	42.2	110
4,6-DINITRO-2-METHYLPHENOL	USEPA 8270D	23.1	119
2,4-DINITROPHENOL	USEPA 8270D	10.0	105
2-NITROPHENOL	USEPA 8270D	44.2	113
4-NITROPHENOL	USEPA 8270D	34.8	109
PENTACHLOROPHENOL	USEPA 8270D	16.2	102
PHENOL	USEPA 8270D	41.5	106
2,4,6-TRICHLOROPHENOL	USEPA 8270D	44.4	108

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**Table 5. Precision Criteria of Lab Control Sample Duplicates (LCSDs) and Matrix Spike Duplicates (MSDs) for Soil**

where: RPD = relative percent difference  
 C<sub>1</sub> = concentration in the first sample  
 C<sub>2</sub> = concentration in the second/duplicate sample

$$RPD = \left| \frac{(C_1 - C_2)}{\left(\frac{C_1 + C_2}{2}\right)} \right| \times 100\%$$

Where both C<sub>1</sub> and C<sub>2</sub> > 5 times the laboratory Method Detection Limit (MDL).

Where one or both C<sub>1</sub> and C<sub>2</sub> are less than 5 times the MDL, the results will be considered within control limits where C<sub>1</sub> and C<sub>2</sub> are ± MDL.

RCRA 8 Metals	USEPA Method	RPD	
		LCSD	MSD
ARSENIC	USEPA 6010C	±20.0%	±20.0%
BARIUM	USEPA 6010C	±20.0%	±20.0%
CADMIUM	USEPA 6010C	±20.0%	±20.0%
CHROMIUM	USEPA 6010C	±20.0%	±20.0%
LEAD	USEPA 6010C	±20.0%	±20.0%
SELENIUM	USEPA 6010C	±20.0%	±20.0%
SILVER	USEPA 6010C	±20.0%	±20.0%
MERCURY	USEPA 7471B	±20.0%	±20.0%

SVL Analytical

Table 6. Accuracy Criteria of Lab Matrix Spike (MS) Samples for Soil

MS and Surrogate Percent Recoveries

where: %R = percent recovery  
 $C_s$  = Measured concentration of Spiked Sample  
 $C_{US}$  = Measured concentration of Un-Spiked sample  
 $C_T$  = True concentration of Spike added

$$\%R = (C_s - C_{US}) / C_T \times 100\%$$

Percent Recovery

RCRA 8 Metals	USEPA Method	Low %	High %
ARSENIC	USEPA 6010C	75	125
BARIUM	USEPA 6010C	75	125
CADMIUM	USEPA 6010C	75	125
CHROMIUM	USEPA 6010C	75	125
LEAD	USEPA 6010C	75	125
SELENIUM	USEPA 6010C	75	125
SILVER	USEPA 6010C	75	125
MERCURY	USEPA 7471B	80	120



SVL Analytical

Table 7. Accuracy Criteria of Lab Control Samples (LCS) for Soil

LCS Percent Recovery

$$\%R = C_M / C_T \times 100\%$$

where: %R = percent recovery  
 C<sub>M</sub> = Measured spike/LCS concentration  
 C<sub>T</sub> = True spike/LCS concentration

RCRA 8 Metals	USEPA Method	Percent Recovery	
		Low %	High %
ARSENIC	USEPA 6010C	80	120
BARIUM	USEPA 6010C	80	120
CADMIUM	USEPA 6010C	80	120
CHROMIUM	USEPA 6010C	80	120
LEAD	USEPA 6010C	80	120
SELENIUM	USEPA 6010C	80	120
SILVER	USEPA 6010C	80	120
MERCURY	USEPA 7471B	80	120

**ESC Lab Sciences and SVL Analytical**  
**Table 8. Integrity of Samples**

<b>QA/QC Criterion</b>	<b>Sample Type</b>	<b>Frequency</b>	<b>Estimated Number</b>	<b>QA Objectives</b>
Integrity of field sample collection and handling	Method Blank	1:20 samples	1	No detects or all sample results >10x detect

**Appendix D Quality Assurance Manual and Method Specific SOPs for SVL Analytical,  
Inc.**

# Quality Manual

SVL ANALYTICAL, INC.

P.O. Box 929

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Kellogg, Idaho 83837

208-784-1258

FAX 208-783-0891

Effective Date: 01/20/2015

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President and CEO  
Wayne R. Sorensen

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Date

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Laboratory Director  
John R. Kern

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Date

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Quality Manager  
Michael Desmarais

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Date

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Technical Director  
Kirby L. Gray

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Date

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Technical Director  
Nan S. Wilson

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Date

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Systems Manager  
Brandan Borgias

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Date

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Supervisor Inorganic Instrument Department  
Danny Sevy

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Supervisor Classical Chemistry Department  
Dianne Gardner

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Date

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Supervisor ABA Department  
Heather Green

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Date

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## **1.0 QUALITY POLICY STATEMENT**

SVL Analytical, Inc. (SVL) recognizes that an effective quality system is paramount to providing analytical data that is scientifically meaningful, legally defensible, technically accurate, and based upon the highest ethical standards. To reinforce the above objectives, SVL has committed itself to follow and be in compliance with the 2009 TNI Standards.

The emphasis of SVL's Quality Manual (QM) is to define control procedures for receipt, handling, and storage of samples; preparation and storage of standards; calibration and maintenance of analytical equipment; performance of analytical methods; customer service; and the generation, review, and reporting of analytical data.

At SVL, quality assurance begins with the definition of Data Quality Objectives (DQOs) and continues on through data reporting. Control procedures are defined for every step of the program as detailed in SVL's Standard Operating Procedures (SOPs). SVL realizes that without these controls in all phases of the analytical process, data may become suspect and hence of less value to our clients. Therefore, SVL is committed to providing data of the highest quality, usability, and defensibility for every project undertaken. SVL personnel concerned with any aspect of environmental testing are required to familiarize themselves with all quality documentation (including this manual) used at SVL and they are required to comply with all policies and procedures outlined therein.

SVL's Technical Management and Quality Manager ensure that this QM complies with all applicable TNI Quality System Standards and sees that it is reviewed annually and revised as needed. Evidence of signatory approval by senior management of this QM and SVL SOPs are available in PDF format by request.

SVL's commitment to client confidentiality (including national security concerns) and their proprietary rights is paramount to all operations conducted within its quality system; as such, a signed confidentiality statement is maintained in each employees personnel file.

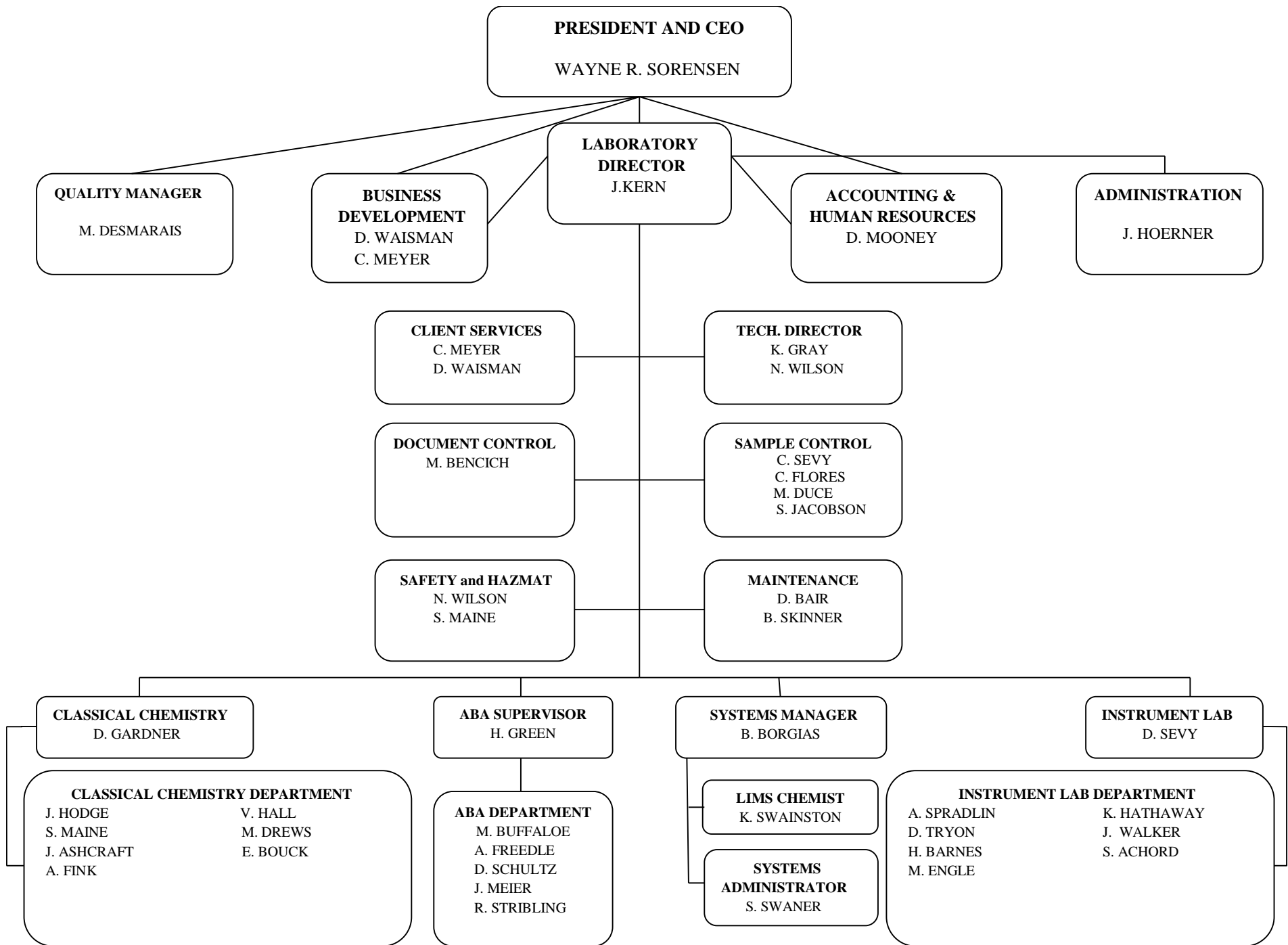
After reading this document employees are required to sign a signature page. By signing, the employee confirms that they have read, understood, and will comply with the Quality Manual and the TNI Standards it is based upon.

## **2.0 ORGANIZATION AND STRUCTURE**

The organizational structure of SVL follows a traditional scheme of management with a few modifications. The President/CEO is at the top of the chain of command followed immediately by the Laboratory Director, Quality Manager, Business Development and Accounting/Human Resources. The following supervisors/departments are managed under the Laboratory Director: Business Development, Accounting/Human Resources, Administration, Maintenance, Technical Directors, Document Control Office, Client Services, Safety and HAZMAT, Classical Chemistry, ABA, Instrument Lab and Systems Manager. The Systems Administrator and LIMS Chemist report to the Systems Manager. The Classical Department reports to the Classical Department Supervisor. The Instrument Lab Department reports to the Instrument Lab Department Supervisor. The ABA, Alkalinity and TDS labs report to the ABA Department Supervisor.

### **2.1 Organization Chart**





## 2.2 Employee List

Position	Employee	Degree	Years of Lab Experience
President and CEO	Wayne Sorensen	BS 1962	47
Laboratory Director	John R. Kern	MS 1982	31
Systems Manager	Brandan A. Borgias	PhD 1985	33
Document Control Officer	Melba Bencich		33
Client Services Manager	Christine Meyer		35
Business Development Manager	Dave Waisman	MS 1985	20
Technical Director	Kirby L. Gray	BS 1972	31
Technical Director/Safety Officer	Nan S. Wilson	BS 1996	18
Supervisor Inorganic Instrument	Danny Sevy		28
Supervisor Classical Chemistry	Dianne Gardner	BA 1987	10
Supervisor ABA	Heather Green	BS 2009	6
Systems Administrator	Scott Swaner		11
LIMS Chemist	Kale Swainston	BS 1998	16
Accounting and Human Resources	Donella Mooney		23
Quality Manager	Michael Desmarais	BS 1995	17
ICP Spectroscopist	Anne Spradlin	BA 1983	28
ICP Chemist	Matt Engle	MS 2012	2
ICP Analyst	David Tryon		10
ICP-MS and GFAA Analyst	Kevin Hathaway		28
IC Chemist	Justin Walker	BS 2014	<1
CVAA Chemist	Sam Achord	BS 2009	3
Analyst	Heidi Barnes		11
Chemist	Jim Hodge		47
Chemist	Victoria Hall	BS 2013	1
Chemist/HAZMAT Coordinator	Sherry Maine	MS 2004	13
Chemist	Matthew Drews	BS 2011	2
Chemist	Alicia Fink	BS 2014	<1
Chemist	Mikel Buffaloe	BS 2013	2
Analyst	Anita Guzman-Freedle	BS 1979	6
Analyst	Eric Bouck		6
Analyst	Debbie Schultz		12
Analyst	Robin Stribling		8
Analyst	Jerry Meier		4
Analyst	Judy Ashcraft		43
Sample Control Officer	Crystal Sevy		11
Sample Receiving	Cindy Flores		12
Sample Receiving	Mark Duce		3
Sample Receiving	Shelley Jacobson		1
Maintenance	Dan Bair		7
Maintenance	Ben Skinner		1
Receptionist	Jena Hoerner		1

## **2.3 Key Employee Resumes**

See Resumes pg. 60.

## **3.0 JOB DESCRIPTIONS**

### **3.1 Laboratory Director**

The Laboratory Director supervises day-to-day operations of the laboratory. Responsible for monitoring standards of performance in quality control and quality assurance, and for monitoring the validity of the analyses performed, and data generated in the laboratory. The Laboratory Director holds a weekly staff meeting to discuss client and technical issues.

### **3.2 Systems Manager**

The Systems Manager supervises operations of the Information Technology groups. The Systems Manager uses Excel, Crystal Reports, and other software to develop and maintain client reports and electronic data deliverables (EDDs). Element is the Laboratory's Information Management System (LIMS) and the Systems Manager works with the LIMS Chemist to make sure that Element meets the needs of SVL.

### **3.3 Department Supervisor**

Department supervisors conduct the day-to-day operations of the analytical departments. They are responsible for department safety and analyst training. They are also responsible for review of out-going analytical data.

### **3.4 Quality Manager**

The Quality Manager is responsible for implementation of the quality system. The Quality Manager manages the performance testing and NPDES program and conducts laboratory audits. The Quality Manager obtains and maintains laboratory accreditations, reviews and approves SOPs, conducts staff training in integrity and quality systems, and manages the CAR/PAR program. The Quality Manager is a TNI member.

### **3.5 Document Control Officer (DCO)**

DCO is responsible for the generation and the retention of analytical reports and records, including but not limited to Chains-of-Custody and

sample shipping documents. DCO is also responsible for delivering electronic data deliverables.

### **3.6 Sample Control Officer (SCO)**

SCO is responsible for sample receipt, job creation/verification, sample storage, and sample disposal.

### **3.7 Technical Director**

Technical Directors provide technical support to laboratory staff and provide final reviews of analytical data packages. Other responsibilities include Level III reporting.

### **3.8 Safety**

Safety Officer is responsible for revising the Chemical Hygiene Plan annually, conducts safety training and oversees response teams. Other duties include providing accident reports to the state.

### **3.9 Hazmat Officer**

Hazmat Officer is responsible for overseeing SVL's hazardous waste program (including setting up 8-hour refresher courses annually).

## **4.0 APPROVED LABORATORY SIGNATORIES**

The Laboratory Director John Kern, Systems Manager Brandan Borgias, Technical Directors Kirby Gray and Nan Wilson have full authority. Department Supervisors Dianne Gardner, Heather Green and Danny Sevy are approved laboratory signatories for analytical reports. LIMS Chemist Kale Swainston, DCO Melba Bencich and Quality Manager Michael Desmarais have report generation privileges.

## **5.0 RECORDS AND DOCUMENT CONTROL**

All records and documents are kept for 5 years unless otherwise specified by client contract. Electronic instrument data and LIMS data are kept for 10 years.

### **5.1 Standard Operating Procedures (SOPs)**

The Quality Manager retains the master copies of SOPs. Electronic copies are available on the laboratory's computer network. Signed and dated SOPs are available by request in PDF format. All SOPs are

scheduled for review each year. Electronic copies are available on the laboratory network on the date of the Quality Manager's final review. The SOP's effective date is two weeks after the date the Quality Manager signs the controlled copy. When a revision is created, the previous version is removed from the master file and electronic database. The retired controlled copy is retained in the SOP archive file.

## **5.2 Quality Manual (QM)**

The Quality Manager retains the controlled copy of the QM. The QM is scheduled for review annually or when revisions are needed.

Management may make hard copies available to accrediting authorities, laboratory staff and clients as needed; otherwise, the QM is available in electronic format. A signed and dated QM is available by request in PDF format. When a revision is created, copies are sent out to our accrediting bodies and previous versions are removed from use. The retired controlled copy is retained in the QM archive file.

## **5.3 Analytical Data**

The DCO retains analytical data, including calibration records and quality control. Documents are secured in storage containers.

## **5.4 Training Records**

The Quality Manager maintains records of analyst training and proficiency; ref, SOP SVL 1010. Documents are secured in storage containers.

## **5.5 Performance Testing Samples**

The Quality Manager maintains records of analysis of performance testing samples and the reports associated with the analyses. Reports are stored in the Quality Managers Outlook account under Inbox/ERA.

## **5.6 External and Internal Audits**

The Quality Manager retains records of external and internal audits. Reports are stored in Quality Managers office.

## **5.7 Corrective Action Reports (CARs)**

CARs are kept electronically and filed by hardcopy. CARs are stored in Quality Managers office.

## **5.8 Laboratory Logbooks**

SVL controls the issue, use, and closure of laboratory logbooks. The process is described in SOP SVL 2017. Examples of logbooks may include: the conductivity of laboratory water, preparation of reagents

and standards, preparation of samples, calibration of balances, calibration of micropipets, volumetric pipets and repipettors, maintenance of instruments, temperatures of ovens and refrigerators, etc. The Quality Manager assigns and archives logbooks. Documents are secured in storage containers. SVL is encouraging employees to switch over to electronic logbooks where possible.

#### **5.9 Chain of Custody (COC)**

The DCO is in charge of COC retention; they are currently held for five years, unless a longer time is required by contract. Sample log-in and job creation are maintained in SVL's LIMS. COCs and sample receiving check-in sheet are scanned into PDF format, which can be accessed through Element. Documents are secured in storage containers.

#### **5.10 Analytical Reports**

The DCO creates and retains both hardcopies and PDFs of analytical reports. Both types of analytical reports are stored in secured storage containers to protect them from damage.

#### **5.11 Backup and Storage of Electronic Data**

**5.11.1 Electronic Data Collection:** Currently the backup server is protected with an administrative password, which is changed every 6 months; it is in control of the Systems Administrator; ref, SOPs SVL 2020 and 2021.

**5.11.2 Archives of Electronic Data:** Data files that reside on the SVL file servers are backed up on a daily basis and kept onsite for 90 days: a full backup of the data files residing on the server is done monthly and sent to an offsite storage facility for 10 years (longer if required by contract). All software used to recover data files is also stored at the offsite facility for the same time frame.

**5.11.3 Backup Storage:** A secure fire-proof safe is maintained inside SVL to house the electronic data collected via the current backup system.

### **6.0 TRACEABILITY OF MEASUREMENTS**

#### **6.1 Chemicals and Reagents**

SVL uses reagent grade or better chemicals. Some equivalent grades are "VWR Omni-Trace", "Fisher Trace Metals", "Baker Instra-Analyzed",

“Baker A.C.S.”, “Baker Analyzed”, “Fisher A.C.S.”, and “Fisher Certified”. SVL requires a certificate of analysis or purity (certificates are scanned and attached to Element), for stock standards and reagents. Upon receipt, all chemical containers are labeled and entered into SVL’s LIMS.

SVL records the preparation of reagents and standards in controlled logbooks or electronically in the LIMS. The initials of the preparer, the date prepared, the lot number and amount of stock materials, the final volume, the matrix, instructions for preparation, and the expiration date are all recorded. A label is created within the LIMS with unique identifiers attached to all aliquots of the reagent/standard.

Preparation instructions are included in the SOPs for standards and reagents used in the analytical methods. SVL labels containers of prepared reagents and standards with their contents, a unique reference number, date prepared, disposal (expiration) date and a perceived hazard warning. Every aliquot is assigned a unique identifier.

SVL routinely obtains reference standards from commercial sources. These standards are used to check and document the concentration of calibration standards and validate method QC requirements.

SVL stores reagents and standards separately from samples.

## 6.2 Water

The primary reagent water in the laboratory is furnished by a reverse osmosis system followed by a micropore filter with an ion-exchange resin cartridge. This satisfies the specifications of ASTM Type II water. When Type I (18 MΩ-cm) water is required, SVL inserts a four-cartridge ion-exchange system or a Millipore Synergy UVR into the line. SVL measures and records the resistivity of the laboratory water each weekday.

## 7.0 TEST METHODS

### 7.1 Analyses Performed by SVL

SVL routinely performs the following analytical methods.

ANALYTE	METHOD	TECHNIQUE
Aluminum	EPA 200.7, SW846 6010B&C	ICP
Antimony	EPA 200.7, SW846 6010B&C	ICP
Antimony	EPA 200.8, SW846 6020&A	ICPMS

<b>ANALYTE</b>	<b>METHOD</b>	<b>TECHNIQUE</b>
Arsenic	EPA 200.7, SW846 6010B&C	ICP
Arsenic	EPA 200.8, SW846 6020&A	ICPMS
Barium	EPA 200.7, SW846 6010B&C	ICP
Barium	EPA 200.8, SW846 6020&A	ICPMS
Beryllium	EPA 200.7, SW846 6010B&C	ICP
Beryllium	EPA 200.8, SW846 6020&A	ICPMS
Boron	EPA 200.7, SW846 6010B&C	ICP
Boron	EPA 200.8, SW846 6020&A	ICPMS
Cadmium	EPA 200.7, SW846 6010B&C	ICP
Cadmium	EPA 200.8, SW846 6020&A	ICPMS
Calcium	EPA 200.7, SW846 6010B&C	ICP
Chromium	EPA 200.7, SW846 6010B&C	ICP
Chromium	EPA 200.8, SW846 6020&A	ICPMS
Chromium, Hexavalent	SM 3500 CR B&D	Colorimetry
Cobalt	EPA 200.7, SW846 6010B&C	ICP
Cobalt	EPA 200.8, SW846 6020&A	ICPMS
Copper	EPA 200.7, SW846 6010B&C	ICP
Copper	EPA 200.8, SW846 6020&A	ICPMS
Gallium	EPA 200.7, SW846 6010B&C	ICP
Gold	EPA 231.2	GFAA
Iron	EPA 200.7, SW846 6010B&C	ICP
Lanthanum	EPA 200.7, SW846 6010B&C	ICP
Lead	EPA 200.7, SW846 6010B&C	ICP
Lead	EPA 200.8, SW846 6020&A	ICPMS
Lithium	EPA 200.7, SW846 6010B&C	ICP
Magnesium	EPA 200.7, SW846 6010B&C	ICP
Manganese	EPA 200.7, SW846 6010B&C	ICP
Manganese	EPA 200.8, SW846 6020&A	ICPMS
Mercury	EPA 245.1, SW846 7470A, 7471B	CVA
Molybdenum	EPA 200.7, SW846 6010B&C	ICP
Molybdenum	EPA 200.8, SW846 6020&A	ICPMS
Nickel	EPA 200.7, SW846 6010B&C	ICP
Nickel	EPA 200.8, SW846 6020&A	ICPMS
Potassium	EPA 200.7, SW846 6010B&C	ICP
Scandium	EPA 200.7, SW846 6010B&C	ICP
Selenium	SM 3114C	Hydride AA
Selenium	EPA 200.7, SW846 6010B&C	ICP
Selenium	EPA 200.8, SW846 6020&A	ICPMS
Silica	EPA 200.7	ICP
Silicon	SW846 6010B&C	ICP
Silver	EPA 200.7, SW846 6010B&C	ICP
Silver	EPA 200.8, SW846 6020&A	ICPMS
Sodium	EPA 200.7, SW846 6010B&C	ICP
Strontium	EPA 200.7, SW846 6010B&C	ICP
Thallium	EPA 200.7, SW846 6010B&C	ICP
Thallium	EPA 200.8, SW846 6020&A	ICPMS
Tin	EPA 200.7, SW846 6010B&C	ICP
Titanium	EPA 200.7, SW846 6010B&C	ICP
Uranium	EPA 200.8	ICPMS
Vanadium	EPA 200.7, SW846 6010B&C	ICP



<b>ANALYTE</b>	<b>METHOD</b>	<b>TECHNIQUE</b>
Vanadium	EPA 200.8, SW846 6020&A	ICPMS
Zinc	EPA 200.7, SW846 6010B&C	ICP
Zinc	EPA 200.8, SW846 6020&A	ICPMS
Acidity	SM 2310 B	Automated Titration
Alkalinity	SM 2320 B	Automated Titration
Ammonia	EPA 350.1	Automated Colorimetry
Bromide	EPA 300.0	Ion Chromatography
Chemical Oxygen Demand	EPA 410.4	Colorimetry
Chloride	EPA 300.0	Ion Chromatography
Color	SM 2120 B	Colorimetry
Conductivity	EPA 120.1	Wheatstone Bridge
Corrosivity	SM 2330 B	Langelier Index
Cyanide, Total	EPA 335.4, SW 846 9012 B	Automated Colorimetry
Cyanide, Free	ASTM D-7237-10	Amperometry
Cyanide, WAD	SM 4500 CN I	Automated Colorimetry
Dissolved Organic Carbon	SM 5310 B	Combustion
Fluoride	EPA 300.0	Ion Chromatography
Hardness	SM 2340B, Ca as CaCO <sub>3</sub> by 200.7	ICP Sum
Ignitability	SW846 1010A	Pensky-Martin
Nitrate	EPA 300.0	Ion Chromatography
Nitrate + Nitrite	EPA 353.2	Automated Colorimetry
Nitrate + Nitrite	EPA 300.0	Ion Chromatography
Nitrite	EPA 300.0	Ion Chromatography
Odor	SM 2150B	Sniff Panel
ortho-Phosphate	SM 4500 P E	Colorimetry
pH (aqueous)	SM 4500-H <sup>+</sup> B	Electrometric
pH (soil)	EPA 9045C&D	Electrometric
Paste pH	EPA 600/2-78-054	Electrometric
Phosphate, Total	SM 4500 P E	Persulfate Digestion
Residue, Filterable (TDS)	SM 2540 C	Gravimetric
Residue, Non Filterable (TSS)	SM 2540 D	Gravimetric
Specific Conductance	EPA 120.1, SM 2510 B	Wheatstone Bridge
Sulfate	EPA 300.0	Ion Chromatography
Sulfide	SM 4500 S <sup>2-</sup> F	Titrimetric
Surfactants (MBAS)	SM 5540 C	Colorimetry
Total Nitrogen	D 5176-91	Combustion
Total Solids	SM 2540 B	Gravimetric
Total Kjeldahl Nitrogen	EPA 351.2	Automated Colorimetry
Total Organic Carbon	SM 5310 B	Combustion
Total Volatile Solids	EPA 160.4, SM 2540 E	Gravimetric
Turbidity	EPA 180.1	Nephelometric
TCLP (Toxicity Characteristic Leaching)	SW846 1311	Extraction
SPLP (Synthetic Precipitation Leaching)	SW846 1312	Extraction
STLC (Soluble Threshold Limit Concentration)		Extraction
MWMP (Meteoric Water Mobility)	ASTM E2242-12	Extraction

<b>ANALYTE</b>	<b>METHOD</b>	<b>TECHNIQUE</b>
CA-WET (California Waste Extraction Test)		Extraction
CEC (Cation Exchange Capacity)	SW846 9081, 9080	Exchange
Textural Analysis (Particle Size)	ASA "Methods of Soil Analysis" Number 9, Part 1	
Specific Gravity	ASA 9	Displacement
TOM/TOC	USDA, HB60(24)	Colorimetry
ANP (Acid Neutralization Potential)	EPA 600/2-78-054	Combustion
NCV (Net Carbonate Value)	EPA 600/2-78-054	Combustion
NAG (Net Acid Generation)	EPA 600/2-78-054	Combustion
ABA (Acid Base Account)	ASTM E 1915-05 & EPA 600/2-78-054	Combustion
Total Sulfur + Sulfur Forms	EPA 600/2-78-054	Combustion
Total Carbon	ASTM E 1915-05	Combustion
Textural Class	EPA 600/2-78-054	Hydrometer
Arsenic Speciation	K.S. Subramanian et al.	GFAA
Iron Speciation	HACH-8146	Colorimetry
Loss on Ignition	Soil & Plant Analysis Council	Gravimetric
Percent Silica	ASTM D-2795 and D-3682-78	Colorimetry
Tot Suspended Particulates	40 CFR 50, App B amend 12/6/82	Gravimetric
Flash Point	SW-846 1010, ASTM D93-80	Pensky-Martin

**6010B, 6020 and 7471A are maintained for those states that haven't implemented the EPA request to use the current promulgated method.**

## **7.2 References**

2009 TNI Standard.

Methods for the Determination of Metals in Environmental Samples Supplement I, EPA/600/R-94/111, May 1994.

Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993.

Field and Laboratory Methods Applicable to Overburden and Minesoils, EPA 600/2-78-054.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW 846), Third Edition, Update IV, January 2008.

Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup> Edition, 1992.

Standard Methods for the Examination of Water and Wastewater, 19<sup>th</sup> Edition, 1995.

Standard Methods for the Examination of Water and Wastewater, 20<sup>th</sup> Edition, 1999.

Standard Methods for the Examination of Water and Wastewater, 21<sup>st</sup> Edition, 2005.

Standard Methods for the Examination of Water and Wastewater, 22<sup>nd</sup> Edition, 2012

ASTM Book of Standards, part 31.

Soil Testing and Plant Analysis, 3<sup>rd</sup> Edition, Soil Sciences Society of America, 1990.

American Society of Agronomy, "Methods of Soil Analysis" Number 9, Parts 1 and 2.

U.S. Department of Agriculture, Handbook #60.

U.S. Department of the Interior, Bureau of Reclamation, Procedure for Determining Moisture, Ash, and Organic Content of Soil, USBR 5430-89.

Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition.

40 CFR Method Update Rule, April 17, 2012.

## **8.0 NEW WORK**

The Business Development group discusses new work with clients before the work is received. If the work being requested involves tests not usually performed by SVL, the project is discussed with Department Supervisors to determine if the work can be accepted. Quotes and projects are logged so that there is no confusion about what is expected by the client. If work is received that does not adhere to the guidelines put forth in the quote or project, the client will be contacted for clarification. It is SVL's responsibility to inform the client that appropriate tests and/or calibration methods have been selected that are capable of meeting the client's requirements. Occasionally SVL will receive a work order with no prior notification that requests unusual tests, or tests to be conducted in a time frame not suitable for the work requested. When this occurs, the SCO reviews the job with the Laboratory Director,

Client Services and/or Department Supervisors to determine if the work can or should be accepted. Routine work from established clients normally is not reviewed with the clients before jobs are set up, unless there is a problem with sample integrity or information on the COC.

SVL reviews and makes available in LIMS, the parameters associated with a client's project (project or work order memos shall be attached when special instructions are involved). A schedule is accessible for the work that has been received; this allows the staff to plan workloads and to track jobs. A Laboratory/Technical Director or Client Services member shall review all work orders. Adjustments to work schedules and staff deployment are made based upon the workload. Department supervisors keep equipment and supplies on hand for routine work and for many non-routine tests as well. For further detail regarding the above, see SOP SVL 1027.

## **8.1 Sample Acceptance Policy**

- 8.1.1** Samples received at SVL will be accepted for testing if the following criteria are all met at the time of sample receipt:
- 8.1.2** A proper SVL or client COC will accompany the sample shipment and must be completed in full (unless a project number is specified and is on file with SVL), including but not limited to; the client's name, address, phone number/fax number/email address, contact person, unique sample identification of individual samples, sample locations (if applicable), date and time of collection, collector's name, preservative type, sample matrix, filtered or unfiltered, number of bottles, analytes and/or tests to be performed, method of analysis, and any comments concerning sample specifics or QC requirements.
- 8.1.3** The use of correct sample containers (with proper preservation) for the sample matrices collected and ensuring that sufficient sample volume is provided for the tests requested (including extra volumes for QC requirements).
- 8.1.4** Accurate labeling of sample bottles using coded, water resistant labels and permanent ink, with said labels being cross referenced with information contained in the COC.
- 8.1.5** Adherence to holding time requirements as required by test or method requested.

**8.1.6** In the event that a sample is received in non-compliance with this policy, the sample in question will be segregated and the client notified by telephone or email. The client may direct SVL to continue on with analysis of the non-conforming sample(s). Non-conformities will be noted on the sample receipt/chain of custody and within the final report if applicable; ref, SOP SVL 2001.

**8.1.7** New clients will be informed of this policy through Client Services or Sample Receiving. They will be provided with a copy of the QM (hard copy or electronically) or a hand out on sample acceptance (located in SVL's waiting room or in Sample Receiving). As a reminder current clients/samplers will receive a copy of the sample acceptance policy if they submit samples that do not meet SVL's requirements.

## **9.0 CALIBRATION**

### **9.1 Thermometers**

Calibration of thermometers is described in SOP SVL 1004.

Quality Control Services calibrates SVL's NIST-certified thermometers.

SVL calibrates in-house liquid-in-glass thermometers against a NIST-certified thermometer annually. Digital thermometers are calibrated against a NIST certified thermometer quarterly. The IR gun is calibrated against a NIST certified thermometer quarterly. The calibrated thermometers are labeled with the appropriate correction factors.

### **9.2 Balances**

Servicing and calibrating balances is described in SOP SVL 1025.

Quality Control Services calibrates SVL's balances.

SVL checks the calibration of a balance before each day of use with at least two weights traceable to a NIST traceable standard. For analytical balances, the measured weight must agree with the certified weight within 0.1%. Balances that fail the criterion are checked with Class-1 weights. If they fail again, they are removed from service.

### **9.3 Balance Weights**

Calibration of balance weights is described in SOP SVL 1025.

Quality Control Services calibrates SVL's set of Class-1 weights, with Reference Standards Traceable to NIST.

SVL uses certified Class-1 weights to certify the Class-4 weights used for the daily calibration of balances.

### **9.4 Micropipets**

The calibration of micropipets is described in SOP SVL 1026.

SVL checks the calibration of variable-volume micropipets weekly. Fixed-volume micropipets are checked quarterly. If a measurement is out of control the mean of three measured volumes is taken and must agree with the expected value within 3%. Micropipets that fail this criterion are repaired or removed from service.

### **9.5 Repipettors**

The calibration of repipettors is described in SOP SVL 1026.

SVL checks the calibration of repipettors quarterly. The measured volume must agree with the expected value within 3%. Repipettors that fail this criterion are repaired or removed from service.

### **9.6 Refrigerators**

SVL records the temperature of sample, standard, and reagent storage refrigerators each weekday. The process is described in SOP SVL 2004. The temperature must meet the 0-6°C as described in SOP SVL 2001. If a temperature is outside this criterion, the temperature is recorded again after one hour. If the temperature is still outside the acceptance range, samples, standards, and reagents are transferred to alternate refrigerators or coolers.

### **9.7 Ovens**

SVL records the temperature of ovens every day that the oven is in use. The required temperature of each oven is stated in the applicable SOPs.

### **9.8 Inductively Coupled Plasma Mass Spectrometer (ICP-MS)**

SVL calibrates its ICP-MS in accordance with EPA methods 200.8 and 6020A. A tune standard analysis is performed prior to calibration. Five calibration standards and a calibration blank are analyzed at the beginning of a sequence. The software creates a linear calibration curve that must have a correlation coefficient of at least 0.995. Calibration

points are verified against the curve. The low calibration standard should be within  $\pm 30\%$  and the remaining calibration standards within  $\pm 10\%$  of the indicated concentration. An Initial Calibration Verification (ICV) from a secondary source follows to verify the calibration. An Initial Calibration Blank (ICB) indicates the system is clean. A Reporting Limit Check Standard (RLCS) indicates that the results derived at the reporting limit can be recovered within our acceptance criteria. Analysis of a Continuing Calibration Verification (CCV) and a Continuing Calibration Blank (CCB) follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOPs SVL 4111, 4112 and 4132.

#### **9.9 Inductively Coupled Plasma Spectrometer (ICP)**

SVL calibrates ICPs in accordance with EPA methods 200.7 and 6010C. A single calibration standard and a calibration blank are analyzed at the beginning of a sequence. Interference check standards are run to show that interelement correction factors are current. An RLCS indicates that the results derived at the reporting limit can be recovered within our acceptance criteria. An ICV from a secondary source follows to verify the calibration. An ICB indicates the system is clean. Analysis of a CCV and a CCB follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOP SVL 4102 & 4135. RLCSs are analyzed at the end of drinking water and 6010C runs.

#### **9.10 Graphite Furnace Atomic Absorption Spectrometer (GFAA)**

SVL calibrates its GFAA in accordance with EPA method 231.2 for gold and K.S. Subramanian et al. for arsenic speciation. Three calibration standards and a calibration blank are analyzed at the beginning of a sequence. Perkin-Elmer instruments create a linear calibration curve that must have a correlation coefficient of at least 0.995. Calibration points are verified against the curve, the low calibration standard should be within  $\pm 30\%$  and the remaining calibration standards within  $\pm 10\%$  of the indicated concentration. An ICV from a secondary source follows to verify the calibration. An ICB indicates the system is clean. An RLCS indicates that the results derived at the reporting limit can be recovered within our acceptance criteria. Analysis of a CCV and a CCB follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOPs SVL 4115 and 4082.

#### **9.11 Mercury Analyzer (CVAA)**

SVL calibrates its CVAA in accordance with EPA methods 245.1, 7470A, and 7471B. Six calibration standards and a calibration blank are analyzed at the beginning of a sequence. The instrument creates a linear calibration curve that must have a correlation coefficient of at least 0.995. Calibration points will be verified against the curve (see SVL 1020). The low calibration standard should be within  $\pm 30\%$  and the remaining calibration standards within  $\pm 10\%$  of the indicated concentration. An ICV from a secondary source follows to verify the calibration. An ICB indicates the system is clean. An RLCS indicates that the results derived at the reporting limit can be recovered within our acceptance criteria. Analysis of a CCV and a CCB follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOP SVL 4010.

#### **9.12 Flame Atomic Absorption Spectrometer (FLAA)**

SVL calibrates FLAAs in accordance with analytical method requirements. The acceptance criteria are defined in SOP SVL 4105.

#### **9.13 Ion Chromatograph (IC)**

SVL calibrates ICs in accordance with EPA method 300.0. Five calibration standards and a calibration blank are analyzed. The instrument creates a quadratic calibration curve that must have a correlation coefficient of at least 0.995. Calibration points will be verified against the curve (see SVL 1020). The low calibration standard should be within  $\pm 30\%$  and the remaining calibration standards within  $\pm 10\%$  of the indicated concentration. An ICV from a secondary source follows to verify the calibration. An ICB indicates the system is clean. An RLCS indicates that the results derived at the reporting limit can be recovered within our acceptance criteria. A CCV and a CCB follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOPs SVL 4122 and 4133.

#### **9.14 Flow-Injection Auto Analyzer (FIA)**

SVL calibrates FIAs in accordance with EPA methods 335.4 (Total Cyanide), 350.1 (Ammonia), 351.2 TKN, 353.2 (Nitrate and Nitrite), 9012 B (Total Cyanide), and Standard Methods 4500-CN-I (WAD Cyanide), and ASTM D-7237-10 (Amperometric Free Cyanide). A minimum of five calibration standards and a calibration blank are analyzed at the beginning of each analytical sequence. The instrument software creates a linear or quadratic calibration curve that must have a correlation coefficient of at least 0.995. Calibration points will be



verified against the curve (see SVL 1020). The low calibration standard should be within  $\pm 30\%$  and the remaining calibration standards within  $\pm 10\%$  of the indicated concentration. An LCS and ICV from a secondary source verifies the calibration curve. An ICB indicates the system is clean. An RLCS indicates that the results derived at the reporting limit can be recovered within our acceptance criteria. Analysis of a CCV and a CCB follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOPs SVL 4012, SVL 4045, SVL 4099, SVL 4048, SVL 4075, and SVL 4131.

#### **9.15 Total Organic Carbon Analyzer (TOC)**

SVL calibrates TOC analyzers in accordance with SM 5310 B. Six calibration standards for total carbon are analyzed and a linear curve is constructed, the curve must have a correlation coefficient of at least 0.995. Calibration points will be verified against the curve (see SVL 1020). The low calibration standard should be within  $\pm 30\%$  and the remaining calibration standards within  $\pm 10\%$  of the indicated concentration. An ICV from a secondary source verifies the calibration curve. An ICB indicates the system is clean. An RLCS indicates that the results derived at the reporting limit can be recovered within our acceptance criteria. A CCV and CCB are analyzed at the beginning of each analytical sequence, after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOP SVL 4116.

#### **9.16 UV/Visible Spectrophotometers (UV/VIS)**

SVL calibrates its UV/Visible spectrophotometer in accordance with the applicable published methods. A minimum of three calibration standards and a calibration blank are analyzed at the beginning of each analytical sequence. The calibration curve must have a correlation coefficient of at least 0.995. Calibration points will be verified against the curve (see SVL 1020). The low calibration standard should be within  $\pm 30\%$  and the remaining calibration standards within  $\pm 10\%$  of the indicated concentration. An ICV from a secondary source follows to verify the calibration. An ICB indicates the system is clean. A CCV and CCB are analyzed at the beginning of each analytical sequence, after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOPs 4037, 4040, 4042, 4043, 4044, 4123 and 4125.

### **9.17 LECO Carbon/Sulfur Analyzer**

ABA, Total Sulfur, and Total Carbon are determined from analysis of a small aliquot of crushed sample using a LECO furnace. In addition, organic and inorganic carbon and pyrolysis loss and residual sulfur may be determined by roasting a sample, analyzing it by LECO, and calculating the difference between the pre and post roast carbon and sulfur values. Three sets of three calibration standards for carbon and sulfur are analyzed to prepare a calibration curve that must have a correlation coefficient of at least 0.995. Calibration points will be verified against the curve (see SVL 1020). The low calibration standard should be within  $\pm 30\%$  and the remaining calibration standards within  $\pm 10\%$  of the indicated concentration. An ICV from a secondary source follows to verify the calibration. An ICB indicates the system is clean. An RLCS indicates that the results derived at the reporting limit can be recovered within our acceptance criteria. A CCV and CCB are analyzed at the beginning of each analytical sequence, after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOPs SVL 4097, 4061 and 4129.

### **9.18 pH and Ion Selective Electrode Meters (ISE)**

SVL calibrates pH and ISE meters in accordance with the applicable published methods.

### **9.19 Class A Glassware**

Class A glassware is verified, assigned a unique identifier and logged in upon receipt as described in SOP SVL 1026.

## **10.0 SAMPLING, SAMPLE RECEIVING, AND STORAGE**

### **10.1 Sampling**

SVL does not conduct sampling. Sampling procedures that lead to contamination of client's samples in the field are beyond SVL's control.

Sample preservation is critical for sample integrity. Chemical and biological reactions may occur that begin to change some chemical species upon sample collection. Unfortunately, for most samples, immediate analysis is neither economically feasible nor logistically possible. Although no chemical preservative exists that is valid for every parameter, SVL strongly recommends the preservation methods, container type, sample size and estimated maximum holding times for

collection of water and wastewater samples summarized in Table 1. Solid samples are best preserved by cooling the sample to a range between 0- 6°C.

**Table 1**

<b>Analysis</b>	<b>Volume Required (mL)</b>	<b>Container</b>	<b>Preservative</b>	<b>Holding Time</b>
Color	50	P,G	Cool to ≤ 6 °C	48 Hours
Conductance	100	P,G	Cool to ≤ 6°C	28 Days
Hardness	100	P,G	HNO <sub>3</sub> to pH<2	6 Months
Odor	300	G only	Cool to ≤ 6°C	24 Hours
pH	25	P,G	None Required	* ASAP
Temperature	1000	P,G	None Required	* ASAP
Turbidity	100	P,G	Cool to ≤ 6 °C	48 Hours
Filterable Residue (TDS)	100	P,G	Cool to ≤ 6 °C	7 Days
Non-Filterable Residue (TSS)	100	P,G	Cool to ≤ 6 °C	7 Days
Total Residue	100	P,G	Cool to ≤ 6 °C	7 Days
Volatile Residue	100	P,G	Cool to ≤ 6 °C	7 Days
Settleable Matter	1000	P,G	Cool to ≤ 6 °C	48 Hours
Dissolved Metals	200	P,G	Filter on site; HNO <sub>3</sub> to pH<2	6 Months
Total Metals	100	P,G	HNO <sub>3</sub> to pH<2	6 Months
Chromium (VI)	200	P,G	Cool to ≤ 6 °C	24 Hours/ 28 days**
Mercury, Dissolved	100	P,G	Filter; HNO <sub>3</sub> to pH<2	28 Days
Mercury, Total	100	P,G	HNO <sub>3</sub> to pH<2	28 Days
Acidity	100	P,G	Cool to ≤ 6 °C	14 Days
Alkalinity	100	P,G	Cool to ≤ 6 °C	14 Days
Bromide	100	P,G	None Required	28 Days
Chloride	50	P,G	None Required	28 Days
Cyanide	500	P,G	Cool to ≤ 6 °C; NaOH to pH>12	14 Days
Fluoride	300	P	None Required	28 Days
Ammonia	400	P,G	Cool to ≤ 6 °C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 Days
Total Kjeldahl Nitrogen	500	P,G	Cool to ≤ 6 °C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 Days
Nitrate plus Nitrite	100	P,G	Cool to ≤ 6 °C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 Days
Nitrate	100	P,G	Cool to ≤ 6 °C	48 Hours
Nitrite	50	P,G	Cool to ≤ 6 °C	48 Hours

<b>Analysis</b>	<b>Volume Required (mL)</b>	<b>Container</b>	<b>Preservative</b>	<b>Holding Time</b>
Ortho-Phosphate Dissolved	50	P,G	Filter on site; Cool to ≤ 6 °C	48 Hours
Total Phosphate	50	P,G	Cool to ≤ 6 °C; H <sub>2</sub> SO <sub>4</sub> to pH<2	28 Days
Total Dissolved Phosphate	50	P,G	Filter on site; Cool to ≤ 6 °C; H <sub>2</sub> SO <sub>4</sub> to pH<2	28 Days
Silica	50	P only	Cool to ≤ 6 °C	28 Days
Sulfate	50	P,G	Cool to ≤ 6 °C	28 Days
Sulfide	500	P,G	Cool to ≤ 6 °C add 2 mL zinc acetate plus NaOH to pH>9	7 Days
COD	50	P,G	Cool to ≤ 6 °C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 Days
Total Organic Carbon	25	40 mL amber vials	Cool to ≤ 6 °C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 Days
Phenolics	500	G only	Cool to ≤ 6 °C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 Days
MBAS	1200	P,G	Cool to ≤ 6 °C	48 Hours

\* pH and temperature should be measured in the field whenever possible. They are subject to rapid change. Measurements of pH and temperature made in the laboratory will almost always be out of holding time.

\*\* If preserved in the following manner add 0.45 mL buffer solution to each vial. Adjust the pH to 9.3 – 9.7 using about 2 drops of 10 N sodium hydroxide and about 3-5 drops of 1N sodium hydroxide.

SVL has formed alliances with other laboratories for the analysis of organic parameters. The recommended containers and preservatives are

<b>Analysis</b>	<b>Amount Required</b>	<b>Container</b>	<b>Preservative</b>	<b>Holding Time Until Extraction</b>	<b>Holding Time After Extraction Until Analysis</b>
Mercury, Low Level***					
524.2 (Volatile Organic Compounds)	3x40mL vials	G,T	Cool to ≤ 6 °C; HCl to pH<2	14 days	NA
608 (Pesticides and/or PCBs)	3 L	amber G,T	Cool to ≤ 6 °C	7 days	40 days
624 (Volatile Organic Compounds)	3x40mL vials	G,T	Cool to ≤ 6 °C; HCl to pH<2	14 days	NA
625 (Semi-volatile Organic Compounds)	3 L	amber G,T	Cool to ≤ 6 °C	7 days	40 days

Analysis	Amount Required	Container	Preservative	Holding Time Until Extraction	Holding Time After Extraction Until Analysis
Mercury, Low Level***					
1664 Hexane Extractable Materials	2L	G only	Cool to $\leq 6$ °C H <sub>2</sub> SO <sub>4</sub> or HCl to pH<2	28 days	NA
8081A (Pesticides)	8 oz (soil) 1L (aqueous)	amber G,T	Cool to $\leq 6$ °C	14 days 7 days	40 days
8082 (PCBs)	8 oz (soil) 1 L (aqueous)	G,T	Cool to $\leq 6$ °C	14 days 7 days	40 days
8260B (Volatile Organic Compounds)	4 oz (soil) 3x40mL (aq)	G,T	Cool to $\leq 6$ °C; HCl to pH<2	14 days	NA
8270C (Semi-volatile Organic Compounds)	8 oz (soil) 1 L (aqueous)	amber G,T	Cool to $\leq 6$ °C	14 days	40 days
8015 (TPH-Gasoline)	4 oz (soil) 3x40 mL (aq)	amber G,T	Cool to $\leq 6$ °C; HCl to pH<2	14 days	35 days
8015AZ ****	8 oz (soil)	G,T	Cool to $\leq 6$ °C	48 hours	14 days for extraction and analysis
8260BAZ****	4 oz (soil)	G,T	Cool to $\leq 6$ °C	48 hours	NA
8015 (TPH-Diesel Motor Oil)	1 L (aq) 8 oz (soil)	amber G,T	Cool to $\leq 6$ °C; HCl to pH<2	14 days	40 days

\*\*\* Call for sampling and hold time requirements.

\*\*\*\* TPH 8015AZ and 8260AZ (soils) have a 48 hour hold time before extraction.

## 10.1 Sampling Cont'd

Field blanks allow for identification of systemic and random sample contamination that may result from the sampling equipment, storage containers, sampling agents, or chemicals added to preserve samples. Field blanks consist of a sample container of distilled or deionized water with the appropriate chemical preservative. Preservation, filtration, storage, handling, and analysis are performed as if the field blanks were samples. To achieve accurate and meaningful data, field blank containers should be filled with analyte-free water and the appropriate preservative at the sampling site.

Sources of sample contamination include unclean sample containers and filters; impure solvents and reagents; and use of cleaning products inappropriate for the proposed analysis. Hair, tobacco smoke, and dust also are appreciable sources of contamination, so sampling should be conducted in as careful a manner as possible.

Before filtering samples for dissolved parameters, the filter paper should be rinsed with de-ionized or distilled water and with a small portion of sample. The filtration apparatus should also be rinsed with de-ionized or distilled water between samples. Handle filter paper only on the edge, using appropriate forceps (plastic for trace metals analysis).

Use the proper sample container for the parameter specified. Samples for trace metals analysis must not come into contact with any metallic surface; samples for organic analysis must not come into contact with any plastic surface.

Sampling personnel should complete a COC form that documents sampler, sample identification, sampling date and time, sample location (state of sample origin if applicable), matrix type, number of sample containers, type of preservation, whether samples have been filtered, and the parameters to be analyzed.

### **10.1.1 Sub-sampling**

In the event that SVL must undertake sub-sampling, SVL will use the appropriate container (uniquely identified) and the proper preservation. If SVL undertakes the sub-sampling of matrices that are required to be performed in the field, SVL will identify those samples on the analytical report; ref, SOP SVL 2018.

## **10.2 Sample Receiving and Storage**

SOPs SVL 2001, SVL 2003, and SVL 2004 describe sample receiving, job creation, and sample storage, respectively.

SVL takes a temperature reading from the sample shipping containers (coolers) upon receipt and opening. Each sample is checked for visible damage and the presence of an intact custody seal (if required). SVL gives each group of samples a unique job number (e.g., " W1E0027"). Sample ID's are automatically assigned a serial number suffix (-01 thru - 99) appended to the work order number they belong to. The work order number is auto-generated by the LIMS and follows the format W YAnnnn, where Y is the last digit of the year, A corresponds to the month in which the work order was created (A=Jan., B=Feb....L=Dec), and nnnn is a serial number for the work order in a particular month. Individual sample containers are assigned a designator (A, B, C ...) and these are tracked in the LIMS so the particular container used for an analysis can be tracked. For an example "W1E0027-03 D" would be the fourth container for the third sample in the 27th work order of May 2011.

Job numbers remain with the samples throughout the analytical process. Each sample is assigned a unique, sequential identification number. Samples are labeled with a bar code (containing both the sample and job numbers) before storing the sample.

Samples that require refrigeration are stored in walk-in coolers (which are kept between 0°C and 6°C), except during times of sample preparation or analysis. Samples that do not require refrigeration are stored in an ambient temperature storage room. The laboratory does not refrigerate soil samples that were not received on ice. Samples are retained by SVL for a minimum of 30 days (or longer if required by the client) after an analytical report has been issued to the client. At the end of the specified period, samples are returned to the client or discarded in an appropriate manner.

Sample custodians, technicians and analysts use the custody log feature of the LIMS to track sample movement during receipt, preparation, and disposal. SVL personnel are responsible for logging the samples into their custody, where they assume accountability for the sample(s). When use of the sample is complete, personnel must scan samples back into the appropriate home location or another employee may assume custody by scanning/logging the sample into their custody via the LIMS.

### **10.3 Sample Disposal and Hazardous Waste**

Procedures for sample disposal are described in SOP SVL 1001. Disposal procedures follow federal and state regulatory requirements. SVL's hazardous waste program is described in SOP SVL 1008.

## **11.0 EQUIPMENT AND INSTRUMENTS**

SVL uses the following instruments to generate analytical data and to calibrate other instruments.

**11.1** SVL performs instrument maintenance as recommended by the manufacturer. SVL maintains service contracts with vendors for its major analytical instrumentation. Maintenance logbooks are kept to provide a record of major and minor repairs; as well as, preventative maintenance.

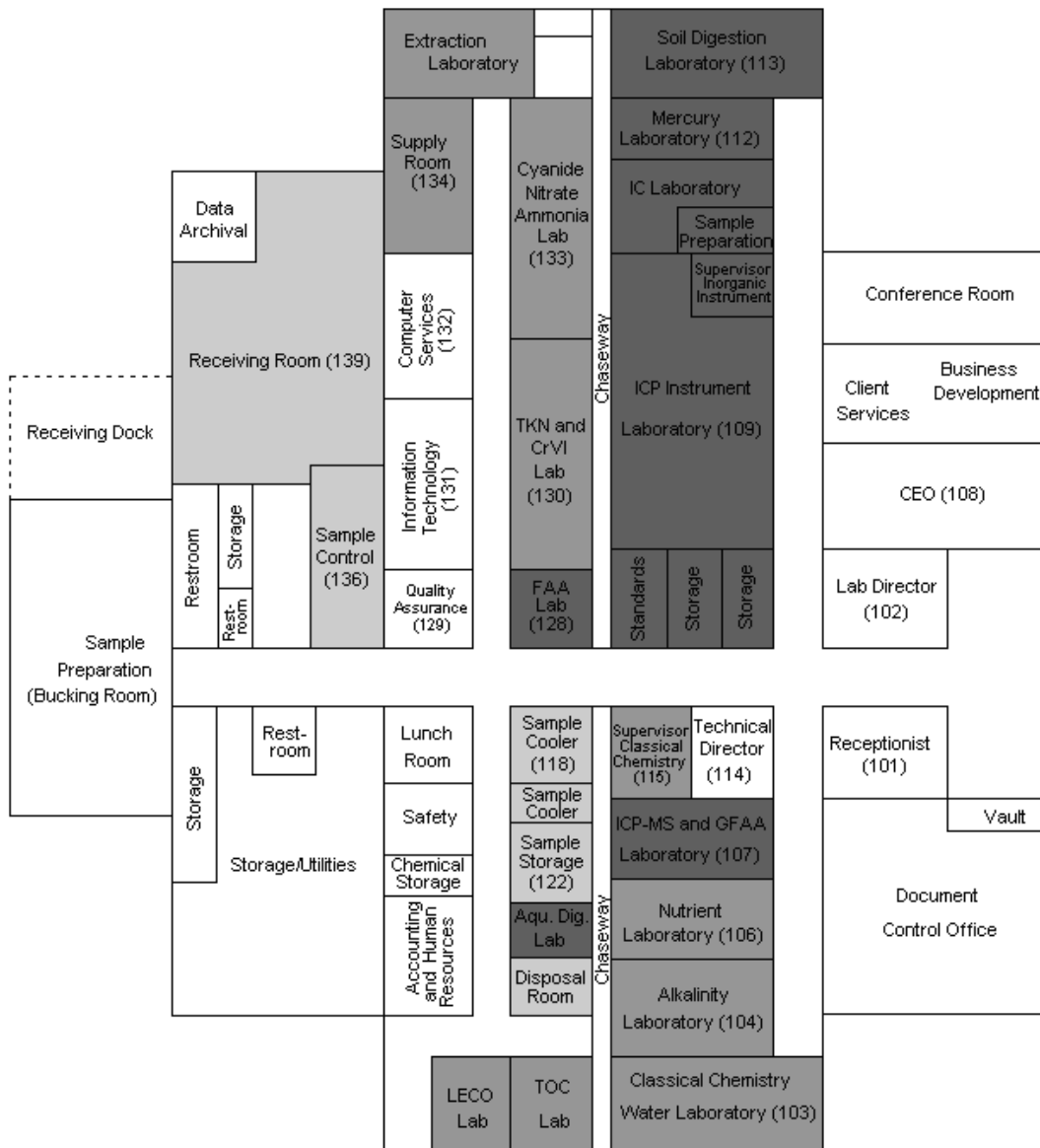
**11.2** The analysts and supervisors will determine if a repair has created a need to update instrument MDLs, linear ranges, calibrations etc.

<b>INSTRUMENT</b>	<b>MANUFACTURER</b>	<b>MODEL</b>	<b>SERIAL NUMBER</b>
Spectrometer (ICP-MS)	Perkin-Elmer	ELAN 5000	W0660402
Spectrometer (ICP-MS)	Agilent	7700 Series	JP10490758
Spectrometer (ICP) Thermo 1	Thermo Electron	iCAP 6500 Duo	IC5D20130703
Spectrometer (ICP) Thermo 2	Thermo Electron	iCAP 6500 Duo	IC65DC133703
Spectrometer (ICP) Thermo 3	Thermo Electron	iCAP 7400 Duo	IC74DC141807
Spectrometer (ICP) Optima A	Perkin-Elmer	Optima 8300	078N2080202
Atomic Absorption Spectrometer with Graphite Furnace	Perkin-Elmer	Analyst 600	601S3090501
Atomic Absorption Spectrometer with Vapor Generation Assembly	Varian	AA 55B	EL03048142
Mercury Analyzer with Autosampler	CETAC	M-6100	021202QT6
Mercury Analyzer with Autosampler	CETAC	M-7500	110801QTA
11 Digestor Blocks	Environmental Express	Hot Block	
Ion Chromatograph	Dionex	ICS900	08041118
Ion Chromatograph	Dionex	ICS90	04090417
Ion Chromatograph	Dionex	ICS900-C	09040981
Ion Chromatograph	Dionex	ICS-1100	12120925
Automated Flow Analyzer	O-I-Analytical	FS3100-2	Multi-component
Automated Flow Analyzer	O-I-Analytical	FS3100	Multi-component
2 Micro Distillation Units	Lachat	ID 001	A2000-828 and 081100001017
3 MIDI Distillation Units	BSL		
2 Ammonia Distillation Units	Andrews Glass		
Ammonia/N analyzer	Astoria Pacific	A2	200104
Automated Flow Analyzer	Alpkem	Alpkem TKN	200220
Block Digestor	Westco Scientific	Easy Digest 40/20	As. # INS0030HW
Auto Titrator with Autosampler 4	Metrohm	Titrimo 809 Titrimo	1809001007108
Auto Titrator with Autosampler 5	Metrohm	Titrimo 809 Titrimo	1809001013143
UV/Visible Spectrophotometer A	Genesys	20	3SGN243026
UV/Visible Spectrophotometer B	Genesys	20	3SGN341012
UV/Visible Spectrophotometer	Genesys	10	2D5G261004
Turbidimeter A	Hach	2100N	95041453
Turbidimeter B	Hach	2100N	080906024269
COD Reactor	VELP Scientifica	ECO 25	101448
COD Reactor	VELP Scientifica	ECO 25	171440
pH Meter	Accumet	AB15	AB92325857
pH Meter	Accumet	AB15	AB92326969
pH Meter	Beckman	11 pH Meter	0224055
pH Meter B	Thermo	Orion A III	J06383
pH Meter	Thermo	Orion 320	019525
pH Meter C	Thermo	Orion A III	J06171
Dissecting Microscope	Nikon	104	



<b>INSTRUMENT</b>	<b>MANUFACTURER</b>	<b>MODEL</b>	<b>SERIAL NUMBER</b>
Polarizing Microscope	Nikon	106	
Centrifuge	Beckman	GS-6 Centrifuge	
Centrifuge	MISTRAL	3000i	51149
Centrifuge	IEC	K	70652271
Flashpoint detector	Precision Scientific	74537	108A-2
Conductance Meter	Fisher	AB30	AB 92329154
Conductance Meter	Fisher	AB30	AB 92338713
Conductance Meter	Orion	115	002176
Elemental Analyzer B	LECO	SC632	3208
Elemental Analyzer A	LECO	SC632	3526
Carbon/Nitrogen Analyzer (TOC)	Shimadzu	TOC-VCSH-N	H51104135009 C5
Carbon/Nitrogen Analyzer (TOC)	Shimadzu	TOC-LCSN/TNM-L	H54105000234
Semi-Micro Balance	Mettler	AE-240	K89952
Semi-Micro Balance	Mettler	AE-240	G43270
Analytical Balance	Mettler	PJ 360	F89531
Analytical Balance	Mettler	PJ 360	G49684
Analytical Balance	Mettler	PB30	A04506
Analytical Balance	Mettler	PJ360	F89533
Analytical Balance	Ohaus	N1D110	1122352966
Analytical Balance	Ohaus	RD60LS	3374276-7HQ
Analytical Balance	Ohaus	EOF110	F2221120252601
Analytical Balance	Ohaus	AR2140	1203121033
Analytical Balance	Ohaus	AR1530	1203200181P
Analytical Balance	Ohaus	AS 313	8028301193
Analytical Balance	Ohaus	AV-114	8029081142
Analytical Balance	Leco	050	329
Analytical Balance	Sartorius	CPA1245	26250271
IR Thermometer	VWR	1832 Degree	101839552
IR Thermometer	Raytek	Ranger ST	98660090
Thermometer	HBI	145°C to 205°C	4B1321
Thermometer	Thermco	145°C to 205°C	3268
Thermometer	Ertco	-20°C to 110°C	5283
Thermometer	HB	-20° C to 150°C	L94280
Thermometer	HB	-1° C to 201°C	3846

# 12.0 FACILITIES



- Inorganic Instrument Department
- Sample Control
- Classical Chemistry Department
- Administrative, Accounting, QA, Computer, Documents and Other

**12.1** SVL is an analytical laboratory specializing in the performance of tests and methods used in the characterization of environmental and mining samples. Since 1972, SVL has analyzed water, soil, sediment, sludge, oil, paint, rock, animal tissue, vegetation, air filters, and various other sample types. SVL occupies a 25,000 square foot laboratory facility architecturally designed and specifically organized to ensure efficient operation and meet the needs of a large capacity analytical laboratory. Building access, security and safety features have been carefully considered. Access through the outside laboratory entrance and to internal areas is limited to laboratory staff and other essential personnel. Visitors are logged in/out and made aware of safety protocols during their stay at SVL.

### **13.0 STANDARD OPERATING PROCEDURES**

SVL performs work in accordance with the requirements of its SOPs. SVL's SOPs are listed below and describe all aspects of its work performance including Safety and Quality Assurance (1000 Series), Sample and Document Management (2000 Series) and Inorganic Analysis (4000 Series).

<b>SOP NUMBER</b>	<b>DESCRIPTION</b>
SVL 1001	SAMPLE DISPOSAL
SVL 1002	WRITING AND REVISING STANDARD OPERATING PROCEDURES
SVL 1004	CALIBRATING THERMOMETERS
SVL 1005	INTERNAL QUALITY ASSURANCE AUDITS
SVL 1007	SOIL STERILIZATION
SVL 1008	DISPOSAL OF HAZARDOUS WASTE
SVL 1010	TRAINING
SVL 1011	PERFORMING AN MDL STUDY
SVL 1015	PROCUREMENT, RECEIVING, AND SUBCONTRACTING
SVL 1017	RECORDS RETENTION AND PROTECTION
SVL 1019	CORRECTIVE ACTION
SVL 1020	CALIBRATION FOR ANALYTICAL METHODS
SVL 1021	MANUAL INTEGRATION
SVL 1023	SOFTWARE VERIFICATION
SVL 1025	CALIBRATING BALANCES
SVL 1026	CALIBRATING MICROPIPETS, REPIPETTORS, AND GLASSWARE
SVL 1027	CLIENT SERVICES
SVL 1028	CALCULATIONS FOR ANALYTICAL METHODS
SVL 1029	PERFORMANCE TESTING SAMPLES
SVL 1030	INITIAL, PERIODIC AND AFTER-MAINTENANCE CHECKS

<b>SOP NUMBER</b>	<b>DESCRIPTION</b>
SVL 1031	COMPUTER AND INFORMATION SECURITY POLICY
SVL 1032	CHEMICAL REAGENTS, PREPARED STANDARDS, AND QC SOLUTIONS
SVL 1033	ACCEPTANCE LIMITS AND TRENDING
SVL 2001	SAMPLE RECEIVING
SVL 2003	SVL JOB CREATION
SVL 2004	SAMPLE STORAGE AND SECURITY
SVL 2006	DATA CORRECTIONS
SVL 2007	CASE FILE ASSEMBLY
SVL 2009	DATA REVIEW
SVL 2013	DATA PACKAGE PRODUCTION
SVL 2015	LEVEL 3 – CLP DATA PACKAGE
SVL 2017	LOGBOOK CONTROL
SVL 2018	PREPARATION AND SUBSAMPLING OF EARTH, ROCK, AND TISSUE SAMPLES
SVL 2019	REANALYSIS PROCEDURES
SVL 2020	COMPUTER-RESIDENT SAMPLE DATA CONTROL
SVL 2021	DATA BACKUP AND RESTORE
SVL 2022	SAMPLE RECEIVING – FOREIGN SOILS
SVL 4010	EPA 245.1, SW-846 7470A and 7471A; DETERMINATION OF MERCURY (CVAA)
SVL 4012	EPA 335.4, SM 4500 CN E and SW-846 9012B; TOTAL CYANIDE BY MICRODIST™ and MIDI DISTILLATION FOLLOWED BY AUTOMATED COLORIMETRY
SVL 4013	GLASSWARE WASHING FOR CLASSICAL CHEMISTRY
SVL 4021	FILTER DIGESTION
SVL 4022	PERCENT SOLIDS/ PERCENT MOISTURE
SVL 4024	SM 2120 B; COLOR
SVL 4025	EPA 120.1 and SM 2510 B; CONDUCTIVITY
SVL 4026	EPA 180.1; TURBIDITY
SVL 4028	SM 4500 H <sup>+</sup> B; pH
SVL 4029	SPECIFIC GRAVITY
SVL 4031	SM 2310 B; ACIDITY
SVL 4032	SM 4500 S <sup>2-</sup> F; SULFIDES BY TITRATION
SVL 4034	SM 2540 C and SM 2540 D; TOTAL DISSOLVED SOLIDS AND SUSPENDED SOLIDS
SVL 4035	SM 2540 B and EPA 160.4; TOTAL AND VOLATILE SOLIDS
SVL 4037	SM 5540 C; METHYLENE BLUE ACTIVE SUBSTANCES
SVL 4040	SM 4500 P E; TOTAL PHOSPHORUS (AQUEOUS SAMPLES)
SVL 4042	SM 4500 P E; ORTHO-PHOSPHATE (AS P)
SVL 4043	EPA 410.4; CHEMICAL OXYGEN DEMAND
SVL 4044	TOTAL ORGANIC MATTER
SVL 4045	EPA 351.2; TOTAL KJELDAHL NITROGEN
SVL 4048	EPA 353.2; NITRATE/NITRITE AS N: AUTOMATED CADMIUM RE REDUCTION
SVL 4049	SW-846 9081; CATION EXCHANGE CAPACITY

<b>SOP NUMBER</b>	<b>DESCRIPTION</b>
SVL 4060	LOSS ON IGNITION (SVL METHOD)
SVL 4061	DETERMINATION OF ACID GENERATION POTENTIAL (AGP), ACID NEUTRALIZATION POTENTIAL (ANP), AND ACID-BASE ACCOUNT (ABA)
SVL 4065	METEORIC WATER MOBILITY EXTRACTION
SVL 4068	SW-846 1312; SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP)
SVL 4075	SM 4500 CN I; WAD CYANIDE BY MIDI DISTILLATION FOLLOWED BY SEMI-AUTOMATED COLORIMETRY
SVL 4078	EPA METHOD 3020A; SAMPLE DIGESTION FOR TOTAL METALS IN AQUEOUS SAMPLES FOR ICP-MS
SVL 4079	EPA METHOD 3010A; SAMPLE DIGESTION FOR TOTAL METALS IN AQUEOUS SAMPLES FOR ICP
SVL 4080	EPA METHOD 3005A; SAMPLE DIGESTION FOR TOTAL RECOVERABLE METALS IN AQUEOUS SAMPLES FOR ICP
SVL 4082	ARSENIC SPECIATION As(III) AND As(V)
SVL 4084	SM 2320 B; DETERMINATION OF ALKALINITY AND pH USING THE AUTOTITRATOR
SVL 4093	CASSETTE FILTER DIGESTION
SVL 4094	EPA METHOD 3050B; SAMPLE DIGESTION FOR METALS IN SOILS
SVL 4095	SW-846 1010; FLASHPOINT DETERMINATION (PENSKY-MARTENS CLOSED TESTER)
SVL 4096	SW-846 9045 C and 90045 D; pH DETERMINATION FOR SOILS
SVL 4097	ASTM 1915-05; TOTAL SULFUR, TOTAL CARBON
SVL 4099	EPA 350.1; AMMONIA BY SEMI-AUTOMATED COLORIMETRY
SVL 4102	EPA 200.7 and SW-846 6010C; ANALYSIS OF METALS BY METHODS 6010C AND 200.7 USING THE PERKIN-ELM OPTIMA ICP
SVL 4105	SM 3114 B; SELENIUM BY HYDRIDE
SVL 4106	METHOD 200.2; SAMPLE DIGESTION FOR TOTAL RECOVERABLE METALS IN AQUEOUS SAMPLES BY ICP AND ICP-MS
SVL 4108	SAMPLE PREPARATION FOR DISSOLVED AND POTENTIALLY DISSOLVED METALS IN AQUEOUS SAMPLES
SVL 4111	EPA METHOD 200.8; ANALYSIS OF METALS BY ICP-MS
SVL 4112	SW-846 6020A; ANALYSIS OF METALS BY ICP-MS
SVL 4114	SW-846 1311; TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)
SVL 4116	SM 5310 B; TOTAL ORGANIC CARBON
SVL 4118	CALIFORNIA WASTE EXTRACTION TEST (CA-WET)
SVL 4119	PREPARATION OF QC SOLUTIONS FOR METALS ANALYSIS
SVL 4120	ASTM D-5176; TOTAL NITROGEN
SVL 4121	SM 2150 B; DETERMINATION OF THRESHOLD ODOR NUMBER (TON)
SVL 4122	EPA 300.0; INORGANIC ANIONS BY ION CHROMATOGRAPHY USING THE DIONEX DX100 AND ICS-90
SVL 4123	ASTM D-2795 and D-3682-78 SOLID SILICA
SVL 4124	EPA 231.2; OPERATION OF PERKIN/ELMER GFAA: ANALYSIS OF GOLD BY GRAPHITE FURNACE
SVL 4125	SM 3500 Cr B; HEXAVALENT CHROMIUM
SVL 4127	pH DETERMINATION FOR PASTE
SVL 4128	SOIL ELECTRICAL CONDUCTIVITY BY ASA-9

SOP NUMBER	DESCRIPTION
SVL 4129	NET CARBONATE VALUE (NCV)
SVL 4130	NET ACID GENERATION (NAG)
SVL 4132	ANALYSIS OF METALS BY THE AGILENT ICP-MS (EPA METHOD 200.8)
SVL 4133	DETERMINATION OF THIOCYANATE BY ION CHROMATOGRAPHY USING DIONEX ICS-90 AND ICS-900
SVL 4134	ANALYSIS OF METALS BY AGILENT ICP-MS (SW-846 METHOD 6020A)
SVL 4135	ANALYSIS OF METALS BY METHODS 6010C AND 200.7 USING THE THERMO iCAP 6000 SERIES ICP SPECTROMETER
SVL 4136	TEXTURAL CLASS BY EPA-600/2-78-054
SVL 4137	EXTRACTIONS COMPENDIUM
SVL 4138	ASTM D-7275 – RECOVERY of AQUEOUS CYANIDES by EXTRACTION from MINE ROCK and SOIL after REMEDIATION of PROCESS RELEASES

### 13.1 Deviations

Occasionally, a deviation from an SOP is required to generate an accurate result for a given test or client. This may occur when a client specifically requires a modification, or when the sample matrix interferes with the analysis. The Laboratory Director or a Department Supervisor may authorize a deviation. The analyst documents details of the deviation from the SOP on the instrument raw data printout or the job bench sheet with a notation in the work order memo in Element. The deviation will be indicated on the report.

**13.1.1** In the event that an SOP needs to be immediately amended an email will be sent to the Quality Manager outlining the necessary change. The change can go into effect immediately prior to the SOP being amended.

## 14.0 QUALITY CONTROL

### 14.1 Quality Control Parameters

SVL uses a number of quality control parameters to validate calibration, and to measure contamination, accuracy, and precision. Each SVL SOP indicates the parameters required for the method being used.

#### 14.1.1 Blanks

**Method Blank** Is an aliquot of analyte-free water that is put through all the steps of a specific method along with the samples. It is sometimes called a Laboratory Reagent Blank.

**Calibration Blank** The zero-concentration standard analyzed as part of a calibration curve.

**Field Blank** Randomly selected sample container that is filled with analyte-free water and the appropriate chemical preservative in the field.

**Trip Blank** Is a specific type of field blank. A trip blank is not opened in the field. It is a check on sample contamination from the time the container is sealed at the lab or supplier. It is used to verify the container's integrity during sample transport and the container's time on site (it should always be with sampling group).

The acceptance criterion for a blank may be set by the published method, by client DQOs, or by historical statistics. In the absence of these directives, the acceptance criterion may default to less than the reporting limit.

#### 14.1.2 Matrix Spike

Is an aliquot of sample to which a known amount of analyte has been added prior to sample preparation or digestion. It is a measure of the effect of the sample matrix on the analytical method. It is sometimes called the "Laboratory Fortified Matrix".

The recovery is calculated by:

$$\% \text{ Recovery} = 100 \times (MS - S) / SA$$

Where the MS = Spiked Sample Result

S = Sample Result

SA = Spike Added

Acceptance criteria for the matrix spike recovery may be determined by the published method, by client DQOs, or set between 70-80% to 120-130%. For those methods without guidelines the QA Manager will set default limits for the acceptance range. Individual SOPs will have the recovery range acceptance requirements. There are no requirements if the concentration of the analyte in the original sample is greater than five times the concentration of the spike.

### **14.1.3 Analytical Spike or Post-Digestion Spike**

Is an aliquot of sample to which a known amount of analyte has been added after sample preparation. It is a measure of the effect of the matrix on a digestate or extract.

### **14.1.4 Laboratory Control Sample (LCS)**

Is a solution or material of known concentration that is added to an analyte-free matrix and then analyzed to evaluate the recovery and accuracy of a method. It is sometimes called a Laboratory Fortified Blank.

Acceptance criteria for the LCS recovery may be determined by the published method, by the manufacturer of the standard, by client DQOs or the QA Manager will set default limits.

### **14.1.5 Sample Duplicate**

A second similar aliquot of a sample treated exactly the same through preparation and analysis. The Relative Percent Difference (RPD) between the values of the duplicates is a measure of the precision of the analytical method.

$$\text{RPD} = 100 \times | S - D | / [(S + D)/2]$$

The acceptance criterion for the RPD is usually set at 20.

### **14.1.6 Matrix Spike Duplicate (MSD)**

A second similar aliquot that is spiked, it is treated exactly the same as the first matrix spike (MS) through preparation and analysis. The RPD between the recovery values is a measure of the precision of the analytical method.

$$\text{RPD} = 100 \times | \text{MSD} - \text{MS} | / [(\text{MSD} + \text{MS}) / 2]$$

### **14.1.7 Interference Check Sample (ICS)**

A sample with known concentrations of elements used to determine if the inter-element correction factors are valid.



#### **14.1.8 Initial Calibration Verification (ICV)**

A standard made from a second source from the calibration standards. It is analyzed immediately after the calibration to determine the validity of the calibration standards.

#### **14.1.9 Continuing Calibration Verification (CCV)**

A calibration standard (primary or secondary source) analyzed after every ten samples, and at the end of an analytical sequence to verify that the calibration is still valid.

#### **14.1.10 Reporting Limit Check Standard (RLCS)**

A check standard that is constructed out of either a primary or secondary source made up at same concentration as the reporting limit. An acceptance range of  $\pm 30\%$  for single analyte methods and  $\pm 50\%$  for multi-analyte methods was made the default. RLCS results are batched as a Standard Reference Material (SRM) which can be pulled into Element for control charting purposes.

#### **14.1.11 Initial Calibration Blank (ICB)**

A matrix matched deionized water sample ran to prove the system is clean with no carry-over.

#### **14.1.12 Continuing Calibration Blank (CCB)**

A matrix matched deionized water sample ran to prove the system is clean with no carry-over.

#### **14.1.13 Serial Dilution**

Dilute a sample by a minimum of five fold (1+4). Agreement within 10% between the concentration for the undiluted sample and five times the concentration for the diluted sample indicates the absence of interferences.

#### **14.1.14 Quality Control Sample (QCS)**

A solution of method analytes of known concentrations which is used to fortify an aliquot of blank solution or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards.

#### **14.1.15 Instrument Performance Check (IPC)**

A solution of method analytes, used to evaluate the performance of an instrument system with respect to a defined set of method criteria.

### **14.2 Control Charts**

SVL utilizes Element to provide personnel with the up to the minute ability to trend inputted QC results. It is recommended that analysts and technicians regularly consult trending charts to provide themselves with real time information. By trending an analysis, the analyst or technician can look at a current or past snapshot of QC recoveries and possibly determine when prep procedures or QC samples were done incorrectly or when they may have used contaminated or expired components. Trending can also be used to show when an instrument's components begin to degrade or fail.

The process is defined in SOP SVL 1033. RLCSs, prep blanks, LCSs, duplicates and matrix spikes are tracked. A standard X bar control chart is used to plot results. Upper and lower warning limits of  $\pm 2s$  (where  $s$  equals standard deviation) and upper and lower control limits of  $\pm 3s$  are calculated using at least 20 measurements (if possible) during a 6 month period.

### **14.3 Acceptance Limits**

Acceptance limits for quality control parameter recoveries may be set by published analytical methods, DQOs or be default limits set by the QA Manager. Individual SOPs will provide the accepted recoveries for each method. Acceptance limits are also outlined in SOP SVL 1033.

### **14.4 General Frequency of Quality Control Checks**

For those methods that do not have published QC requirements, SVL will use the following QC and frequency if applicable per batch of 20 samples:

Initial Calibration Verification once per calibration.

Initial Calibration Blank once per calibration.

Reporting Limit Check Standards at a minimum of 1 per analytical run.

Method or Instrument Blanks at a frequency of 5%.

Laboratory Fortified Blank or LCS at a frequency of 5%.

Matrix Spiked Samples at a frequency of 10%.

Matrix Spike Duplicates at a frequency of 5%.

Continuing Calibration Verification every ten samples.

Continuing Calibration Blank every ten samples.

## **14.5 Maintenance**

SVL breaks maintenance down into the following categories: initial maintenance, periodic maintenance, and after-maintenance performance checks. The requirements for performing maintenance or filling out maintenance logbooks can be found in SOP SVL 1030. Initial checks can be either checks performed during instrument setup or daily checks performed before the start of operations. Periodic checks are those checks that are performed on set time intervals (i.e. weekly, monthly, biannually, etc). After-maintenance checks are done after repairs have been completed or when an instrument has been moved to a new location. This is done in order to document acceptable ongoing instrument performance.

## **14.6 Uncertainty of Measurement**

SVL uses control charting as a means of determining when selected parameters (batch QC) are out of control. Warning and unacceptable control limits are defined at 2 and 3 sigma, respectively. See QM 14.2 and SOP SVL 1033.

Almost all approved methods used at SVL contain a section related to precision and bias. Random uncertainties cannot be determined statistically and can only be estimated by a trained analyst. Uncertainty represents a bias associated with analytical measurements. The presence and magnitude of bias can be determined by assessment of SVL's QC sample results on our analytical reports.

SVL reports data to 2 or 3 significant figures, dependent upon the sensitivity required by our clients, with the number of decimal places reported determined by the sensitivity of the method.

### **14.6.1 Rounding**

Rounding of analytical results is dependent upon the number of significant figures used by a method. Rounding for percent recovery on QC samples is also dependent upon the number of significant figures. Element is setup and our analysts are directed

to round up to the significant figure assigned to that method. SVL uses the following rounding rule: A result of 5 or greater rounds the results up to the significant figure assigned in Element.

## **15.0 CORRECTIVE ACTION**

The SVL Corrective Action Program is defined in SOP SVL 1019.

Any employee may initiate a Corrective Action Report (CAR) to support the quality system. Some examples are: The need for an SOP revision, incorrect results released to clients, an overdue MDL study, overdue or improper training, incorrect data reduction or review, improper instrument setup or calibration, or use of an incorrect analytical method.

If there is a non-acceptable result on a Performance Test Sample, the Quality Manager documents the failure as a CAR and works with the analysts and supervisors to discover the root cause of the failure. If there are findings from an internal or external audit, the Quality Manager issues a CAR to appropriate staff members so they can prepare a corrective action plan to rectify the issues.

Root cause analysis is the goal of corrective action and as such a cause will be identified, and a process outlined, so that a failure will not re-occur or its re-occurrence will be minimized.

### **15.1 Preventative Action**

A “preventative action” is a pro-active process for dealing with a problem before it happens. It is taken to eliminate the cause of an undesirable situation in order to prevent its occurrence rather than a reaction to the identification of a problem or nonconformity. These actions are taken to reduce the probability that a potential problem will occur. They may also include contingencies to reduce the “seriousness” should a future problem occur. Subjects for “preventative action” may be implemented to address a weakness in the quality system that is not yet causing nonconformities and can be initiated internally or externally (client complaints). The focus for preventative actions should be to avoid creating nonconformities, but may also lead to improved laboratory efficiencies.

SVL uses the CAR template to document ideas, plans or actions whether developed internally or externally. These reports are audited at

a future date to ensure that the changes sought have been implemented and are effective.

## **16.0 TRAINING**

SVL conducts annual training in legal and ethical responsibilities for all staff members. SVL provides training sessions that are developed in order to provide staff members with the analytical tools necessary for ever changing environmental regulatory requirements. New employees will be given various types of introductory training as soon as possible after their hire date.

SVL management and supervisors train staff members in laboratory safety. At a minimum this consists of an annual review of the Chemical Hygiene Plan. It also includes seminars on important safety issues throughout the year.

Staff members also receive training in the quality system and QM. At a minimum this consists of an annual review of the QM.

Department supervisors ensure that staff is adequately trained to perform the analyses assigned to them. The process is defined in SOP SVL 1010. Training includes, as appropriate, quality control requirements, instrument operation, instrument maintenance, software operation, reading the published method, reading the applicable SVL SOPs, and completion of an Initial Demonstration of Capability (IDOC). When an IDOC is not defined by the analytical method, the Quality Manager will create default criteria and outline them in the training summary forms which will be included in their personnel files. Upon completion of training, a Demonstration of Capabilities Certificate is placed within their personal file.

SVL Management defines the required elements for training for analytical methods. A Supervisor or a fully trained analyst provides training, when possible. If no fully trained analyst exists, an analyst may learn a new analysis by reading the appropriate method and instrument manual, then performing an IDOC.

During the training period, an analyst may produce data for clients (after completion of a successful blank and four separately prepared LCSs) under the supervision of a fully trained analyst; if there is not a trained analyst the Department Supervisor will review and sign off on all aspects of the work performed. A Department Supervisor or a fully trained analyst must review and sign all trainee work produced.

**16.1** To document continued proficiency, an analyst must perform one of the following tasks annually:

**16.1.1** Successfully analyze a blind performance sample.

**16.1.2** Complete another IDOC.

**16.1.3** Successfully analyze a blank and four separately prepared LCSs or duplicates (for those methods where a LCS is not commercially available).

**16.2** Analysts and technicians who do not successfully complete a DOC within a year must complete an IDOC before being re-certified for a method.

## **17.0 ETHICS AND CONFIDENTIALITY**

**17.1** SVL is committed to providing its clients with accurate and defensible data and meeting all client requirements for data quality and integrity. To achieve our commitment, and as a condition for employment with SVL, all employees agree to follow SVL's policy regarding ethics and data integrity characterized but not limited to the items listed below.

**17.1.1** All reported data, including dates and times, shall represent actual values obtained and are not modified or manipulated in any manner for which allowances have not been made for in the referenced method.

**17.1.2** There will be no misrepresentation of another analyst's identity.

**17.1.3** Altering the contents of logbooks and/or data sheets to misrepresent data is prohibited.

**17.1.4** Altering any operating procedures or QC to make data "fit" is prohibited.

**17.1.5** Failing to comply with SOPs without proper documentation and approval from the Laboratory Director and/or Quality Manager is prohibited.

**17.1.6** Any attempt to misrepresent data or events as they actually occur in the course of data production, review or reporting is prohibited.

**17.1.7** Deleting files, whether electronic or hard copy of raw data that was used in a reported value is prohibited.

**17.1.8** Engaging in, or being a party to, any practice that ultimately misrepresents data or narratives in any way is prohibited.

**17.2** SVL has established a zero-tolerance policy for improper, unethical, or illegal activities. Improper actions are defined as unapproved deviations from contract-specific or method-specific analytical practices, whether intentional or unintentional. Unethical or illegal actions are defined as the deliberate falsification of analytical or quality assurance results where failed method or contractual requirements are made to appear acceptable. Some examples of improper, unethical, or illegal practices are listed below. Comments in parentheses should each be read as beginning with the phrase “including but not limited to...”

**17.2.1** Improper use of manual integrations to meet calibration or method quality control criteria.

**17.2.2** Intentional misrepresentation of the date or time of analysis.

**17.2.3** Falsification of results to meet method requirements.

**17.2.4** Reporting results without analysis.

**17.2.5** Selective exclusion of data to meet quality control criteria (dropping calibration points).

**17.2.6** Unwarranted manipulation of computer software.

**17.2.7** Improper alteration of analytical conditions (changing voltages or run times).

**17.2.8** Misrepresentation of quality control samples (not preparing them as samples).

**17.2.9** Intentionally reporting results from one sample for those of another.

**18.2.10** Reporting calibration or quality control data not linked to the reported samples.

### **17.3 Confidentiality**

SVL’s commitment to client confidentiality (including national security concerns) and any associated proprietary rights comes first and foremost. We understand the nature of doing business in a litigious society and will seek to protect our client’s interest in all aspects of our work.

## **18.0 DATA REVIEW**

SVL uses a three-tier system for data review via the LIMS. The first level is conducted by the analyst, the second level by a peer or supervisor, the third by a signatory, DCO, Technical Director or the Laboratory Director. Reviews take place upon the review of raw data or within the LIMS (which uses a system of locks to assure data is secure from accidental overwriting). Most data is available in PDF, which can be reviewed at any work station. The process is governed by SOP SVL 2009.

In the case that erroneous data does leave the lab, the Laboratory Director or Client Services will contact the affected clients as soon as all of the facts are available. SVL will work with the clients in seeking a new or alternative strategy to meet the client's needs.

### **18.1 Electronic Signatures**

For all levels of review up to the final review Element provides an audit trail of who has uploaded and reviewed results. Employees are directed to log in and out of Element so that they are identified when conducting data uploads or reviews; it is not permissible to use another employee's password or misrepresent an analyst or reviewer by not logging in to a computer system under the correct username and password (see SOP SVL 1031).

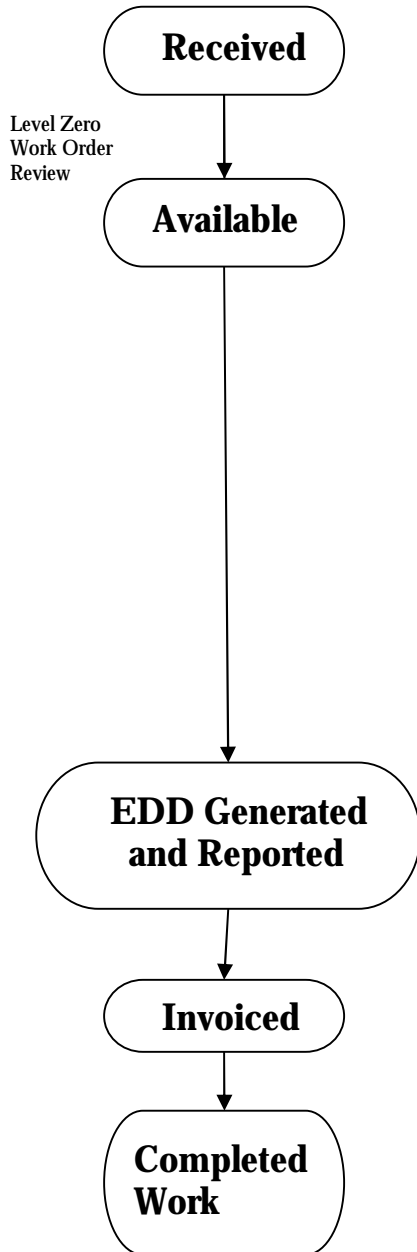
The electronic signature affixed to the Final Report will be assigned by the Document Control Office dependent upon which reviewer signed the Work Order Review Checklist.



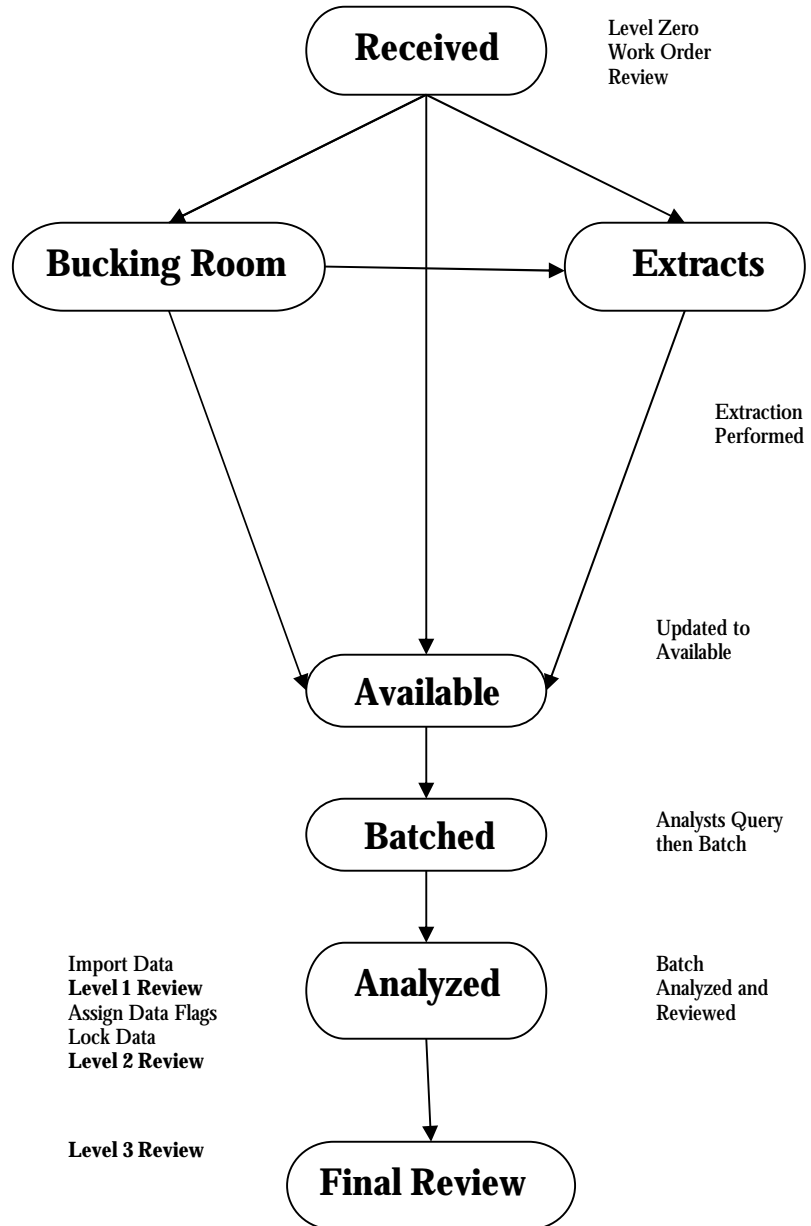
## 18.2 Data Review Flow Chart

### Work Order Status

Samples are logged in, and analyses assigned



### Analysis Status



## **19.0 REPORTING**

SVL has a single standard report format for nearly all results (SVL\_Sample) generated by Element. This includes a case narrative, sample report, and QC report.

Reports are also available in a number of routine and custom hardcopy formats. EDDs can be provided in ASCII, spreadsheet, and database formats, including EQWin, GIS/Key, and EnviroData Solutions. If a client has a specific format, SVL is usually able to provide data compatible with their preferred format.

Data that will be used to create EPA CLP-like deliverable packages may be done in Element or can be loaded into a third party data review and reporting system MARRS that will generate the forms required to complete a data package. SVL has the capability of providing a hardcopy and EDD format. EDDs are available in standard EPA CLP formats, as well as popular spreadsheet and database files.

## **20.0 AUDITS AND VERIFICATION PRACTICES**

### **20.1 Performance Testing Program**

SVL participates in two WS, two SOIL, and two WP Performance Testing (PT) studies each year. SVL uses the first WP Study to meet the DMRQA requirements of our clients. The PT samples are logged in as single-blinds and ran as if they were normal samples in all aspects. The Quality Manager is responsible for preparing all PT samples. QuiK™ Response samples are used when SVL does not pass an analyte required by our accreditation.

### **20.2 Internal System Audits**

The Quality Manager conducts a minimum of one internal system audit per year per lab. The audit provides an overview of the implementation of procedures and policies set forth in the laboratory's QM and SOPs; ref, SOP SVL 1005. Other audits (that may be limited in scope) may be undertaken at any time in response to external audits, CARs, or at the request of the Laboratory Director.

The Quality Manager prepares an internal audit plan based on information garnered from previous audits both internal and external, CARs, method changes, new instrumentation and requests or complaints from clients. The Quality Manager may use written

checklists and/or quizzes to assess an analyst's knowledge of the QM, methods and current SVL SOPs.

The Quality Manager will interview the analyst(s) and conduct reviews of records, logbooks, and data packages.

At the close of the audit, a post-audit meeting is held to discuss the audit findings. The assessor or Laboratory Director can close a finding during this discussion if the laboratory staff can satisfactorily demonstrate that the finding is inappropriate or easily remedied.

The Quality Manager will deliver the audit report to the President, Laboratory Director, Technical Director, supervisor and appropriate staff. A report will contain at a minimum the following parameters: Date and location of the audit, personnel involved in the audit, laboratory operations audited, any minor or major findings that require corrective action (major findings require the issuance of a CAR) and the assessor's summation.

### **20.3 Reference Materials**

Companies like ERA, High Purity, Fisher and Baker have been approved (see SVL's approved vendor list) to provide SVL with reference materials and reagents. SVL uses a second source verification for all calibrated methods. When there is not a secondary source provider available, SVL will verify and then purchase a separate lot from the primary vendor (lots must not be from the same parent batch).

### **20.4 Internal Quality Control Schemes**

SVL has instituted a Reporting Limit Check Standard (RLCS) to verify recovery at the reporting limit; this check has been instituted at SVL for SDWA, CWA and Solid Waste analytical runs. SVL has also instituted a calibration curve verification policy where calibration standard recoveries are fitted back into the curve. A standard at the reporting level must be within 30% of the true value and the remaining standards must be within 10% of their true values. Any exception to this rule will be outlined in the appropriate SOP.

### **20.5 Data Audits**

The Quality Manager performs a data audit of several data packages each year. Data audits can also be triggered by audits, CARs or requests from the Laboratory Director. The purpose behind the data audits is to alert SVL to any errors, systemic problems or trends that may be developing.

## **21.0 MANAGEMENT REVIEW**

The Management of SVL conducts a review of the adequacy of the quality system weekly. The reviews take into account reports from supervisory personnel, Client Services, Technical Directors, Document Control Officer, Systems Manager, LIMS Chemist, Quality Manager and President. Recent internal audits, external audits, the results of PT samples, changes to the volume or type of work undertaken, feedback from clients, instrumentation issues, personnel issues and CARs are a few of the items discussed. Conclusions or action items are addressed; any changes deemed necessary are then incorporated into revisions to the QM and SOPs as soon as practicable and communicated to relevant employees to provide direction for day-to-day operations. Notes from these meetings are kept in an electronic file located on SVL's network.

## **22.0 CONTRACTS**

SVL has established a Project/Bid Review Sheet to meet the TNI requirements of Section 4.4 "Review of Requests, Tenders, and Contracts." Any differences between the request or tender and the contract shall be resolved before any work commences. Each contract will be acceptable to both the laboratory and the customer. Records of reviews (including significant changes) are maintained in the appropriate client files. Customers will be informed of any deviation from the contract including those by subcontractors. If a contract needs to be amended a new Project/Bid Review sheet will be utilized with all applicable parties being informed of the changes.

## **23.0 SUBCONTRACTING AND PURCHASING**

### **23.1 Subcontracting**

Prior to subcontracting work to another laboratory, the Laboratory Director or Client Services will ensure that the subcontracted laboratory is NELAP accredited, or is certified by the appropriate state (for the tests being subcontracted) if required. SVL will advise the customer in writing or email as to the need for subcontracting and will receive in return the client's approval (to be placed in the client's file). The Quality Manager upon being provided sub-contractor information will verify that the subcontracting laboratory has an active Quality Assurance Program (QAP) that meets SVL's and our client's

DQOs. The Sample Custodian is responsible for verifying that the subcontracting lab received the correct samples and that they were assigned the requested analyses. The subcontracting laboratory will be identified on the final report.

### **23.2 Purchasing**

SVL maintains a vendor file which contains the vendors approved to supply products to SVL.

SVL ensures that purchase orders contain the required technical and quality specifications prior to submission. If a method or instrument requires specific technical and quality criteria (like grade or purity) then the Department Supervisor will ensure this is the product indicated on the purchase order. Identification of the product is by description and catalog number (see appropriate method SOPs).

SVL tests reagents and standards prior to analyzing samples and reporting data. New reagents and standards will be used in a laboratory fortified blank at RLCS levels; if the QC requirements are met then those reagents are deemed to be acceptable; ref, SOPs SVL 1015 and SVL 1032.

## **24.0 SERVICE TO THE CLIENT**

SVL seeks to have an excellent working relationship with our clients. In order to monitor client's concerns, SVL will place both positive and negative feedback in the client's file. If clients do not provide feedback, Client Services will ask questions or provide clients with a written survey to assess any unspoken concerns.

### **24.1 Complaints**

The Client Services Department strives to resolve all complaints from clients regarding analytical reports or service. Client Services will contact the appropriate Director, or Department Supervisor to investigate and resolve issues. Actions may include reanalysis of samples and/or explanations surrounding technical issues/lab procedures.

### **24.2 Reanalysis**

Reanalysis, whether requested by a client or by SVL personnel, must have reasonable justification for it to be valid. Before proceeding with the reanalysis of sample, it is important to understand what SVL's or the

client's objective is in requesting the reanalysis. The SOP will outline procedures to be followed when a reanalysis is requested. It will discuss the documentation (reanalysis request form and work order memos) associated with the reanalysis. This documentation will provide the laboratory with a means of tracking changes to our work orders and providing the necessary information for historical reconstruction. Definitions for words used in this SOP may be found in the QA Manual. SVL does not conduct reanalysis in order to "result hunt". Reanalysis is conducted by SVL at the request of clients or SVL personnel in order to confirm a possible error on the part of SVL or by any of the sample custodians listed on the chain of custody. SVL will report out (at the Lab Director's discretion) all sample results when a reanalysis is requested by a client, such data will be accompanied by a case narrative. When reanalysis is requested on a method that has multiple analytes, the sample shall be reanalyzed for all of the analytes originally requested (at the supervisors discretion the other analytes may not be re-reported if it is shown that they are scientifically indistinguishable from the original results) under that method. Work order memos will be established when a client requests a reanalysis and should be updated throughout the reanalysis run and review. Case narratives will be written up to explain any discrepancies between the original test results and the reanalysis conducted (any reissued report will contain a case narrative). Samples that are reanalyzed in-house will have the reason for the request clearly identified on the reanalysis request form. Whether internal or external, the reanalysis request form must be filled out completely to assist with the historical re-construction of the data and to assist in writing up case narratives or CARs; ref, SOPs SVL 2019 and SVL 1019.

## **25.0 TRANSFER OF ANALYTICAL REPORTS, RECORDS, and SAMPLES**

In the event that SVL Analytical, Inc. goes out of business or there occurs a transfer of ownership, the following plans will apply.

All current clients and past clients going back 5 years, longer if bound by contract, will be contacted by registered mail, return receipt requested, at their current or last known address, and made aware of the permanent closure or transfer of ownership of SVL.

Clients will be requested to respond in writing by return mail, fax or email within 10 business days with the instructions as to the final disposition of (in

the case of closure) or as to how they wish to proceed with the new ownership, concerning: their reports, records and/or samples, including work that is in progress.

Options for the client may include complete transfer of all reports, records and samples to their business location, or complete destruction of all documents and samples. SVL does not take ownership of client samples at any time or under any circumstances, and title to all reports, records and samples resides with the client. SVL will not be responsible for disposal of hazardous materials.

Methods of reports and records transfer may be by hard copy purge file, hard copy reports only, or by electronic data deliverables (EDD) for all date accessible records stored in SVL's database. No customized EDDs will be available.

Should a client decide to stay with the new ownership, any business relationship between the two parties will constitute a new relationship independent of any involvement by SVL. The maintenance of reports and records, and the completion of the work in progress (but not completed by SVL) shall be under the sole control of the new owner. SVL will be relinquished from any and all responsibilities concerning the business relationship between the parties.

## 26.0 GLOSSARY

Calculations and definitions may be found in SOP SVL 1028.

**Acceptance Criteria:** Specified limits placed upon characteristics of an item, process, or service defined in required documents.

**Accuracy:** The degree of agreement of a measured value with the true or expected value of the quantity of concern.

**Acid Base Accounting (ABA):** The Acid-Base Account is determined by calculation from the ANP and AGP results. The Acid-Base Account may be reported as the ABA, Acid Base Potential (ABP), or Net Neutralizing Potential (NNP) at a client's request.

**Acid Generating Potential (AGP):** The acid generating potential is established by determining three sulfur content numbers, the "Total Sulfur", "Non-Extractable Sulfur", and "Non-Sulfate Sulfur" or "Non-Sulfate Sulfur-HCl". Total Sulfur is determined from analysis of a 0.2 g aliquot taken from a sample that has undergone a 200 mesh screening. Non-Extractable Sulfur is

determined after digestion with 2N nitric acid, then filtered, and analyzed by a LECO analyzer. Non-Sulfate Sulfur is determined after digestion with hot water, then filtered, and analyzed by a LECO analyzer. Non-Sulfate Sulfur-HCl is determined after digestion with a 2:3 HCl solution, then filtered, and analyzed by a LECO analyzer.

**Acid Neutralizing Potential (ANP):** The amount of neutralizing bases, including carbonates, present in overburden materials is found by treating a sample with a known excess of standardized hydrochloric acid. The sample and acid are heated to insure that the reaction between the acid and the neutralizers goes to completion. The calcium carbonate equivalent of the sample is obtained by determining the amount of unconsumed acid by titration with standardized sodium hydroxide.

**Aliquot:** A portion of a sample.

**Alkalinity:** A measure of the acid-neutralizing ability of the sample.

**Analytical Spike:** An aliquot of sample to which a known amount of analyte has been added after sample preparation. It is a measure of the effect of the matrix of a digest or extract. It is sometimes known as a post-digestion spike.

**Batch:** Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same reagents. For SVL's purposes a batch will not include more than 20 samples.

**Bias:** A systematic error inherent in a method or caused by some idiosyncrasy of the measurement system. Temperature effects, extraction efficiencies, contamination, mechanical losses, and calibration errors create bias. Bias may be either positive or negative.

**Blank:** An artificial sample designed to monitor the introduction of contamination into the process. For aqueous samples, reagent water is used as a blank matrix.

**Blind Sample:** A sample submitted for analysis whose concentration is unknown to the analyst.

**Buffers:** Solutions of a weak acid and a salt of the acid or weak base and a salt of the base that are capable of maintaining pH on addition of acid or base.

**Calibration:** Comparison of an instrument response with a standard or a certified instrument. Commonly it is performed with a set of known standards plotted versus a response.



**Calibration Blank:** See Section 14.0 Quality Control.

**Calibration Curve:** Graphical plot of instrument response against amount of analyte in standards. The relationship can usually be modeled as linear or quadratic.

**Completeness:** The percentage of measurements that meet quality control acceptance criteria for requested determinations. Percentage completeness is defined by client DQOs.

**Continuing Calibration Verification (CCV):** See Section 14.0 Quality Control.

**Continuing Calibration Blank (CCB):** See Section 14.0 Quality Control.

**Control Chart:** A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.

**Custody Log:** A system for tracking samples from the time they enter the lab until a final report is generated.

**Digestion:** Solubilizing of metal analytes through heating with a variety of acids or oxidizers.

**Dissolved Analytes:** An aqueous sample that has been passed through a 0.45  $\mu\text{m}$  filter. The filtered portion is then run for dissolved analysis.

**Double Blind Sample:** A sample known by the submitter but submitted to an analyst in such a way that its identification as a check sample is unknown.

**Duplicate Sample:** See Section 14.0 Quality Control.

**Extraction:** The process of removing analytes through the addition of acids or water from a solid/semi-solid matrix. SVL performs TCLP, SPLP, CA-WET and Meteoric Water Mobility extractions.

**Field Blank:** See Section 14.0 Quality Control.

**Field Duplicate:** Duplicate samples obtained in the field and analyzed in the lab to assess field precision in sampling.

**Hardness:** Dissolved metal content of water, expressed as calcium carbonate equivalents.

**Homogeneity:** The degree to which a property or substance is evenly distributed throughout a material.

**Initial Calibration Verification (ICV):** See Section 14.0 Quality Control.

**Instrument Detection Limit (IDL):** The smallest concentration detectable on a specific instrument. It is statistically determined by analysis of at least seven replicates of a blank that has not been digested.

**Interference Check Sample (ICS):** A sample with known concentrations of elements used to determine if the inter-element correction factors of the ICP are accurate.

**Inter-element Correction Factor (IECs):** The effect one element has on other elements due to wavelength overlap. These effects are accounted for and subtracted out resulting in a less biased result.

**Internal Standard:** Pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not in the sample.

**Initial Calibration Blank (ICB):** See Section 14.0 Quality Control.

**Instrument Performance Check (IPC) Solution:** A solution of method analytes, used to evaluate the performance of the instrument system with respect to a defined set of method criteria. The CCV or LCS may fit this criteria.

**Laboratory Control Sample (LCS):** See Section 14.0 Quality Control.

**Laboratory Fortified Blank (LFB):** Another term for a laboratory control sample.

**Laboratory Fortified Matrix (LFM):** Another term for a matrix spike.

**Laboratory Information Management System:** A software-based laboratory and information management system that offers a set of key features that support a modern laboratory's operations.

**Laboratory Reagent Blank (LRB):** Another term for a method blank.

**Langlier's Index:** An analytical measure of the corrosivity of water.

**Limit(s) of Detection (LOD):** 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.

**Limit(s) of Quantitation (LOQ):** The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

**Linear Calibration Range (LCR):** The calibration range over which the instrument response to analyte is linear.

**Linear Dynamic Range (LDR):** The concentration range over which the instrument response to analyte is linear.

**Manual Integration:** Anytime a chromatogram is altered by an analyst from the original software determined chromatogram, usually performed by adjusting how the baseline was assigned.

**Material Safety Data Sheet:** Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire and reactivity data including storage, spill and handling precautions.

**Matrix:** The substrate of a test sample.

**Matrix Spike (MS):** See Section 14.0 Quality Control.

**Matrix Spike Duplicate (MSD):** See Section 14.0 Quality Control.

**Maximum Contaminant Levels:** Regulatory action levels for primary drinking water analytes.

**Mean:** The sum of all observations divided by the number of observations.

**Method:** A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order they are to be performed.

**Method Blank:** See Section 14.0 Quality Control.

**Method of Standard Addition:** Commonly used to determine the concentration of an analyte in a complex matrix. The matrix may contain other components that interfere with the analytical signal causing inaccuracy in the determined concentration. Known concentrations are added to a volume of sample to develop a curve based upon the interferences from that sample, so that a reliable concentration can be derived for the sample.

**Method Detection Limit (MDL):** The smallest concentration detectable on an instrument with 99% certainty by a specific method. It is statistically determined by analysis of seven replicates of a low-level standard, prepared in the same way as a sample.

**NTU:** Nephelometric turbidity unit.

**Net Carbon Value (NCV):** A method used in the determination of Acid Generation Potential and Acid Neutralizing Potential using the Net Carbonate Value method AGP is calculated via sulfur pyrolysis and ANP is calculated using digestion with hydrochloric acid.

**Net Acid Generation (NAG):** A solution of hydrogen peroxide is added to rock samples which have been reduced to pass through a -200 mesh screen. The sample and the hydrogen peroxide are heated to ensure the reaction goes to completion. The hydrogen peroxide reacts with the sulfides, carbonates and other materials in the sample to produce a net pH.

**Performance Test (PT) sample:** A sample, the composition of which is unknown to the laboratory is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.

**pH:** The negative log of activity of the hydrogen atom.

**Precision:** The degree of agreement of independent measurements under specified conditions.

**Quality Assurance:** A system of activities used to ensure defined standards of quality.

**Quality Control:** A system for verifying and maintaining the desired level of accuracy and precision of an analytical method.

**Quality Control Sample (QCS):** A solution of method analytes of known concentrations which is used to fortify an aliquot of LRB or sample matrix. The QCS is prepared from a secondary source. The ICV fits these criteria.

**Relative Standard Deviation (%RSD):** The Standard Deviation divided by the Mean and multiplied by 100.

**Relative Percent Difference (%RPD):** The difference between two values divided by the average of the values, expressed as a percent.

**Reporting Limit (RL):** The smallest concentration usually reported for an analyte. It is usually at least three times the Method Detection Limit.

**Reporting Limit Check Standard (RLCS):** See Section 14.0 Quality Control.

**Residues:** Remainder after removal of water or other liquids, see solids and total solids.

**Retention Time:** Elapsed time between the injection of sample to the elution of the sample.

**Run Logs:** A log book for each instrument listing consecutively what was run, the method, when, by whom, and what file name the raw data is filed under.

**Serial Dilution:** See Section 14.0 Quality Control.

**Standard Operating Procedure (SOP):** A written procedure that defines a laboratory operation or analytical method.

**Sub-sample:** A portion taken from a sample.

**Standard Deviation:** The square root of the variance. A measure of the average spread around the mean.

**Titration:** Any number of methods for determining volumetrically the concentration of a desired substance in solution by adding a standard solution of known volume and strength until the reaction is complete, usually as indicated by a change in color due to an indicator.

**Total Recoverable Metals:** Follow the digestive method outlined in 40 CFR 136 Appendix C Section 9.4. Results are reported as “total metals”. This is SVL’s default total metals method unless both total and total recoverable metals are requested.

**Traceability:** The ability to trace the history, application, or location of an entity (e.g., standard, reagent, sample). SVL tracks the entities from the moment it enters the premises until the time it is disposed of.

**Trip Blank:** See Section 14.0 Quality Control.

**Tuning Solution:** A solution which is used to correct instrument performance prior to calibration and sample analysis.

**Variance:** The value approached by the average of the sum of the squares of deviations of individual measurements from the mean.

## **27.0 CERTIFICATIONS**

**SVL maintains certification for analysis of drinking water in the following states:**

Arizona  
Florida  
Idaho  
Nevada  
Washington

**SVL maintains certification for analysis of CWA and SW-846 samples in the following states:**

Arizona  
California  
Florida  
Nevada  
Washington

**NELAC Certification Awarded – Primary Accreditation Florida**

**27.1** Copies of the Scopes of Accreditation can be located at [www.svl.net](http://www.svl.net) .

## **28.0 RESUMES**

### **WAYNE R. SORENSEN**

#### **PROFESSIONAL EXPERIENCE:**

##### **SVL Analytical, Inc. - Kellogg, ID 1991- Present**

President / CEO - Administers company policies and formulates business strategies.

##### **SVL Analytical, Inc. - Kellogg, ID 1987-1991**

Laboratory Director: Responsible for all analytical and operational activities of the laboratory; supervised personnel

##### **SVL Analytical, Inc. - Kellogg, ID 1973-1987**

Analytical Chemist: Analyzed soils and water for metals by flame atomic absorption and graphite furnace (7000 methods), for mercury by cold vapor atomic absorption (methods 7470 and 7471); for cyanide (method 9012), fluoride (method 340.2), phosphate (method 365.2), pH (method 150.1), turbidity (method 180.1), and conductivity (120.1); analyzed soils and house dusts for lead, arsenic, cadmium; analyzed hi-vol filters for metals by flame atomic absorption; performed baseline study analyses for permitting mine sites; conducted analysis for Remedial Investigation and Feasibility Study for Bunker Hill Superfund Site..

##### **The Bunker Hill Company - Kellogg, ID October 1969-April 1973**

Supervised a large integrated mine, mill and smelter analytical laboratory and trained personnel.

##### **Kennecott Copper, Ray Mines Division March 1968-October 1969**

Chief Chemist: Supervised an assay lab, trained assayers for new analytical methods and conducted applied research.

##### **Kennecott Copper, Western Mining Division Research Center May 1965-March 1968**

Analytical Chemist: Analytical methods development and applied metallurgical research on copper.

#### **EDUCATION:**

##### **Utah State University - Logan, UT 1958-1962**

B.S. Chemistry (minor: mathematics, physics)

##### **Salt Lake Trade Tech - Salt Lake City, UT 1965**

Basic Industrial Statistics

## John R. Kern

### PROFESSIONAL EXPERIENCE:

#### **SVL Analytical, Inc. - Kellogg, ID October 2007 - present**

**Laboratory Director:** Manage and direct the activities of the laboratory; establish ethical norms; evaluates personnel performance; conduct QA/QC reviews of incoming work and completed reports; work with the QA Manager to evaluate compliance with SOPs and methods.

#### **P3 Scientific - Oakdale, MN September 2005 - April 2007**

**Laboratory Manager – Chemistry:** Management and operation of a laboratory at a cGMP/GLP compliant CRO, providing analytical (organic and inorganic analysis) and microbial services to the chemical industry.

#### **Arena Pharmaceuticals, - Inc. San Diego, CA January 2003 - August 2005**

**Associate Director, Analytical Chemistry – Pharmaceutical Development:** Direct the analytical chemistry laboratory within the pharmaceutical development unit at a start-up biotech/pharmaceutical company.

#### **LC Resources - McMinnville, OR 1991 - 2003**

**Laboratory Director:** Started and built up a contract research laboratory specializing in HPLC and LC/MS/MS services for the pharmaceutical and chemical industries. Oversaw the growth of the lab from 2 to 20 employees, with annual sales of over 3 million. Directly responsible for the day-to-day operation of the lab including project management, experimental design, preparation of proposals, client interface, contracts, budget, oversight of QA and QC departments, SOP and protocol preparation. This position involved extensive interaction with major pharmaceutical companies in negotiating contracts, planned studies, allocating resources, report preparation, and discussing technical issues. Experience was also gained in the direction of projects involving analysis of a wide variety of pharmaceutical products from OTC to complex proteins, and drugs in biological matrices.

#### **Syntex USA, Inc. – Palo Alto, CA 1984 - 1991**

**Senior Chemist:** Development of analytical methods for the analysis of active pharmaceutical ingredients (AIP) and determining release specifications. Prepared analytical sections for IND and NDA applications. Supervised laboratory staff and project team membership.

### EDUCATION:

#### **Montana State University - 1982**

M.S. Chemistry

#### **Eastern Michigan University - 1978**

B.S. Biochemistry



## **KIRBY L. GRAY**

### **PROFESSIONAL EXPERIENCE:**

#### **SVL Analytical, Inc. - Kellogg, ID Dec. 2004-present**

Technical Director - Conducts QA/QC reviews of commercial and EPA (ILMO5.4) incoming work and completed reports; supervises laboratory activities related thereto; primary contact with EPA (SMO); verifies SDGs, and responsible for MARRS (electronic data deliverable system) in coordination with DCO prior to reporting.

#### **SVL Analytical, Inc. - Kellogg, ID March 1987-2004**

Inorganic Instrumental Chemistry Department Supervisor -- Responsible for sample analysis by ICP, GFAA, FLAA, IC and CVAA.

#### **Radersburg Mining Co. - Toston, MT September 1986-March 1987**

Chemist: -- Responsible for fire assay, FLAA, and sample preparation.

#### **IDHW, State of Idaho - Kellogg, ID August 1986**

Environmental Technician: -- Operated X-ray fluorescence meter and collected soil samples.

#### **Sunshine Mining Co. - Kellogg, ID May 1984-May 1986**

Chemist -- Responsible for fire assay, FLAA, and classical chemistry.

#### **The Bunker Hill Co. - Kellogg, ID May 1972-May 1982**

Material Recovery Supervisor -- Responsible for operation and maintenance of water treatment plant, sulfuric acid plant, baghouse, cadmium refinery, and electric reverberatory furnace at a lead smelter.

### **EDUCATION:**

#### **University of Idaho - Moscow, ID Sept 1968-May 1972**

B.S. Geological Engineering

#### **North Idaho College-Coeur d'Alene, ID Sept 1966-June 1968**

Engineering major

## NAN WILSON

### PROFESSIONAL EXPERIENCE:

#### SVL Analytical, Inc. - Kellogg, ID March 2003 -- present

**Technical Director** October 2007 – present: Conducts QA/QC reviews of incoming work and completed reports, supervises laboratory activities.

**Laboratory Director** October 2006—October 2007: Manage and direct the activities of the laboratory; establish ethical norms; evaluates personnel performance; conduct QA/QC reviews of incoming work and completed reports; work with the QA department to evaluate compliance with SOPs and methods.

**QA Coordinator** April 2006-October 2006: maintain Quality Systems, draft & approve SOPs, coordinate Quality System Audits, coordinate PT testing.

**QA Chemist** September 2004 – March 2006: maintain Quality Systems, draft SOPs, assisted with Quality System Audits.

**Safety Director** September 2004-October 2006: maintain Chemical Hygiene Plan, coordinate safety training and record keeping.

**Organics Department Chemist** March 2003-August 2004: Analyzes samples for volatile organic compounds by GC.

#### LC Resources—McMinnville, OR September 1997-January 2003

**Manager, Pharmaceutical Analysis** January 2001-January 2003: Supervised HPLC method development; coordinated work for chemists and technicians; directed method validation; wrote SOPs and validated protocols; prepared client reports; trained chemists and technicians on SOPs and computer software; presented data and reports; responsible for client contact; administered Millennium32 chromatography software

**Chemist** September 1997-January 2001: Developed HPLC methods for pharmaceuticals; operated, calibrated, and maintained HPLC, UV/Vis, pH meters, balances, pipettes; wrote client reports; administered Millennium32 chromatography software

#### SVL Analytical—Kellogg, ID 1987-1996

**Laboratory Technician**—Performed meteoric water mobility tests; analyzed for acid base accounting; alkalinity, acidity, pH, sulfur forms by LECO, carbonate, oil and grease, TSS, TDS, gravimetric and colorimetric methods

#### Willamette University—Salem, OR 1995-1996

**Laboratory Teaching Assistant**—Assisted organic chemistry students in successfully carrying out lab experiments

### EDUCATION:

#### Willamette University—Salem, OR 1992-1996

B.A. Chemistry and Russian

#### Simferopol State University—Simferopol, Ukraine 1995

Semester abroad

### ADDITIONAL COURSES:

#### Laboratory Safety Institute, Tuscon AZ 2005

Two Day Lab Safety Short Course

## **Brandon A Borgias**

### **PROFESSIONAL EXPERIENCE:**

#### **SVL Analytical, Inc. – Kellogg, ID 1991-Present**

Systems Manager, Computational Chemist – Oversees the Laboratory's Information Management System (LIMS) and works with our clients on custom reporting and electronic deliverables.

#### **Cray Research– San Ramon, CA Jan 1989-1990**

Software Technical Support Analyst 0 Co-administrator of network, composed of eight file servers and over 50 client work stations distributed throughout the western U.S. Unix (Sun OS and Cray UNICOS) operating systems experience

#### **University of California, UCSF – San Francisco, CA 1985-1989**

Postdoctoral Scholar – Developed computer programs (FORTRAN) for the refinement and analysis of macromolecular structure. VAX, Sun, and Cray computers and VMS and UNIX operating systems.

### **EDUCATION:**

#### **University of California, Berkley – Berkley, CA 1979-1985**

Ph.D. Chemistry

#### **Reed College – Portland, OR 1975-1979**

B.S. Chemistry/Physics

# **MICHAEL S. DESMARAIS**

## **PROFESSIONAL EXPERIENCE:**

### **SVL Analytical, Inc. - Kellogg, ID Oct. 2006 - Present**

Quality Assurance Manager -- Coordinates and develops quality assurance and training programs for the laboratory, maintains laboratory accreditations, writes standard operating procedures, reviews data, conducts audits, performs root cause analysis.

### **SVL Analytical, Inc. - Kellogg, ID June 2004 – Oct. 2006**

Chemist Inorganic Instrument Department – Responsible for analysis of samples for trace metals by EPA methods 200.7 and 6010B. Interprets and reports data.

### **SVL Analytical, Inc. - Kellogg, ID April 2004 – June 2004**

Chemist Organic Chemistry Department – Responsible for analysis of samples for pesticides and PCBs by EPA methods 608, 8081A, and 8082. Interprets and reports data.

### **U.S. Army Engineer District-Alaska – Umiat, AK May 2003 - Sept. 2003**

Alaska Dept. Environmental Conservation approved field chemist. Established field laboratory, developed and implemented QA/QC under USACE and ADEC requirements. Surveyed, sampled and tested soils and waters under a Total Environmental Restoration Contract (TERC).

### **North Creek Analytical Oct. 1997 - Dec. 2002**

Senior Metals Chemist and Health/Safety Officer - Developed, revised and implemented safety and HAZMAT procedures. Developed and documented standard operating procedures. Maintained analytical instrumentation and analyzed samples for trace metals (ICP, AA and GFAA) and BTEX/GRO.

## **EDUCATION:**

### **Eastern Washington University – Cheney, WA 1996-1997**

Graduate coursework in Hydrology and Fisheries.

### **Washington State University – Pullman, WA August 1993-June 1995**

B.S. in Physical Science (emphasis in Chemistry, Geology, and Environmental Science).

### **Yakima Valley Community College 1991**

A.A.

## **Dianne Gardner**

### **PROFESSIONAL EXPERIENCE:**

#### **SVL Analytical, Inc. - Kellogg, ID May 2011 - Present**

Classical Chemistry Department Supervisor -- Supervises the staff and operation of SVL's TDS, Nutrient, TKN, cyanide, NOX/NH<sub>4</sub>, Leco, and extraction labs. Ensures that EPA, ASTM and Standard Method methods are correctly followed. Requisitions instrumentation and supplies. Reviews manually entered lab data prior to entry into Element (LIMS). Reviews level 1 data entry prior to submission to DCO for reporting.

#### **SVL Analytical, Inc. -- Kellogg, ID January 2007- May 2011**

Instrument Department Analyst – Responsible for analysis of digested samples by ICP-AES and ICP-MS for trace metals by EPA methods 200.7, 200.8, 6010B, 6020B, and EPA SOW ILMO5.4. Interprets and up loads data to Element (LIMS). Back up analyst for GFAA.

#### **SVL Analytical, Inc. - Kellogg, ID – April 2004 to January 2007**

Classical Chemistry Department Chemist—Analyzed soil and aqueous samples for Cyanide.

### **EDUCATION:**

#### **Cedarville University – Cedarville, OH June 1987**

B.A. Chemistry

#### **North Idaho College – Coeur D'Alene, ID 1997**

Coursework in Microbiology

## **DANNY J. SEVY**

### **PROFESSIONAL EXPERIENCE:**

#### **SVL Analytical, Inc. - Kellogg, ID Dec 2004-present**

Instrument Department Supervisor – Supervises staff and operation of SVL's ICP-AES, ICP-MS, CVAA, GFAA, FLAA, and IC labs and their respective sample preparation labs. Ensures that EPA and Standard Method methods are correctly used, including EPA SOW ILMO5.4. Approves lab data in Element (LIMS) prior to submission to DCO for reporting.

#### **SVL Analytical, Inc. - Kellogg, ID 1996-2004**

Inorganic Instrument Operator -- Performs metals analysis by ICP and IC.

#### **SVL Analytical, Inc. - Kellogg, ID 1994-1996**

Classical Chemistry Analyst -- Performed classical Wet Chemistry analyses on water and soil sample, including the preparation and analysis of cyanide and nitrate/nitrite (as N) tests for soil and water samples.

#### **SVL Analytical, Inc. - Kellogg, ID 1988-1994**

Instrument Operator -- Analyzed samples using Cold Vapor Atomic Absorption and Ion Chromatography

#### **SVL Analytical, Inc. - Kellogg, ID 1987-1988**

Laboratory Technician -- Performed inorganic sample preparation and operated CVAA and GFAA instruments.

### **EDUCATION:**

#### **Perkin Elmer April 2008**

Inorganic Workshop Series

#### **Perkin Elmer July 2004**

ICP-MS with Elan Software & Elan DRC Accessory Training Course

#### **Perkin Elmer November 2001**

Optima Instrument Series with ICP WinLab Software

#### **OI Corporation January 2001**

Operation of FS-3000 Auto-analyzer

#### **North Idaho College - Coeur d' Alene, ID 1989-1990**

Chemistry and Mathematics courses

## Heather Green

### PROFESSIONAL EXPERIENCE:

#### **SVL Analytical, Inc. -- Kellogg, ID June 2011 – Present**

Acid/Base Department Supervisor – Responsible for analysis and technicians within the department. Responsible for method interpretation and development.

#### **SVL Analytical, Inc. -- Kellogg, ID Sept. 2010 – June 2011**

Leco Analyst – Responsible for the following methods: ABA, AGP, ANP, NCV, NAG, total carbon and total sulfur.

#### **SVL Analytical, Inc. -- Kellogg, ID Sept. 2009 – Sept, 2010**

Classical Chemistry Floater – Responsibilities will include becoming certified in multiple disciplines in order to back-up primary analysts and technicians.

#### **Bio Medics Plasma Center - Moscow, ID – Nov. 2007 to May 2009**

Duties included: calibrating equipment, screening donors, conducting historical surveys and performing various test on blood samples.

Worked under highly regulated guidelines with strict adherence to SOPs.

### EDUCATION:

#### **University of Idaho, Moscow, ID 2005-09**

B.S. Microbiology

## Sherry Maine

### PROFESSIONAL EXPERIENCE:

#### **SVL Analytical, Inc. - Kellogg, ID Sept. 2011 - Present**

Safety/Hazmat Officer - Responsible for revising the Chemical Hygiene Plan annually, conducts safety training and oversees response teams. Other duties include providing accident reports to the state and overseeing SVL's hazardous waste program (including setting up 8-hour refresher courses annually).

#### **SVL Analytical, Inc. - Kellogg, ID Nov. 2005 - Present**

Classical Chemistry Department Chemist—Analyzes and interprets soil and aqueous samples for: Total and ortho phosphorous, COD, TOC/TN, sulfide, MBAS, ammonia, nitrate/nitrite, TKN, hexavalent chromium, TOM, LOI and gravimetric silica.

#### **UNR-Chem. - Reno, NV Aug. 2001 - June 2004**

She synthesized and analyzed compounds to determine their chemical structure. She also tested soils and water for inorganic analysis.

#### **Nestle/Simplot - Nampa, ID April 1999 - July 2001**

Quality Assurance Technician – Tested and evaluated product throughout entire course of production.

#### **ESI - Grandview, ID Dec. 1995 - Aug. 1997**

Hazardous Waste Technician - Identified incoming hazardous waste samples (GC and ICP technician). Assisted in the development of formulas to stabilize hazardous waste in accordance with federal standards.

### EDUCATION:

#### **University Of Nevada - Reno, NV 2004**

M.S. Chemistry

#### **Northwest Nazarene College - Nampa, ID 1995**

B.S. Chemistry

#### **Southern Nazarene University - Bethany OK 1986-1989**

Took classes towards a nursing degree.



## **CRYSTAL SEVY**

### **PROFESSIONAL EXPERIENCE:**

**SVL Analytical, Inc. - Kellogg, ID**

**2006-Present**

Sample Receiving Department Supervisor— Supervises SVL's sample receiving staff and is Sample Custodian for samples received under EPA SOW ILMO5.4. Responsible for setting up Work Orders within Element (LIMS), case narratives and point of contact with clients and their representatives. Works closely with SVL's Client Services and Technical Director to ensure that projects are setup and priced correctly.

**SVL Analytical, Inc. - Kellogg, ID**

**1996-2006**

Sample Receiver—Verifies sample temperature, integrity and security on receipt; creates laboratory jobs; ensures proper sample storage prior to analysis supervises sample disposal; ships sample containers to clients.

## **MELBA BENCICH**

### **PROFESSIONAL EXPERIENCE:**

#### **SVL Analytical, Inc. - Kellogg, ID, February 1988 - Present**

Document Control Manager – Supervises data reporting using Element (LIMS) for commercial clients and SDG reporting for EPA's CLP SOW ILMO5.4.

#### **Shoshone Insurance – Kellogg, ID, 1984 – 1988**

Duties included accounting, customer service relations and updating manuals

#### **Travel People – Coeur d' Alene, ID, 1982 – 1984**

Travel Consultant

#### **Farmer's Insurance – Kellogg, ID 1982-1984**

Duties included accounting, customer service relations and updating manuals

#### **The Bunker Hill Company – Kellogg, ID, 1974 – 1981**

Data Control Analyst

### **EDUCATION:**

#### **North Idaho College – Coeur d' Alene, ID, 1967 – 1968**

General studies

#### **International Correspondence School, 1980**

Mathematics

## 29.0 QUALITY MANUAL RELEASES

<b>Date</b>
January 2010
January 2011
February 2012
February 2013
February 2014
January 2015

Page 72 is the last page in this document

**DETERMINATION OF MERCURY (CVAA)**  
**By EPA 245.1, 7470A and 7471B**

**Revised by: Michael Desmarais**

**Approved by: \_\_\_\_\_ Date: \_\_\_\_\_**  
**Mercury Department Supervisor**

**Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_**  
**Quality Assurance Manager**

DETERMINATION OF MERCURY (CVAA) by EPA 245.1, 7470A and 7471B

SVL 4010 Version 19.1

Effective Date: 07/01/2016

Page 2 of 33

I have read, understood and will comply with SOP (SVL 4010 Version 19.1)

Print Name	Signature	Date
<u>Steven Frederick</u>	_____	_____
<u>Sophie Milam</u>	_____	_____
<u>Heidi Barnes</u>	_____	_____
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## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to describe the procedure used to analyze samples for mercury, including the operation of a CETAC Mercury Analyzer. The procedure measures total mercury (organic and inorganic) in water, soil, sediment, bottom deposits, and sludge. SVL uses a Laboratory Management Information System (LIMS) – Element, to manage client's samples. In Element the current aqueous MDL is 0.04 µg/L and the soil MDL is 0.0053 mg/kg. The aqueous reporting limit is 0.2 µg/L and the soil reporting limit is 0.033 mg/kg. Definitions for words used in this SOP may be found in SVL's Quality Manual. The holding time is 28 days when aqueous samples are preserved with HNO<sub>3</sub> to pH <2, aqueous samples are stored at ambient temperature and solid samples are stored between 0-6 °C.

## **2.0 SUMMARY OF METHOD**

This SOP is intended to satisfy the requirements of EPA methods 245.1, 7470A, and 7471B. Samples must be digested prior to analysis. Organic mercury compounds are oxidized by a mixture of potassium permanganate and potassium persulfate. Then the mercury is reduced to the elemental state by stannous chloride and aerated from solution in a closed system. Sample analysis is performed by a CETAC mercury analyzer. The mercury vapor passes through a cell positioned in the light path of the CETAC. Concentration is measured as a function of absorbance by mercury atoms at 253.7 nm.

## **3.0 INTERFERENCES**

**3.1** Very high levels of chloride interfere. During the oxidation step chloride is converted to free chlorine. Free chlorine absorbs at the same wavelength as mercury vapor. An excess of hydroxylamine reagent will remove the chlorine.

## **4.0 SAFETY**

**4.1** Hydrochloric acid can cause severe burns if it comes into contact with skin or eyes. The fumes are also irritating to nasal and lung tissues. Work with hydrochloric acid in a hood. Wear safety glasses or goggles. Wear gloves and a lab coat or an apron. In the case of exposure, flush with water for at least fifteen minutes.

- 4.2** Sulfuric acid can cause severe burns if it comes into contact with skin or eyes. Wear safety glasses or goggles. Wear gloves and a lab coat or an apron. In the case of exposure, flush with water for at least fifteen minutes.
- 4.3** Nitric acid is a strong oxidizer and can cause severe burns if it comes into contact with skin or eyes. The fumes are also irritating to nasal and lung tissues. Work with Nitric Acid in a hood. Wear safety glasses or goggles. Wear gloves and a lab coat or an apron. In the case of exposure, flush with water for at least fifteen minutes.
- 4.4** Mercury is a toxic heavy metal. Do not swallow, inhale or absorb through skin.
- 4.5** Read the MSDSs for the chemicals used in this SOP. Be aware of the possible hazards.
- 4.6** Addition of acids to some samples may release toxic gases such as hydrogen cyanide or hydrogen sulfide. Add acids under a hood.

## **5.0 EQUIPMENT, INSTRUMENTATION AND MATERIALS**

Equivalent equipment, instrumentation and materials may be used.

- 5.1** CETAC M6100 Mercury Analyzer
- 5.2** CETAC M7500 Mercury Analyzer
- 5.3** ASX – 510 Autosampler
- 5.4** ASX520 AUTOSAMPLER
- 5.5** Nafion Dryer, CETAC
- 5.6** 254 nm Mercury Lamp, BHK, Inc. Model No. 80-8024-01
- 5.7** Argon, 99.99% pure, or better
- 5.8** Volumetric flasks, 50 mL, 100 mL, 200 mL, 1 L
- 5.9** Micropipets, Corning, Lambda, Wheaton, Socorex
- 5.10** “Snap cap” vials, 4 oz., with 100-mL fill line, Fisher Catalog No. 14377807



- 5.11 Block digestors, capable of maintaining a temperature of 90 to 95 degrees, Environmental Express
- 5.12 Balance, capable of weighing to the nearest 0.01 gram
- 5.13 pH strips, 0 – 6 pH range, EM colorpHast, Fisher M95863
- 5.14 Sieves and splitters, project specific, see SOP 2018

## **6.0 REAGENTS AND STANDARDS**

Guidelines for the storage, tracking and expiration of chemicals and reagents can be found in SOP SVL 1032. The procedure for purchasing chemicals and reagents can be found in SOP SVL 1015. Any exceptions to the above mentioned SOPs will be found in this section: as well as, all of the preparatory steps needed to construct or prepare reagents, and standards. Equivalent reagents or standards may be used.

- 6.1 Concentrated sulfuric acid ( $H_2SO_4$ ): Fisher TraceMetals Grade
- 6.2 Concentrated nitric acid ( $HNO_3$ ): Fisher TraceMetals Grade
- 6.3 Concentrated hydrochloric acid (HCl): Fisher TraceMetals Grade
- 6.4 Potassium permanganate: Fisher Certified ACS/Hg Determination
- 6.5 Potassium persulfate: J.T. Baker Instra-Analyzed
- 6.6 Stannous chloride ( $SnCl \cdot 2H_2O$ ): CCI GCS Grade
- 6.7 Hydroxylamine hydrochloride, Fisher Certified ACS/Hg Determination
- 6.8 Sodium chloride, CCI ACS Grade Crystals
- 6.9 Potassium dichromate, MCB Reagent, crystals
- 6.10 Hydroxylamine hydrochloride solution: Dissolve 300 g of sodium chloride (6.8) and 300 g of hydroxylamine hydrochloride (6.7) in deionized water (6.21). Dilute to 2.5 L with deionized water. Mix well. Expiration date is six months after date prepared.
- 6.11 Potassium permanganate, 5% solution: Dissolve 125 g of potassium permanganate (6.4) in 2.5 L of deionized water (6.21). Mix well. Expiration date is six months after date prepared.

- 6.12** Potassium persulfate, 5% solution: Dissolve 125 g of potassium persulfate (6.5) in 2,500 mL of deionized water (6.21). Mix well. Expiration date six months after date prepared.
- 6.13** Aqua regia: Prepare immediately before use by carefully adding three volumes of concentrated hydrochloric acid (6.3) to one volume of concentrated nitric acid (6.2). Mix well. Prepare 5 mL for each “Snap-cap” vial that will be used.
- 6.14** Stannous chloride solution, 10% solution: Prepare according to the CETAC instrument manufacturer’s instructions by dissolving 400 g of SnCl<sub>2</sub>·2H<sub>2</sub>O (6.6) in 280 mL concentrated hydrochloric acid (6.3) and allow to sit overnight. Dilute to 4 L with deionized water (6.21). Expiration date six months after date prepared.
- 6.15** Rinse solution: Solution of 5.2% hydrochloric acid (6.3) and 2% nitric acid (6.2). For a 2.5 L bottle combine 130 mL HCl and 50 mL HNO<sub>3</sub> and bulk with DI water.
- 6.16** Stock mercury solution, 1000 µg/mL mercury: Commercially-prepared High Purity Standards.
- 6.17** Stock mercury solution, 100 µg/mL mercury: Commercially-prepared High Purity Standards.
- 6.18** Intermediate standard solution, 10 µg/mL mercury: Transfer 1.00 mL of 1000 µg/mL stock mercury solution (6.16) to a 100 mL volumetric flask. Add 2 mL concentrated nitric acid (6.2). Dilute to the mark with deionized water (6.21). Mix well. Prepare each day of use.
- 6.19** Working standard solution, 100 µg/L mercury: Transfer 2.00 mL of Intermediate standard solution (6.18) to a 200 mL volumetric flask. Add 2 mL concentrated nitric acid (6.2). Dilute to the mark with deionized water (6.21). Mix well. Prepare each day of use.
- 6.20** Mercury ICV stock solution: Transfer about 50 mL deionized water (6.21) to a 100 mL volumetric flask. Add 10 mL concentrated nitric acid (6.2). Then dissolve 0.05 g potassium dichromate (6.9) in the solution. Add 0.50 mL of the 100 µg/mL Hg single-element stock solution (6.17). Dilute to the mark with deionized water (6.21) and mix well. The final concentration of the mercury solution is 500 µg/L. Expiration date is six months after date prepared, but no later than the expiration date of the stock solution.
- 6.21** ASTM Type II deionized water.

**6.22** Sand, Fisher S23-3.

## **7.0 INSTRUMENT SETTINGS**

**7.1** Verify that the power is on for both the autosampler and the mercury lamp.

**7.2** Fill the reagent reservoir with stannous chloride solution (6.14).

**7.3** Fill the rinse reservoir with rinse solution (6.15).

## **8.0 CALIBRATION**

**8.1** Verify the volume of the “Snap-cap” vials.

**8.1.1** Select three vials at random from each lot.

**8.1.2** Place each vial on the balance. Tare the weight.

**8.1.3** Fill the vials to the 50 mL fill line with deionized water (6.21).

**8.1.4** Weigh the full vials. Record the weights in the Soil Digest logbook.

**8.1.5** All three of the net weights should fall within the range 48.50 to 51.50 g.

**8.1.6** If any of the net weights fall outside the range 48.50 to 51.50 g, return the entire box of vials to the manufacturer for replacement.

**8.1.7** Repeat the test with three vials filled to the 100 mL fill line with deionized water.

**8.1.8** All of the net weights should fall within the range 97.0 to 103.0 g.

**8.1.9** If any of the net weights fall outside the range 97.0 to 103.0 g, return the entire lot of vials to the manufacturer for replacement.

**8.2** For aqueous samples, prepare calibration standards by transferring the following amounts of working standard solution (6.19) into “Snap-cap” vials. Dilute to the 50 mL mark with deionized water (6.21) Mix well. Follow the steps outlined in 10.1.10 thru 10.1.15.

<u>Standard Conc.</u>	<u>Volume of Working Standard</u>	<u>Final Volume</u>
0.0 µg/L	0.00 mL	50 mL
0.2 µg/L	0.10 mL	50 mL
0.5 µg/L	0.25 mL	50 mL
1.00 µg/L	0.50 mL	50 mL
2.00 µg/L	1.00 mL	50 mL
5.00 µg/L	2.50 mL	50 mL
10.0 µg/L	5.00 mL	50 mL

- 8.3** For soil/sludge samples, prepare calibration standards by transferring the following amounts of working standard solution (6.19) into “Snap-cap” vials. Add enough deionized water (6.21) to make an initial volume of 10 mL. This initial volume will be brought up to a final volume of 100 mL (the concentrations below are based upon the 100 mL final volume. Follow the steps outlined in 10.2.1 thru 10.2.14.

<u>Standard Conc.</u>	<u>Volume of Working Standard</u>	<u>Initial Volume</u>
0.0 µg/mL	0.00 mL	10 mL
0.2 µg/mL	0.20 mL	10 mL
0.5 µg/mL	0.50 mL	10 mL
1.0 µg/mL	1.00 mL	10 mL
2.0 µg/mL	2.00 mL	10 mL
5.0 µg/mL	5.00 mL	10 mL
10.0 µg/mL	10.0 mL	10 mL

## **9.0 SAMPLE HANDLING AND PRESERVATION**

- 9.1** Samples for total metals should have been preserved by acidification with nitric acid to a pH of 2 or lower immediately upon collection in the field or upon being accepted by Sample Receiving as per SOP SVL 2001.
- 9.2** Refrigerate solid samples between 0-6 °C until analysis is completed.
- 9.3** Ask the supervisor for the client instructions on sample preparation. Prepare the sample in accordance with those instructions. Record the instructions in the Mercury Prep Logbook. Some possible instructions are:
- 9.3.1** Do not dry the sample. Determine percent moisture on a separate portion according to SOP 4022.
- 9.3.2** Dry the sample at ambient temperature. Sieve the sample through a 10-mesh screen. Divide the sample into two parts. Archive one portion for future analysis. Sieve the second portion through an 80-

mesh screen. Digest the – 80 mesh fraction.

**9.3.3** Dry the sample at ambient temperature. Sieve the sample through an 80-mesh screen. Digest the – 80 mesh fraction.

**9.4** If the sample contains a noticeable amount of liquid, ask the supervisor for instructions. Prepare the sample in accordance with those instructions. Record the decision in the Mercury Prep Logbook and in a Work Order memo. Some possible instructions are:

**9.4.1** Analyze the entire sample, homogenizing the solids and the liquid.

**9.4.2** Decant the liquid from the sample. Analyze the solid fraction.

**9.4.3** Decant the liquid. Analyze the liquid and the solids as two different samples.

**10.0 SAMPLE PREPARATION AND ANALYSIS**

**10.1** Preparation of aqueous samples for method 245.1 and 7470A see table 10.1 for variations for different aqueous methods.

	T&D 245.1	T&D 7470A	MWM 245.1	SPLP, TCLP, CWET 7470A	Level 3
Extract blank provided for additional blank sample	N	N	Y	Y	N
Extract blank used in LCS	N	N	Y	Y	N
Matrix spike sample supplied	N	N	N	Y	N
Second matrix spike required if more than 10 samples in batch	Y	N	Y	N	N
Duplicate of sample used for matrix spike required	N	N	N	N	Y

**Table 10.1: procedural differences between aqueous methods**

- 10.1.1** Prepare a method blank for every 20 samples by transferring 50 mL of deionized water (6.21) into a “Snap-cap” vial. If batch is an extract change the name of the second blank on the benchsheet to EB-“work order number” (i.e. EB-W600001) using the work order on the extract bottle.
- 10.1.2** Prepare an LCS for every 20 samples. Transfer 0.5 mL of the mercury ICV stock solution (6.20) to a “Snap-cap” vial. Dilute to the 50 mL mark with deionized water (6.21). The LCS concentration is 5.0 µg/L. Designate the spike ID for the LCS on the benchsheet and if the batch is an extract change the name of the LCS on the benchsheet to LCS-“work order number” (i.e. LCS-W600001) using the work order on the extract bottle.
- 10.1.3** Mix the sample well to ensure that it is homogenous. Transfer 50 mL of each sample to a “Snap-cap” vial. If the history of the sample indicates that this volume may contain more than 1.0 µg mercury, use of a smaller volume diluted to 50 mL is permitted. A smaller volume may also be used if the client has provided insufficient sample but the initial and final volumes on the benchsheet must be adjusted (samples should be qualified; ref, SOP SVL 2009).
- 10.1.4** Prepare a matrix spike by transferring an additional 50 mL aliquot of sample to a “Snap-cap” vial and adding 0.50 mL of working standard solution (6.19). On the benchsheet adjust final volume to 50.5 mL and designate the source sample and spike ID. The expected concentration of the spike added is 1.0 µg/L.
- 10.1.5** Prepare a matrix spike duplicate by following the steps outlined in 10.1.4.
- 10.1.6** Prepare an ICV. Transfer 0.5 mL of the mercury ICV stock solution (6.20) into a “Snap-cap” vial. Dilute to the 50 mL mark with deionized water (6.21). The ICV concentration is 5.0 µg/L.
- 10.1.7** Prepare an ICB and a CCB by transferring 50 mL of deionized water (6.21) into a “Snap-cap” vial.
- 10.1.8** Prepare a CCV by transferring 1.0 mL of the working standard solution (6.19) to a “Snap-cap” vial. Dilute to the 50 mL mark with deionized water (6.21) and mix well. The final concentration of the CCV is 2.0 µg/L.

- 10.1.9** Prepare a RLCS by transferring 0.1 mL of the working standard solution (6.19) to a “Snap cap” vial. Dilute to the 50 mL mark with deionized water (6.21) and mix well. The final concentration of the RLCS is 0.2 µg/L.

To all standards (calibration and QC) and samples do the following:

- 10.1.10** Add 2.5 mL concentrated sulfuric acid (6.1) to each vial and mix.
- 10.1.11** Add 1.25 mL concentrated nitric acid (6.2) to each vial and mix.
- 10.1.12** Add 7.5 mL potassium permanganate solution (6.11) to each vial. For sewage samples, or samples with high organic content, additional permanganate may be required. Shake and add additional portions of potassium permanganate solution, if the purple color does not persist for at least 15 minutes reduce the concentration of the original sample by dilution (dilute with deionized water (6.21)) and repeat 10.1.9 thru 10.1.11 on 50 mL of the diluted sample.
- 10.1.13** Add 4.0 mL potassium persulfate solution (6.12) to each vial.
- 10.1.14** Heat the vials in a block digester for 2 hours at 95°C.
- 10.1.15** Cool the vials and add 5.0 mL of hydroxylamine hydrochloride (6.10) to reduce the excess permanganate.

**10.2** Preparation of soil/sludge samples for method 7471A.

- 10.2.1** Prepare a method blank by weighing 0.60 g ± 0.005 g of sand (6.22) add 5.0 mL of deionized water (6.21) into a “Snap-cap” vial. Mix well.
- 10.2.2** Prepare an LCS by transferring 1.0 mL of the mercury ICV stock solution (6.20) to 0.60 g ± 0.005 g of sand (6.22) in a “Snap-cap” vial, add 4.0 mL deionized water (6.21). Mix well. The concentration of the LCS is 5.0 µg/L.
- 10.2.3** Weigh out a 0.60 g ± 0.005 g portion of a well homogenized sample in a “Snap-cap” vial. Add 5.0 mL deionized water (6.21).
- 10.2.4** Prepare a matrix spike by weighing another 0.60 g ± 0.005 g portion of a well homogenized sample in a “Snap-cap” vial and add 2.0 mL of the working standard solution (6.19). Add 3.0 mL deionized water (6.21). The initial concentration of the matrix

spike is 2.0 ppb.

- 10.2.5** Prepare a matrix spike duplicate. Follow the process outlined in 10.2.4.
- 10.2.6** Prepare an ICV by transferring 1.0 mL of the mercury ICV stock solution (6.20) into a “Snap-cap” vial. Add 9.0 mL of deionized water (6.21). The final concentration of the ICV is 5.0 µg/L
- 10.2.7** Prepare an ICB and a CCB by transferring 10.0 mL of deionized water (6.21) into a “Snap-cap” vial.
- 10.2.8** Prepare a CCV by transferring 2.0 mL of the working standard solution (6.19) into a “Snap-cap” vial. Add 8.0 mL of deionized water (6.21). The final concentration of the CCV is 2.0 µg/L
- 10.2.9** Prepare an RLCS by transferring 0.2 mL of the working standard solution (6.19) into a “Snap-cap” vial. Add 9.8 mL of deionized water (6.21). The initial concentration of the RLCS is 0.2 µg/L.

To all standards (calibration and QC) and samples do the following:

- 10.2.10** Add 5.0 mL of aqua regia (6.13) into each “Snap-cap” vial.
- 10.2.11** Heat the vials in a block digester for two minutes at  $95 \pm 3^\circ\text{C}$ .
- 10.2.12** Cool the vials and add 40 mL deionized water (6.21).
- 10.2.13** Add 15 mL potassium permanganate solution (6.11) into each “Snap-cap” vial. Mix thoroughly, and then wait 15 minutes. Add additional portions of permanganate solution, if needed to standards and blanks until the purple color persists for at least 15 minutes. If more potassium permanganate is added ensure that equal amounts of permanganate are also added to standards and blanks. Place samples or standards into the block digester for 30 minutes at  $95 \pm 3^\circ\text{C}$ .
- 10.2.14** Cool the vials and add 6 mL of hydroxylamine hydrochloride solution (6.9) to reduce the excess permanganate. Add 49 mL deionized water (6.21) into samples, prep blank, LCS, matrix spike and matrix spike duplicate. Add 44 mL deionized water to the calibration standards, ICB, CCBs, RLCS, ICV, and CCVs. Add proportionally less water if additional permanganate is used. The final volume of all standards and samples should be 120 mL.



### **10.3 Operation of the CETAC M6100 Mercury Analyzer**

- 10.3.1** Turn the computer on and double-click the “QuickTrace™” icon. The “M6100A Mercury Analyzer” window will appear.
- 10.3.2** Turn on lamp and connect the pump tubing.
- 10.3.3** Put the stannous sipper in deionized water and the acid rinse tube in the acid rinse and turn the pump on.
- 10.3.4** Turn on the argon gas valve, and set the psi to 35.
- 10.3.5** Pinch the Nafion dryer waste line and allow the waste solution to coat the post in the gas/liquid separator.
- 10.3.6** Stop pinching the line and allow liquid to flow through the waste line. Connect the “Hg Vapor” tubing to the gas/liquid separator. Switch the stannous sipper from water to the stannous bottle. Allow the system to equilibrate for about 15 minutes.
- 10.3.7** Click the down arrow on the “load” button. Select the “new from” option and the “create a new worksheet from template worksheet” window will appear.
- 10.3.8** Click the “browse” button for the template worksheet field. Navigate to the QuickTrace™ templates folder, and select M6100.
- 10.3.9** Choose the appropriate starter template for the analysis. Use the “245.1START2” files to attain proper sequence information.
- 10.3.10** In the new worksheet “name field”, enter the date in the format yymmdd followed by “01” for the first run of the day, “02” for the second run, and so on. This is the file name.
- 10.3.11** Ensure that the “retain sequence information” box is checked, and then click Ok.
- 10.3.13** Highlight lines 9 and 10, right click, and choose insert rows. Right click anywhere on the sequence and select “auto renumber tubes.” Enter ICB for the sample name on line 9, and enter RLCS for the sample name on line 10. Left click the dropdown arrow in the “type” column next to ICB and RLCS. Change the sample types to “Initial Calibration Blank” and “Contract Required Detection limit” respectively.

- 10.3.14** Beginning on line 13 (tube 1:3) enter the SVL sample names to be analyzed.
- 10.3.15.** If analyzing a sample dilution, type the “@” sign followed by the dilution factor (e.g. @10X) immediately after the SVL sample number (in the same column).
- 10.3.16** A CCV and CCB is required per every 10 samples, double check the sequence to ensure that there is a CCV and CCB after every 10 samples.
- 10.3.17** Scroll to the end of the sequence and ensure that there is a final CCV and CCB, if there is not then add them by typing in CCV and CCB for the sample names, change their tube numbers to S:9 for CCV, and S:10 for CCB. Left click in the “type” field next to the CCV, click the dropdown arrow and select “Continuing Calibration Verification.” Left click in the “type” field next to the CCB, click the dropdown arrow and select “Continuing Calibration Blank.”
- 10.3.18** Thoroughly check the “Sample Label” column for errors. Click the “Save” icon in the upper left of the QuickTrace™ window to save your worksheet.
- 10.3.19** Pour the calibration standards, QC standards and samples in the appropriate vials. The autosampler racks are numbered 1-2, from left to right and tubes are numbered from back to front, left to right for each rack (1:1, 2:1, etc.). The standard rack at the back of the autosampler tray is called “S,” with positions 1-10. Pour the calibration standards in S:1-S:7. Pour the ICV in S:8, the CCV in S:9, and the CCB in S:10. Pour the ICB into tube 1:1, and the RLCS in 1:2.
- 10.3.20** Click the “method editor” icon.
- 10.3.21** Click on the “Conditions” button.
- 10.3.22** Click on the “Read a Sample” button. The “Now Zeroing the Mercury Analyzer” window will open.
- 10.3.23** When the zeroing process is finished, the “Select Tube” window will open. Click tube number S:7.
- 10.3.24** Click “OK.” Standard 7 will be analyzed and the absorbance-versus-time will be displayed on the screen.

- 10.3.25** Set the Time Profile by moving the green and red bars to the flattest portion at the top of the signal peak.
- 10.3.26** Click “Save” from the file menu. Close the Method Editor window.
- 10.3.27** Click on the “GO” icon. The “Please set the gas flow to 35 psi” dialogue box will open, ensure the psi is at 35, and then click OK.
- 10.3.28** The instrument will begin analyzing the samples.
- 10.3.29** Click on the “Analysis” icon to see the calibration curve, run progress and sample absorbance displayed.
- 10.3.30** Analysis will stop if a calibration standard or QC standard fails. ICV, ICB, RLCS, CCV, and CCB are allowed to be run a second time (see section 13).
- 10.3.30.1** Once the Calibration standards are analyzed open the Excel sheet “Calibration Curve Check” and click the “enable Macros” button.
- 10.3.30.2** Select “M6100” for the instrument, “Mercury” for the analyte, and press CTRL+; for the date.
- 10.3.30.3** Transfer the r, b, and m values from the QuickTrace™ display into the appropriate cells on the Calibration Curve Check1 worksheet.
- 10.3.30.4** Click the “Update Standard Values” button and fill in the concentrations from the QuickTrace™ analysis.
- 10.3.30.5** When you save the worksheet it will automatically assign the name of the document from the date from the input box simply add the run number to the end.
- 10.3.31** Check the run to ensure that the prep blanks and LCSs meet method criteria. If the sample absorbance display indicates a failure, DO NOT STOP THE ANALYSIS until the analysis has registered.
- 10.3.31.1** All instrument reads must be allowed to go to completion, the Cetac allows for stopping an analysis prior to the final read, this is not allowed. All failures must be re-inserted at a later point in the run.

- 10.3.32** Under 245.1 if the concentration in a sample exceeds 90% of the linear dynamic range, the sample must be re-analyzed at a lower dilution. 7471B requires that samples that exceed the calibration range be diluted back to within the calibration range.
- 10.3.33** Analysis must be stopped to add additional samples or diluted samples to the end of the run. Ensure that there is a CCV and CCB every 10 samples.
- 10.3.34** Save the sequence.
- 10.3.35** Click “GO” to restart the run.
- 10.3.36** Re-running a sequence if Calibration, ICV, ICB, RLCS, CCV, or CCB fail.
- 10.3.36.1** Close the existing sequence. In File menu choose “Close”
- 10.3.36.2** Click the down arrow on the “Load” icon.
- 10.3.36.3** Click “New From.” The “Create A New Worksheet From Template Worksheet” window will open.
- 10.3.36.4** Click the top “Browse” button. The “New Worksheet” window will open.
- 10.3.36.5** On the “Look in” menu, choose “QuickTrace™” and then choose “worksheets.” Choose the file name desired.
- 10.3.36.6** Change the number at the end of the file name.
- 10.3.36.7** Delete any unneeded CCV/CCBs and non-usable data.
- 10.3.36.8** Set the Time Profile and start the run.
- 10.3.37** After the final CCV/CCB of the run move the stannous sipper to a 20% nitric acid solution and allow to rinse for 5-10 minutes, after that move both the stannous sipper and the acid rinse hose to containers of DI water and allow to rinse the system for 10 minutes. Once rinsed with water position the stannous sipper and the acid rinse hose to hang freely in the air for 15 minutes to dry the system. Once all visible hoses are dry, exit the QuickTrace™ software and remove the nafion dryer hose from the GL

separator, shut off the gas, turn off the peristaltic pump and remove the pump tubing.

#### **10.4 Operation of the CETAC M7500 Mercury Analyzer**

**10.4.1** Turn the computer on and double-click the “QuickTrace™” icon. The “CECTA QuickTrace™” window will appear.

**10.4.2** When the “Confirm” window appears, click the “No” button. The “Sequence” page will appear.

**10.4.3** Connect the pump tubing and insert the sipper tube into the Stannous Chloride bottle.

**10.4.4** Open the Argon gas valve.

**10.4.5** Click on the “Instrument” icon. The “Instrument” window will appear.

**10.4.6** Click on the “Analyzer” button.

**10.4.7** In the “Pump” section, enter 50. Click the “Set Speed” button, then click the “On” button.

**10.4.8** In the “Lamp” section, click the “Status” button to determine if the lamp is on. If the lamp is off, click the “Lamp On” button.

**10.4.9** In the “Gas” section, enter 150 and click the “Set Gas” button.

**10.4.10** Close the “Instrument” window.

**10.4.11** Click the “Gas/Liquid Separator” icon. Pinch the Nafion Dryer waste line and allow the waste solution to coat the post in the gas/liquid separator.

**10.4.12** Stop pinching the line and allow liquid to flow through the waste line. Connect the “Hg Vapor” tubing to the gas/liquid separator. Open the “Instrument” window and reset the pump speed. Allow the system to equilibrate for about 15 minutes.

**10.4.13** Click the down arrow on the “Load” icon. Click on “New Form”. The “Create A New Worksheet From Template Worksheet” window will open.

- 10.4.14** Click the top “Browse” button. The “New Worksheet” window will open.
- 10.4.15** Double click on “M7500”. Double click on one of the following: 245.1 START, 7470 START, 7471 STARTER, CLP STARTER.
- 10.4.16** In the “Enter new worksheet name” box, enter the date in the format mm.dd.yy with the letter A (letter B for the second run, letter C for the 3<sup>rd</sup> run, etc.) This is the file name for the run.
- 10.4.17** Click “OK” and the “Sequence” window will appear with the Calibrations and QC Standards entered.
- 10.4.18** Click on the “Sequence Editor” icon. Click on the “Sequence” button.
- 10.4.19** In the “Sample count” space, enter the number of samples to be included in the run. Do not count calibrations standards, ICV/ICB, RLCS, or CCV/CCB.
- 10.4.20** Check the “Begin with calibration” box. Click “Generate Sequence.”
- 10.4.21** The “Sequence” window will appear. On line 10 (CRDL Standard) type in RLCS.
- 10.4.22** Highlight lines 14 and 15 (Duplicate and Matrix Spike). Right click and choose “Delete Selected row(s).”
- 10.4.23** Highlight lines 13, 14 and 15. Right click and choose “Insert Row.”
- 10.4.24** Right click on any line and choose “Auto-Renumber Tubes.” Now the sequence is set for 10 samples between every “CCV/CCB.”
- 10.4.25** Starting on line 13, tube 1:1, in the “Sample Label” column type in the SVL sample numbers for the samples to be analyzed.
- 10.4.26** If analyzing a sample dilution, type the “@” symbol and then the dilution factor (e.g. @10X) immediately after the sample number.
- 10.4.27** Thoroughly check the “Sample Label” column for errors. To make corrections to the sample labels click on the line number. Right click and select the appropriate action.

- 10.4.28** After making any corrections, click on any box then right click and choose "Auto-Renumber Tubes." Make sure there are only 10 samples between each "CCV/CCB."
- 10.4.29** At the end of the sequence there will be 3 extra samples. Click on the line number for each of them. Right click and choose "Delete Selected Row(s)."
- 10.4.30** Save the sequence by clicking the "Save" icon or clicking "File" and choosing "Save."
- 10.4.31** Pour the calibration standards, QC standards and samples in the appropriate vials. The autosampler racks are numbered 1-4, from left to right and tube are numbered from back to front, left to right for each rack (1:1, 2:1, etc.). The standard rack at the back of the autosampler tray is called "S", with positions 1-10. The QC Standards are in rack 4 (ICV = 4:1, ICB = 4:2, RLCS = 4:3, CCV = 4:4 and CCB = 4:5).
- 10.4.32** Click on the "Method Editor" icon.
- 10.4.33** Click on the "Conditions" button.
- 10.4.34** Click on the "Read a Sample" button. The "Now Zeroing the Mercury Analyzer" window will open.
- 10.4.35** When the zeroing process is finished, the "Select Tube" window will open. Click tube number S:7.
- 10.4.36** Click "OK". Standard 7 will be analyzed and the absorbance-versus-time will be displayed on the screen.
- 10.4.37** Set the Time Profile by moving the green and red bars to the flattest portion at the top of the signal peak.
- 10.4.38** Click "Save" from the file menu.
- 10.4.39** Click on the "GO" icon. Click "OK" on the "Confirm" window.
- 10.4.40** The instrument will begin analyzing the samples.
- 10.4.41** Click on the "Analysis" icon to see the calibration curve, run progress and sample absorbance displayed.

**10.4.42** Analysis will stop if a calibration standard or QC standard fails. The ICV, ICB, RLCS, CCV AND CCB are allowed to be run a second time after a failure (see section 13).

**10.4.42.1** Once the Calibration standards are analyzed open the Excel sheet "Calibration Curve Check" and click the "enable Macros" button.

**10.4.42.2** Select "M6100" for the instrument, "Mercury" for the analyte, and press CTRL+; for the date.

**10.4.42.3** Transfer the r, b, and m values from the QuickTrace™ display into the appropriate cells on the Calibration Curve Check1 worksheet.

**10.4.42.4** Click the "Update Standard Values" button and fill in the concentrations from the QuickTrace™ analysis.

**10.4.42.5** When you save the worksheet it will automatically assign the name of the document from the date from the input box simply add the run number to the end.

**10.4.42.6** If batch is Level 3 print this page to turn in with the benchsheet.

**10.4.43** Check the run to ensure that the prep blanks and LCSs meet method criteria. If the sample absorbance display indicates a failure, DO NOT STOP THE ANALYSIS until the analysis has registered.

**10.4.43.1** All analyses are to finish, the Cetac allows for stopping an analysis prior to the final read, this is not allowed. All failures must be re-inserted at a later point in the run.

**10.4.44** Under 245.1 if the concentration in a sample exceeds 90% of the linear dynamic range, the sample must be re-analyzed at a lower dilution. 7471B requires that samples that exceed the calibration range be diluted back to within the calibration range.

**10.4.45** Add additional samples or diluted samples to the end of the run.

**10.4.45.1** Click on "Sequence Editor" icon.



- 10.4.45.2** Enter the number of samples to be added in the "Sample count" box.
  - 10.4.45.3** Uncheck "Begin with Calibration."
  - 10.4.45.4** Click "Generate Sequence."
  - 10.4.45.5** A "Warning" window will open. Click "OK."
  - 10.4.45.6** Delete the CCV/CCB before the added samples.
  - 10.4.45.7** Delete the duplicates and matrix.
  - 10.4.45.8** Add 2 lines. Now the sequence is set for 10 samples between each CCV/CCB.
  - 10.4.45.9** Entered the additional sample and/or diluted sample numbers.
  - 10.4.45.10** Save the sequence.
  - 10.4.45.11** Click on "GO" to restart the run.
- 10.4.46** How to Re-run a sequence if Calibration, ICV, ICV, RLCS, CCV OR CCB fail.
- 10.4.46.1** Close existing sequence. In File menu choose "Close."
  - 10.4.46.2** Click the down arrow on the "Load" icon.
  - 10.4.46.3** Click "New From." The "Create A New Worksheet From Template Worksheet" window will open.
  - 10.4.46.4** Click the top "Browse" button. The "New Worksheet" window will open.
  - 10.4.46.5** On the "Look in" menu, choose "Quick Trace."
  - 10.4.46.6** Choose "worksheets."
  - 10.4.46.7** Choose the file name desired.
  - 10.4.46.8** Change the letter at the end of the file name.

**10.4.46.9** Delete any unneeded CCV/CCBs and no-usable data.

**10.4.46.10** Set the Time Profile and start the run.

**10.4.47** After the final CCV CCB of the run move the stannous sipper to a 20% nitric acid solution and allow to rinse for 5-10 minutes, after that move both the stannous sipper and the acid rinse hose to containers of DI water and allow to rinse the system for 10 minutes. Once rinsed with water position the stannous sipper and the acid rinse hose to hang freely in the air for 15 minutes to dry the system. Once all visible hoses are dry, exit the QuickTrace™ software and remove the nafion dryer hose from the GL separator, shut off the gas, turn off the peristaltic pump and remove the pump tubing.

## **11.0 DATA REDUCTION**

**11.1** The instrument software will generate a linear calibration curve of absorbance versus concentration. The correlation coefficient must be 0.995 or greater. If the correlation coefficient is less than 0.995, recalibrate the instrument prior to analyzing samples. The software will calculate mercury concentrations based on the curve. The analyst must correct for any sample dilution factors manually.

**11.2** Use the calibration verification template (located at H:\Templates\Calibration Curve Check) to verify the curve. There is a 30% acceptance range for the low standard and a 10% acceptance range for the remaining calibration standards. As long as the minimum number of calibration standards are maintained, the low and high standards may be removed and the calibration used; otherwise re-calibrate the instrument (prepare fresh calibration standards as necessary).

**11.3** For aqueous samples:

$$\mu\text{g/L Hg} = (\text{concentration Hg in digest}) (\text{dilution factor})$$

**11.4** To determine mg/kg from a result in  $\mu\text{g/L}$  for soil and sludge samples use:

$$\text{mg/kg Hg} = (\mu\text{g/L Hg in digest})(0.1) / (\text{wt of sample in g}) (\% \text{ dry solids})$$

**11.4** Calculate the percent recoveries of the matrix spikes and the LCS (see SVL 1028).

**11.5** Calculate the relative percent differences (RPDs) of the sample

duplicates.

**11.6** If the result is above the MDL but below the Reporting Limit, report the result to the client as "< (the Reporting Limit)," unless the client instructs otherwise. If the client requests numerical results below the Reporting Limit, report the results with one of the following qualifiers:

**11.6.1** E4 – Concentration estimated. Analyte was detected below laboratory minimum reporting limit.

**11.6.2** J – The reported value was less than the CRDL (Reporting Limit) but greater than or equal to the MDL.

## **12.0 DATA AND RECORDS MANAGEMENT**

**12.1** Data from the CETAC M6100 Mercury Analyzer

**12.2** In the File menu, click on "Export". The "Specify Export File" window will open.

**12.1.2** In the "Save as Type" menu, choose "Text File (\*.txt\*)."

**12.1.3** Choose "Data."

**12.1.4** In the "File Name" box, enter the file name.

**12.3** Click "Save." The "Export Complete" window will open.

**12.1.6** Click "OK."

**12.4** Click on the File drop down menu and select "Printer setup."

**12.5** In the Name drop down menu choose "pdfFactory pro," click "OK."

**12.5.1** Click the "Print button." The "pdfFactory Pro" window will open. Click on "Doc Info" and enter the file name. Click on "Preview," if no corrections or comments are needed click on "Print." Select the correct printer and print the raw data.

**12.5.2** Next, click the "Save" button. Select the correct month. Enter file name and choose PDF in the Save as Type drop down menu. Select "Save" and close out pdfFactory Pro.

**12.6** Data from the CETAC M7500 Mercury Analyzer

- 12.6.1** In the File menu, click on “Export.” The “Specify Export File” window will open.
- 12.6.2** In the “Save as Type” menu, choose “Text File (\*.txt).”
- 12.6.3** Choose “Data.”
- 12.6.4** In the “File Name” box, enter the file name.
- 12.6.5** Click “Save.” The “Export Complete” window will open.
- 12.6.6** Click “OK.”
- 12.6.7** Click the “Print” button. The “pdfFactory Pro” window will open. Click “Save” and make sure that you are in the H:\Mercury\PDFs\Cetac M7500 folder, navigate to the correct monthly folder and save your report with the file designation yymmdd(A, B, C, etc.) format.
- 12.7** Close the “pdfFactory Pro” window, Click on the Shortcut to Cetac M7500 folder on the desktop and navigate to the correct monthly folder. Click on the file name. Corrections and comments can be made here.
- 12.8** Click on “Comments.” Use the “Strikeout Text Tool” or the “Polyline Tool” located under “Drawing Markup Tools” to make a single line cross-out of incorrect data. The pencil on the toolbar is used to initial and date the correction and add any other necessary comments.
  - 12.8.1** Open the CVAA standards sheet called “Hg Log” (either SOIL or H2O template) excel sheet on the desktop and update the standards, prep dates and batch numbers for the run. Save and attach this file to the raw data pdf. This sheet indicates that all QC and the batches indicated have been digested together.
  - 12.8.2** Save the changes.
- 12.9** Procedures for constructing bench sheets can be found at R:\Promium Stuff\How to's\Batching.doc. Make sure that the bench sheet is initialed and dated when the actual preparation of the samples began.
  - 12.9.1** Indicate all reagents used in the batch by including them in the reagent section of the “Batch” screen.
  - 12.9.2** Complete the bench sheet, initial and date the sheet. Enter the

time of digestion and the time of the analytical run. Check the box for the analyzer used. Enter the filename.

**12.9.3** Fill out, print out and attach the Hg Traveler or Hg Traveler SOIL excel sheet located on the desktop that includes all of the support equipment used in preparing and analyzing this batch of samples, identify and record the temperature of the hot block(s) used for the digestion of the samples in the batch. Include the analyst initials and date of sample preparation on the sheet. Sheets located at H:\Instrument lab\TRAVELER SHEETS.

**12.10** File the raw data in the M7500 or M6100 Run log. Include a CVAA standards sheet (Hg Log), with each set of raw data.

**12.11** If the batch is Level 3 the analyst will print the standards sheet from Element for the stock mercury solution (6.16), all standards listed in the CVAA standards sheet and all reagents found on the finalized bench sheet. These will be turned in with the benchsheet, color sheet, Traveler, Calibration Check, and corrected raw data pdf.

**12.12** The analyst will upload the data via Data Tool. They shall perform all reviews on the "Data Entry/Review" page in Element and verify their data uploads.

**12.12.1** If Level 3, the analyst will skip all Data Entry/Review and instead update the status of the samples in the benchsheet editor to read "Analyzed"

**12.13** If an input comes up color coded, apply the appropriate data flags or undertake any corrective actions.

**12.14** The analyst shall assign any qualifiers.

**12.15** The analyst will then update the status of the batch to "Analyzed".

**12.16** The analyst will then lock the results so that any future imports will not overwrite acceptable results.

**12.17** The data review process is outlined in SOP SVL 2009.

**12.18** Corrective action is governed by SOP SVL 1019.

### **13.0 QUALITY CONTROL**

- 13.1** Prepare and analyze a RLCS (10.1.9) (10.2.9). This standard will be prepared from either a primary or secondary source at a concentration that reflects the current reporting limit. Run this standard once per run before the ICV. Acceptance limits are 70 to 130%. If the recovery falls outside these limits, re-analyze the RLCS. If the recovery still falls outside these limits, re-calibrate the instrument. The results of the RLCS will be tracked to show the viability of the current reporting limit.
- 13.2** Analyze a Quality Control Sample (QCS) (10.1.6) (10.2.6) as an ICV immediately after the calibration curve. Acceptance limits are 95 to 105% of the expected value for waters and 90 to 110% for soils. If the recovery falls outside these limits, re-analyze the ICV. If the recovery still falls outside these limits, re-calibrate the instrument.
- 13.3** Analyze an ICB (10.1.7) (10.2.7) after every ICV. The recovery must be less than the half of the Reporting Limit. If the recovery is greater than half of the Reporting Limit re-analyze the blank, if it is still greater than half of the Reporting Limit re-calibrate the instrument.
- 13.4** Analyze a method blank (PBW or PBS) (10.1.1) (10.2.1) at a frequency of one per batch of 20 or fewer samples. The recovery must be less than one half of the Reporting Limit or less than 10% of the lowest concentration of the analyte in all of the samples associated with that batch. If the recovery exceeds the criteria, re-analyze the PBW or PBS. If the recovery still exceeds the criterion, re-digest and re-analyze all samples in the affected batch.
- 13.5** Analyze a Laboratory Control Sample:
- 13.5.1** For aqueous analysis (LCSW) (10.1.2). Analyze a LCSW at a frequency of 1 per batch of 20 or fewer samples. The acceptance limits for the recovery are 85 to 115%. If the recovery falls outside these criteria, re-analyze the LCSW. If the recovery still falls outside these criteria, re-digest and re-analyze the samples associated with the LCSW.
- 13.5.2** For soil analysis (LCSS) (10.2.2). Analyze a LCSS at a frequency of 1 per batch of 20 or fewer samples. The acceptance limits for the recovery are 80 to 120%. If the recovery falls outside these criteria, re-analyze the LCSS. If the recovery still falls outside these criteria, re-digest and re-analyze the samples associated with the LCSS.

**13.5.3** For aqueous extract analysis (EPA 7470A) (LCSW) (10.1.2). Analyze a LCSW at a frequency of 1 per batch of 20 or fewer samples. The acceptance limits for the recovery are 80 to 120%. If the recovery falls outside these criteria, re-analyze the LCSW. If the recovery still falls outside these criteria, re-digest and re-analyze the samples associated with the LCSW.

**13.6** Analyze a matrix spike:

**13.6.1** For aqueous analysis, analyze a matrix spike (10.1.4) at a frequency of 1 per batch of 10 or fewer samples. The acceptance limits for spike recoveries are 70 to 130% of the expected value if the spike added is greater than 25% of the concentration of the un-spiked sample. There are no acceptance limits if the spike added is less than 25% of the concentration in the un-spiked sample. If the recovery falls outside these limits, flag the client report.

**13.6.2** For methods 7470A and 7471B, analyze a matrix spike (10.2.4) at a frequency of 1 per batch on 20 or fewer. The acceptance limits for spike recoveries are 80 to 120% of the expected value. If the recovery falls outside these limits, flag the client report.

**13.7** Analyze a matrix spike duplicate (10.1.5) (10.2.5) (10.2.5) for methods 245.1, 7470A and 7471B. The recoveries are as listed in 13.6.1 and 13.6.2. The acceptance limit for RPD between matrix spike and matrix spike duplicate is 20%. If the recoveries or RPDs fall outside of the above limits, flag the client report.

**13.8** Analyze a Continuing Calibration Verification (CCV) (10.1.8) (10.2.8) at a frequency of every 10 samples, and at the end of the run. The acceptance limits for method 245.1 (wastewater and drinking water) are 90% to 110% of the expected value. The acceptance limits for methods 7470A and 7471B are 80% to 120% of the expected value. If the recovery falls outside these criteria, determine the cause, perform corrective action, and re-analyze it. If the recovery still exceeds these criteria, re-calibrate the instrument and re-analyze all samples run since the last successful CCV.

**13.9** Analyze a Continuing Calibration Blank (CCB) (10.1.7) (10.2.7) at a frequency of every 10 samples, and at the end of the run. The CCB must be less than half of the reporting limit. If the recovery exceeds this criterion, determine the cause, perform corrective action, and re-analyze it. If the recovery still exceeds the limit, re-calibrate the instrument and re-analyze all samples run since the last successful CCB.

- 13.10 Perform a method detection limit (MDL) study for aqueous and soil (using sand (6.22)) matrices annually.
- 13.11 Perform a linear dynamic range study annually.
  - 13.11.1 Calibrate the instrument.
  - 13.11.2 Analyze standards at concentrations above the calibration range until recoveries are outside of 10%.
  - 13.11.3 The determined LDR must be documented and kept on file.
  - 13.11.4 For method 245.1 determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed.
- 13.12 Trend analysis can be found in SOP SVL 1033.
- 13.13 Demonstration of capability requirements can be found in SOP SVL 1010.

#### 14.0 REFERENCES

- 14.1 Method 7470A, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Third Edition, Update III (December 1996).
- 14.2 Method 7471B, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Revision II, February 2007.
- 14.4 Method 245.1, Methods for the Chemical Analysis of Water and Wastes, EPA-600.4-79-020, revised March 1983.
- 14.5 Method 245.1, Revision 3.0, Methods for the Determination of Metals in Environmental Samples—Supplement I, EPA/600/R-94/111, May 1994.
- 14.6 Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition.

#### 15.0 POLLUTION PREVENTION

- 15.1 All standards are prepared and reagents used in volumes consistent with good laboratory practice, to minimize the volume of disposable waste.



**15.2** Efficient laboratory practices that reduce the need for re-digestions and/or re-extractions minimize contributions to pollution.

**16.0 WASTE MANAGEMENT**

**16.1** Waste generated by this method includes plastic-ware, chemicals used in the digestion and/or analysis, and paper.

**16.2** Plastic-ware is emptied, rinsed, and discarded to a sanitary landfill.

**16.3** Most chemicals used during digestion and/or analysis are neutralized and/or diluted prior to disposal by permit to the public sewer. Any hazardous chemicals and/or residues are disposed of through SVL's hazardous waste disposal system (see SOP SVL 1008).

**17.0 CHANGE HISTORY**

DATE	VER.	CHANGE
01/04/10	13.0	Multiple changes were made to the document plus the addition of the CETAC M7500 requirements. Consult the archived version for comparisons.
01/18/11	14.0	1.0 changed to "aqueous samples are stored at ambient temperature". 6.11 changed to 125 grams and 2.5 liters. 6.14 changed to 400 grams SnCl <sub>2</sub> and 280 mL of HCL diluted to 4 liters. 12.1.12 and 12.2.19 added "Procedures for constructing bench sheets can be found at R:\Promium Stuff\How to's\Batching.doc. Make sure that the bench sheet is initialed and dated when the actual preparation of the samples began". 12.1.12.1 and 12.2.19.1 added "Indicate all reagents used in the batch by including them in the reagent section of the "Batch" screen". 12.1.12.2 and 12.2.19.2 added "Identify and record the temperature of the hot block(s) used for the digestion of the samples in that batch". 12.1.12.3 and 12.2.19.3 added "Fill out and attach a sheet that includes all of the support equipment used in preparing and analyzing this batch of samples". 10.3.59 changed to "concentration". Added 13.5.3 "For aqueous extract analysis (EPA 7470A) (LCSW) (10.1.7). Analyze a LCSW at a frequency of 1 per batch of 20 or fewer samples. The acceptance limits for the recovery are 80 to 120%. If the recovery falls outside these criteria, re-analyze the LCSW. If the recovery still falls outside these criteria, re-digest and re-analyze the samples associated with the LCSW".

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DATE	VER.	CHANGE
5/02/12	15.0	<p>6.22 Added "Sand, Fisher S23-3". 9.4 added "Logbook and in a Work Order memo". 10.1.1 added "(samples should be qualified; ref, SVL 2009)". 10.1.3 added "Final volume on benchsheet should be adjusted to 50.5 mL". 10.2.1 added "<math>\pm 0.005</math> g". 10.2.2 added "Final volume on benchsheet should be adjusted to 101.0 mL". Re-wrote section 10.3 to account for the changes in operating the M6100 instrument. 10.2.7 changed to "Prepare an LCS by transferring 1.0 mL of the mercury ICV stock solution (6.20) to 0.6 g <math>\pm</math> 0.005 g of sand (6.22) into a "Snap-cap" vial, add 4.0 mL deionized water (6.21). Mix well. The concentration of the LCS is 5.0 <math>\mu</math>g/L". 10.2.8 changed to "Prepare a method blank by weighing 0.6 g <math>\pm</math> 0.005 g of sand (6.22) add 5.0 mL of deionized water (6.21) into a "Snap-cap" vial. Mix well". 11.1.1 added "Use the calibration verification template (located at H:\Templates\Calibration Curve Check) to verify the curve. There is a 30% acceptance range for the low standard and a 10% acceptance range for the remaining calibration standards. As long as the minimum number of calibration standards are maintained, the low and high standards may be removed and the calibration used; otherwise re-calibrate the instrument (prepare fresh calibration standards as necessary". Re-wrote Section 12.0 to include the M6100 procedures. 13.13 added "(using sand (6.22))".</p>

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DATE	VER.	CHANGE
11/26/12	16.0	<p>4.4 changed to "Do not swallow, inhale or absorb through skin". 8.3 added a column for 10 ml initial volume. 10.1.5 added "Prepare a matrix spike duplicate by following the steps outlined in 10.1.4". 10.2.3 changed to "Weigh out a 0.6 g ± 0.005 g portion of a well homogenized sample in a "Snap-cap" vial". 10.2.4 changed to "Prepare a matrix spike by weighing another 0.6 g ± 0.005 g portion of a well homogenized sample in a "Snap-cap" vial". 10.2.5 changed to "Prepare a matrix spike duplicate. Follow the process outlined in 10.2.4". 10.2.11 added "± 3°C". 10.2.12 changed to "40 mL deionized water". 10.2.13 changed to "15 mL potassium permanganate solution". 10.2.14 changed to "Cool the vials and add 6 mL of hydroxylamine hydrochloride solution (6.9) to reduce the excess permanganate. Add 49 mL deionized water (6.21) into samples, prep blank, LCS, matrix spike and matrix spike duplicate. Add 44 mL deionized water to the calibration standards, ICB, CCBs, RLCS, ICV, and CCVs. Add proportionally less water if additional permanganate is used. The final volume of all standards and samples should be 120 mL". 10.3.31 and 10.4.43 changed to "DO NOT STOP THE ANALYSIS until the analysis has registered". 10.4.43.1 added "All analyses are to finish, the Cetac allows for stopping an analysis prior to a final read, this is not allowed. All failures must be re-inserted at a later point in the run". 10.3.32 and 10.4.44 changed to "Under 245.1 if the concentration in a sample exceeds 90% of the linear dynamic range, the sample must be re-analyzed at a lower dilution. 7471B requires that samples that exceed the calibration range be diluted back to within the calibration range". 13.6.2 changed to "are 80 to 120% of the expected value". 13.7 changed to "Analyze a matrix spike duplicate (10.1.5) (10.2.5) (10.2.5) for methods 245.1, 7470A and 7471B. The recoveries are as listed in 13.6.1 and 13.6.2. The acceptance limit for RPD between matrix spike and matrix spike duplicate is 20%. If the recovery falls outside these limits, flag the client report". 13.14 changed to "Perform a linear dynamic range study annually ". 13.14.1 changed to "Calibrate the instrument". 13.14.2 changed to "Analyze standards at concentrations above the calibration range until recoveries are outside of 10%". 13.14.3 changed to "The determined LDR must be documented and kept on file". 13.14.4 changed to "For method 245.1 determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed". 14.2 changed to "Method 7471B, <u>Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Revision II, February 2007</u>".</p>

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DATE	VER.	CHANGE
12/23/13	17.0	10.2.4 changed "Prepare a matrix spike by weighing another 0.60 g $\pm$ 0.005 g portion of a well homogenized sample in a "Snap-cap" vial and add 2.0 mL of the working standard solution (6.19). Add 3.0 mL deionized water (6.21). The initial concentration of the matrix spike is 2.0 ppb."
1/13/15	18.0	12.12 added "Corrective action is governed by SOP SVL 1019." 13.12 added "Trend analysis can be found in SOP SVL 1033." 13.13 added "Trend analysis can be found in SOP SVL 1033."
01/14/16	19.0	Multiple changes were made to the document plus the addition of extraction requirements. Consult the archived version for comparisons.
06/17/16	19.1	Section 10 added the following statement "To all standards (calibration and QC) and samples do the following:" 12.8.1 added "This sheet indicates that all QC and the batches indicated have been digested together."

**ANALYSIS OF METALS BY METHODS 6010C and 200.7  
USING THE PERKIN-ELMER OPTIMA ICP**

**Revised by: Michael Desmarais**

**Approved by: \_\_\_\_\_ Date: \_\_\_\_\_**

**Inorganic Instrumental Department Supervisor**

**Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_**

**Quality Assurance Manager**

**SVL Analytical, Inc.**



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**1.0 SCOPE AND APPLICATION**

This SOP describes the procedure used for the operation and analysis of samples on the Perkin-Elmer Optima instruments. It is applicable to drinking water, wastewater, soil, and hazardous waste samples. SVL uses a Laboratory Information Management System (LIMS) – Element, to manage client’s samples. In Element the current aqueous and soil MDLs and RLs are shown below. Definitions for words used in this SOP may be found in SVL’s Quality Manual. The holding time for aqueous samples is six months from date of sampling.

<b>Metal</b>	<b>Wavelength (nm)</b>	<b>Aqueous Reporting Limit (mg/L)</b>	<b>Soil Reporting Limit (mg/Kg)</b>	<b>Aqueous MDLs (mg/L)</b>	<b>Soil MDLs (mg/Kg)</b>
Silver	328.06	0.005	0.5	0.0021	0.22
Aluminum	308.21	0.08	8.0	0.036	4.7
Arsenic	193.7	0.025	2.5	0.0096	0.81
Barium	233.52	0.002	0.2	0.00059	0.066
Beryllium	313.10	0.002	0.2	0.00094	0.096
Boron	249.68	0.04	4.0	0.0057	0.35
Bismuth	222.82	0.06	6.0	0.012	1.3



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<b>Metal</b>	<b>Wavelength (nm)</b>	<b>Aqueous Reporting Limit (mg/L)</b>	<b>Soil Reporting Limit (mg/Kg)</b>	<b>Aqueous MDLs (mg/L)</b>	<b>Soil MDLs (mg/Kg)</b>
Calcium	315.89	0.1	10.0	0.058	2.3
Cadmium	226.50	0.002	0.2	0.00086	0.069
Cerium	418.66				
Cobalt	228.62	0.006	0.6	0.0019	0.056
Chromium	267.72	0.006	0.6	0.001	0.16
Copper	324.75	0.01	1.0	0.0023	0.28
Iron	273.95	0.06	6.0	0.048	4.5
Gallium	417.20	0.02	2.0	0.0067	0.55
Potassium	766.47	0.5	50	0.25	15.0

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<b>Metal</b>	<b>Wavelength (nm)</b>	<b>Aqueous Reporting Limit (mg/L)</b>	<b>Soil Reporting Limit (mg/Kg)</b>	<b>Aqueous MDLs (mg/L)</b>	<b>Soil MDLs (mg/Kg)</b>
Lanthanum	379.47	0.005	0.5	0.0031	0.19
Lithium	670.76	0.02	2.0	0.012	0.52
Lutetium	261.53				
Magnesium	279.08	0.2	20.0	0.11	14.0
Manganese	260.57	0.004	0.4	0.0023	0.27
Molybdenum	202.03	0.008	0.8	0.0054	0.21
Sodium	589.57	0.5	50.0	0.1	3.7
Nickel	232.00	0.01	1.0	0.0037	0.22
Phosphorus	213.62	0.05	5.0	0.031	3.4

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<b>Metal</b>	<b>Wavelength (nm)</b>	<b>Aqueous Reporting Limit (mg/L)</b>	<b>Soil Reporting Limit (mg/Kg)</b>	<b>Aqueous MDLs (mg/L)</b>	<b>Soil MDLs (mg/Kg)</b>
Lead	220.35	0.0075	0.75	0.003	0.32
Antimony	206.83	0.02	2.0	0.0091	0.72
Scandium	361.38	0.002	0.2	0.0011	0.17
Selenium	196.03	0.04	4.0	0.015	1.5
Silicon	251.61	0.08		0.056	
Tin	189.93	0.05	5.0	0.012	0.41
Strontium	421.54	0.005	0.5	0.0017	0.061
Titanium	336.12	0.005	0.5	0.0016	0.068
Thallium	190.80	0.015	1.5	0.0086	0.77

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<b>Metal</b>	<b>Wavelength (nm)</b>	<b>Aqueous Reporting Limit (mg/L)</b>	<b>Soil Reporting Limit (mg/Kg)</b>	<b>Aqueous MDLs (mg/L)</b>	<b>Soil MDLs (mg/Kg)</b>
Vanadium	202.40	0.005	0.5	0.0022	0.16
Zinc	206.20	0.01	1.0	0.0039	0.69

## **2.0 SUMMARY OF METHOD**

This SOP is intended to comply with the requirements of EPA methods 200.7 and 6010C. 6010B may also be run under this SOP. Argon is used to establish plasma within a quartz torch. A small amount of digested sample is transported by a peristaltic pump and tubing through an atomizer into the plasma. The metal atoms are excited by the plasma and emit light at characteristic wavelengths. A grating disperses the spectra. A photoelectric detector measures the emission of light by the metal atoms. Background is measured adjacent to the analyte wavelengths. Software creates a linear calibration curve of emission versus concentration and calculates the concentration in the sample.

## **3.0 INTERFERENCES**

- 3.1** Interferences can result from background emission from continuous recombination phenomena. Use the background correction features in the software.
- 3.2** Interferences can result from emission by high concentrations of other elements. Use background correction features in the software.
- 3.3** Interferences can result from overlap of a spectral line of another element. Use an alternative wavelength or the inter-element correction factors in the software.
- 3.4** Interferences can result from differences in viscosity between calibration standards and samples due to high dissolved solids or acid concentrations. Use a peristaltic pump to deliver solutions.

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- 3.5** Interferences can result from molecular compound formation and ionization effects. Modify the incident power or observing position.
- 3.6** Interferences may result from memory of previous samples. Use a rinse blank between samples.

### **4.0 SAFETY**

- 4.1** Extremely high power is required to generate the plasma in the ICP. The RF generator must be shielded to prevent physical injury to operators. The plasma may approach a temperature of 10,000 degrees C.

### **5.0 EQUIPMENT, INSTRUMENTATION AND MATERIALS**

Equivalent equipment, instrumentation and materials may be used.

- 5.1** Perkin-Elmer Optima 8300 DV Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES).
- 5.2** GemCone Nebulizer, Low Flow, Part No. N069-0671.
- 5.3** Concentric Glass Nebulizer (Meinhard), Part No. 0047-2020.
- 5.4** GemTip Cross-Flow Nebulizer, Part No. N077-0546.
- 5.5** Burgener Peek Mira Mist Nebulizer, Part No. N0775-330.
- 5.6** Quartz Torch, Part No. N077-0338.
- 5.7** Alumina Injector, 2.0-mm i.d. Part No. N077-5177, Sapphire Injector, 1.8 mm i.d. Part no. 4060-010978.
- 5.8** Ryton Double-Pass Scott-Type Spray Chamber, Part No. N077-5296.
- 5.9** Perkin-Elmer AS-93plus Autosampler, Perkin-Elmer S10 Autosampler, ESI SCFAST Autosampler.
- 5.10** Volumetric flasks, pyrex, 100-mL, 200-mL, 1-L.
- 5.11** Micropipets, Corning, Lambda, Wheaton, Socorex, variable volume, or equivalent.

- 5.12** WinLab32 Instrument Control Software.
- 5.13** Neslab CFT-75 Chiller.
- 5.14** Peristaltic Pump.
- 5.15** Standard Pump Sample Tubing: 0.76 mm (0.030 in) ID, Part No. 0990-8587.
- 5.16** Silicone Pump Tubing for MIBK solvent: 0.76 mm (0.30 in) inner diameter, Part No. 0047-3552.
- 5.17** Standard Pump Drain Tubing: 1.14 mm (0.045 in) inner diameter, Part No. 0990-8585.
- 5.18** Silicone Pump Drain Tubing for MIBK solvent: 1.14 mm (0.045 in) inner diameter, Part No. N069-1595.
- 5.19** Teflon Tubing, 1/8" outer diameter, Part No. 0250-6483.
- 5.20** Polyethylene Nebulizer Tubing, 0.58 mm inner diameter, Part No. 0990-8265.
- 5.21** PVC Spray Chamber Drain Tubing, 1.5 mm inner diameter, Part No. 0998-5735.
- 5.22** RF Generator Air Filter, Part No. N077-5220.
- 5.23** Spectrometer Air Filter, Part No. 0250-9115.
- 5.24** pH strips, 0 – 6 pH range, EM colorpHast, Fisher M95863 (used to adjust sample pH, if necessary).

## **6.0 REAGENTS AND STANDARDS**

Guidelines for the storage, tracking and expiration of chemicals and reagents can be found in SOP SVL 1032. The procedure for purchasing chemicals and reagents can be found in SOP SVL 1015. Any exceptions to the above mentioned SOPs will be found in this section: as well as, all of the preparatory steps needed to construct or prepare reagents, and standards. Equivalent reagents or standards may be used.

- 6.1** Argon, 99.99%, with regulator set between 550 and 825 kPa (80 and 120 psig).

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- 6.2** Concentrated hydrochloric acid, Fisher TraceMetals Grade, or EMD Omni Trace.
- 6.3** Concentrated nitric acid, Fisher TraceMetals Grade, or EMD Omni Trace.
- 6.4** Commercially-manufactured single-element stock solutions:

<b>Element</b>	<b>Concentration (µg/mL)</b>	<b>Mfg</b>	<b>Part No</b>	<b>Matrix</b>
Aluminum	10000	HPS	10001-1-250	2% HNO <sub>3</sub>
Antimony	1000	HPS	10002-3-250	5% HNO <sub>3</sub> + 0.1% HF
Arsenic	1000	HPS	10003-1-250	2% HNO <sub>3</sub>
Barium	10000	HPS	10M4-1-250	4% HNO <sub>3</sub>
Beryllium	1000	HPS	10005-1-250	2% HNO <sub>3</sub>
Bismuth	10000	HPS	10M6-1-250	4% HNO <sub>3</sub>
Boron	5000	HPS	5M7-4-250	Water
Cadmium	1000	HPS	10008-1-250	2% HNO <sub>3</sub>
Calcium	10000	HPS	10M9-1-250	4% HNO <sub>3</sub>
Cerium	1000	HPS	100010-1-250	2% HNO <sub>3</sub>
Chromium	1000	HPS	100012-1-250	2% HNO <sub>3</sub>
Cobalt	1000	HPS	100013-1-250	2% HNO <sub>3</sub>
Copper	1000	HPS	100014-1-250	2% HNO <sub>3</sub>
Gallium	10000	HPS	10M19-1-250	4% HNO <sub>3</sub>
Iron	10000	HPS	10M26-1-250	10% HNO <sub>3</sub>
Lanthanum	10000	HPS	10M27-1-250	4% HNO <sub>3</sub>
Lead	1000	HPS	100028-1-250	2% HNO <sub>3</sub>
Lithium	10000	HPS	100029-1-250	1% HNO <sub>3</sub>
Lutetium	10000	HPS	10M33-1-250	4% HNO <sub>3</sub>
Magnesium	10000	HPS	10M31-1-250	4% HNO <sub>3</sub>
Manganese	1000	HPS	100032-1-250	2% HNO <sub>3</sub>
Molybdenum	1000	HPS	100034-3-250	2% HNO <sub>3</sub> +0.1% HF
Nickel	1000	HPS	100036-1-250	2% HNO <sub>3</sub>
Phosphorus	10000	HPS	10M39-1-250	0.05% HNO <sub>3</sub>

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<b>Element</b>	<b>Concentration (µg/mL)</b>	<b>Mfg</b>	<b>Part No</b>	<b>Matrix</b>
Potassium	10000	HPS	10M41-1-250	1% HNO <sub>3</sub>
Selenium	1000	HPS	100049-1-250	2% HNO <sub>3</sub>
Scandium	10000	HPS	10M48-1-250	4% HNO <sub>3</sub>
Silicon	10000	HPS	10M50-4-250	Water
Silver	1000	HPS	100051-1-250	2% HNO <sub>3</sub>
Sodium	10000	HPS	10M52-1-250	1% HNO <sub>3</sub>
Strontium	10000	HPS	10M53-1-250	4% HNO <sub>3</sub>
Thallium	1000	HPS	100058-1-250	2% HNO <sub>3</sub>
Tin	10000	HPS	10M61-3-250	4% HNO <sub>3</sub> +2% HF
Titanium	1000	HPS	100062-3-250	2% HNO <sub>3</sub> +0.1% HF
Vanadium	1000	HPS	100065-1-250	2% HNO <sub>3</sub>
Yttrium	1000	HPS	100067-1-250	2% HNO <sub>3</sub>
Zinc	1000	HPS	100068-1-250	2% HNO <sub>3</sub>

- 6.6** Spiking solutions: N5, SVL11, TCLP7, QC-Sc and QC-Sn (see SVL 4119 for preparation).
- 6.7** QC19 stock solution: Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Se, Tl, Ti, V, Zn at 100 µg/mL in 5% HNO<sub>3</sub> and trace HF, CPI S4400-004.
- 6.8** QC26 stock solution: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Se, Ag, Na, Tl, Ti, V, and Zn at 100 µg/mL, K at 1000 µg/mL, and Si at 50 µg/mL, in 4% HNO<sub>3</sub>, High-Purity Standards QCS-26.
- 6.9** Standard 1 Mix: Bi, Ga, Li, Sn and Sr at 500 µg/mL as well as P at 1000 µg/mL, in 10% HCl and Tr HNO<sub>3</sub>. High-Purity Standards SM-150-060. Expiration date as stated by the manufacturer.
- 6.10** Calibration blank (Seq-Cal1@S0): Solution of 2% nitric acid (6.4) and 5% hydrochloric acid (6.3) in deionized water (6.25). This solution is used as the ICB and CCB also.
- 6.11** Calibration standard number 1 (Seq-Cal 2@S): Add deionized water (6.25) to a 1000 mL volumetric flask. Carefully add 20 mL concentrated nitric acid (6.4), 50 mL concentrated hydrochloric acid (6.3). Add 30 mL QC 26 stock solution (6.8) and 10 mL Standard 1 Mix (6.9). Dilute to the



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mark with deionized water and mix well. Store in a brown bottle, or away from light. The final concentrations of the elements in this solution are:

<b>Volume (mL)</b>	<b>Stock Solution (mg/L)</b>	<b>Final Concentration in Standard 1 (mg/L)</b>
30.0	100 Ag	3
30.0	100 As	3
30.0	100 B	3
30.0	100 Ba	3
30.0	100 Be	3
10.0	500 Bi	5
30.0	100 Cd	3
30.0	100 Co	3
30.0	100 Cr	3
30.0	100 Cu	3
10.0	500 Ga	5
30.0	1000 K	30
10.0	800 Li	8
30.0	100 Mn	3
30.0	100 Mo	3
30.0	100 Ni	3
10.0	1000 P	10
30.0	100 Pb	3
30.0	100 Sb	3
30.0	100 Se	3
10.0	500 Sn	5
10.0	500 Sr	5
30.0	100 Ti	3
30.0	100 Tl	3
30.0	100 V	3
30.0	100 Zn	3

**6.12** Calibration standard number 2 (Seq-Cal 3@S): Add deionized water (6.25) to a 1000 mL volumetric flask. Carefully add 20 mL concentrated nitric acid (6.4), 50 mL concentrated hydrochloric acid (6.3). Add the following amounts of single-element stock solutions (6.5). Then dilute to the mark with deionized water and mix well. The final concentrations of the elements in this solution are:

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<b>Volume (mL)</b>	<b>Stock Solution (mg/L)</b>	<b>Final Concentration in Standard 2 (mg/L)</b>
5.0	10000 Al	50
5.0	10000 Ca	50
5.0	10000 Fe	50
5.0	10000 Mg	50
5.0	10000 Na	50
2.5	10000 Si	25.0
2.5	10000 SiO <sub>2</sub>	53.5

- 6.13** Calibration standard number 3 (Seq-Cal 4@S): Add deionized water (6.25) to a 1000 mL volumetric flask. Carefully add 20 mL concentrated nitric acid (6.4), 50 mL concentrated hydrochloric acid (6.3). Add 10 mL of Standard 3 Mix (6.26) and 2.5 mL Si stock solution. Then dilute to the mark with deionized water and mix well. The final concentrations of the elements in this solution are:

<b>Volume (mL)</b>	<b>Stock Solution (mg/L)</b>	<b>Final Concentration in Standard 3 (mg/L)</b>
1.0	10000 La	10.0
0.2	10000 Sc	2.0
2.5	10000 Si	25.0
2.5	10000 SiO <sub>2</sub>	53.5

- 6.14** Calibration standard number 4 (Seq-Cal 5@S): Add deionized water (6.25) to a 1000 mL volumetric flask. Carefully add 20 mL concentrated nitric acid (6.4), 50 mL concentrated hydrochloric acid (6.3). Add the following amount of single-element stock solution (6.5). Then dilute to the mark with deionized water and mix well. The final concentrations of the elements in this solution are:

<b>Volume (mL)</b>	<b>Stock Solution (mg/L)</b>	<b>Final Concentration in Standard 4 (mg/L)</b>
1.0	1000 Ce	1.0

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- 6.15** ICS-AB stock solution: Add deionized water (6.25) to a 200 mL volumetric flask. Carefully add 2 mL concentrated nitric acid (6.4), 10 mL concentrated hydrochloric acid (6.3). Add the following amounts of single-element stock solutions (6.5). Then dilute to the mark with deionized water and mix well. The final concentrations of the elements in this solution are:

<b>Volume (mL)</b>	<b>Stock Solution (mg/L)</b>	<b>Final Concentration in ICS-AB (mg/L)</b>
10.0	1000 Be	50
10.0	1000 Co	50
10.0	1000 Mn	50
10.0	1000 Cr	50
10.0	1000 Cu	50
10.0	1000 V	50
10.0	1000 Cd	50
10.0	1000 Pb	50
10.0	1000 Ni	50
10.0	1000 Zn	50
1.00	10000 Ba	50
10.0	1000 As	50
10.0	1000 Se	50
10.0	1000 TI	50
10.0	1000 Sb	50

- 6.16** ICSA (containing Fe, Mg, Ca, Al), HPS Catalog No. 4400-INTA1-500.
- 6.17** Working ICS-A solution: Add deionized water (6.25) to a 200 mL volumetric flask. Add 4 mL concentrated nitric acid (6.4) and 10 mL of concentrated hydrochloric acid (6.3). Add the following amounts of stock solutions. Then dilute to the mark with deionized water and mix well. The final concentrations will be Al at 500 mg/L, Ca at 500 mg/L, Mg at 500 mg/L, Fe at 200 mg/L, and Cr, Cu, Mn, Ni, Ti, and V each at 10 mg/L. Use the following volumes of the stock standards:

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<b>Stock Solution (mg/L)</b>	<b>Volume Stock (mL)</b>
Interference A Standard	20.0
1000 Cr	2.0
1000 Cu	2.0
1000 Mn	2.0
1000 Ni	2.0
1000 Ti	2.0
1000 V	2.0

**6.17.1** The concentrations for the working ICSA solution are listed below:

<b>Element</b>	<b>Final Conc. (mg/L)</b>	<b>Element</b>	<b>Final Conc. (mg/L)</b>
Al	500	Ca	500
Cr	10	Cu	10
Fe	200	Mg	500
Mn	10	Ni	10
Ti	10	V	10

**6.18** Working ICS-AB solution: Add deionized water (6.25) to a 200 mL volumetric flask. Carefully add 4 mL concentrated nitric acid (6.4) and 10 mL concentrated hydrochloric acid (6.3). Add the following amounts of stock solutions. Then dilute to the mark with deionized water and mix well. Store in a brown bottle, or away from light. . Use the following volumes of the stock standards:

<b>Stock Solution</b>	<b>Volume (mL)</b>
Interference A Standard	20.0
ICS-AB Stock Solution	2.00
1000 mg/L Ag	0.10

**6.18.1** The concentrations for the working ICS-AB solution are listed below:

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<b>Element</b>	<b>Final Conc. (mg/L)</b>	<b>Element</b>	<b>Final Conc. (mg/L)</b>
Al	500	Sb	0.5
As	0.5	Ba	0.5
Be	0.5	Cd	0.5
Ca	500	Cr	0.5
Co	0.5	Cu	0.5
Fe	200	Pb	0.5
Mg	500	Mn	0.5
Ni	0.5	Se	0.5
Ag	0.5	Tl	0.5
U	0.5	V	0.5
Zn	0.5		

**6.19** Reporting Limit Check Solution (RLCS) stock: Add deionized water (6.25) to a 1000 mL volumetric flask. Add 20 mL of concentrated nitric acid (6.4) and 50 mL of concentrated hydrochloric acid (6.3). Add the following amounts of single-element stock solutions. Preparation of intermediate solutions of the single-element stock solutions is permissible. Then dilute to the mark with deionized water and mix well. The final concentrations of the elements in this solution are:

<b>Stock Solution</b>	<b>Volume of Stock Solution (mL)</b>	<b>Final Concentration (mg/L)</b>
1000 Ag	0.50	0.50
10000 Al	0.80	8.00
1000 As	2.5	2.50
5000 B	0.80	4.00
10000 Ba	0.02	0.20
1000 Be	0.20	0.20
10000 Bi	0.60	6.00
10000 Ca	1.00	10.00
1000 Cd	0.20	0.20
1000 Cr	0.60	0.60
1000 Co	0.60	0.60
1000 Cu	1.0	1.00
10000 Fe	0.60	6.00

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<b>Stock Solution</b>	<b>Volume of Stock Solution (mL)</b>	<b>Final Concentration (mg/L)</b>
10000 Ga	0.20	2.00
10000 K	5.0	50.0
10000 La	0.050	0.50
10000 Li	0.20	2.00
10000 Mg	2.00	20.00
1000 Mn	0.40	0.40
1000 Mo	0.80	0.80
10000 Na	5.0	50.0
1000 Ni	1.0	1.00
10000 P	0.50	5.00
1000 Pb	0.75	0.75
1000 Sb	2.0	2.00
10000 Sc	0.020	0.20
1000 Se	4.0	4.00
10000 Si	0.80	8.00
10000 Sn	0.50	5.00
10000 Sr	0.050	0.50
1000 Ti	0.50	0.50
1000 Tl	1.5	1.50
1000 V	0.50	0.50
1000 Zn	1.0	1.00

**6.20** ICV and CCV solution. Add deionized water (6.25) to a 1000 mL volumetric flask. Carefully add 20 mL concentrated nitric acid (6.4) and 50 mL concentrated hydrochloric acid (6.3). Add the following amounts of stock solutions. Dilute to the mark with deionized water and mix well. Make sure to use a secondary source or different lot number than was used for the calibration standards.

<b>Stock Solution (mg/L)</b>	<b>Volume (mL)</b>
QC26 Stock Solution	20.0
10000 Si	0.90
10000 P	1.0
10000 Sc	0.10
10000 Sr	0.20

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<b>Stock Solution (mg/L)</b>	<b>Volume (mL)</b>
10000 Na	1.8
10000 Bi	0.40
10000 Ga	0.40
10000 La	0.40
10000 Li	0.40
10000 Sn	0.40

**6.20.1** Low Level Reporting Limit Check Solution (LLRLCS) solution: Add deionized water (6.25) to a 1000 mL volumetric flask. Carefully add 20 mL concentrated nitric acid (6.4) and 50 mL concentrated hydrochloric acid (6.3). Add 0.4 mL of 1000 ppm silver stock solution (6.5). To this is added 10 mL of Low Level RLCS Stock Fe and Al (6.26). Dilute to the mark with deionized water and mix well. Make sure to use a secondary source or a different lot number than was used for the calibration standards for the silver.

This solution (LLRLCS) is to verify low level silver for 200.7 as well as confirming low level values for aluminum and iron as required for special projects. These values are 0.040 mg/L for aluminum and 0.050 mg/L for iron. The solution replaces the method required low level ICV. For the LLRLCS values see (6.26).

<b>Stock Solution (mg/L)</b>	<b>Volume (mL)</b>
1000 Ag	0.40
4 Al	0.04
5 Fe	0.05

**6.20.2** The concentrations for the ICV/CCV solution are listed below.

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<b>Element</b>	<b>Final Conc. (mg/L)</b>	<b>Element</b>	<b>Final Conc. (mg/L)</b>
Ag	2	Mg	2
Al	2	Mn	2
As	2	Mo	2
B	2	Na	20
Ba	2	Ni	2
Be	2	P	10
Bi	4	Pb	2
Ca	2	Sb	2
Cd	2	Sc	1
Cr	2	Se	2
Co	2	Si	10
Cu	2	Sn	4
Fe	2	Sr	2
Ga	4	Ti	2
K	20	Tl	2
La	4	V	2
Li	4	Zn	2

**6.21** RLCS working: Add deionized water (6.25) to a 500 mL volumetric flask. Carefully add 10 mL concentrated nitric acid (6.3) and 25 mL concentrated hydrochloric acid (6.4). Add 5.0 mL of the RLCS stock (6.19). Store in a brown bottle, or away from light. The final concentrations of the elements in this solution are:

<b>Element</b>	<b>Final Concentration (µg/L)</b>	<b>Element</b>	<b>Final Concentration (µg/L)</b>
Ag	5	Mg	200
Al	80	Mn	4
As	25	Mo	8
B	40	Na	500
Ba	2	Ni	10
Be	2	P	50
Bi	60	Pb	7.5
Ca	100	Sb	20
Cd	2	Sc	2



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<b>Element</b>	<b>Final Concentration (µg/L)</b>	<b>Element</b>	<b>Final Concentration (µg/L)</b>
Cr	6	Se	40
Co	6	Si	80
Cu	10	Sn	50
Fe	60	Sr	5
Ga	20	Ti	5
K	500	Tl	15
La	5	V	5
Li	20	Zn	10

- 6.22** Internal standard: Add deionized water (6.25) to a 3000 mL bottle. Add 60 mL concentrated nitric acid (6.4) and about 6 mL of a 10000 ppm lutetium stock solution (6.5). Dilute to the mark with deionized water and mix well.
- 6.23** Zinc alignment solution: This is a 2 mg/L solution provided by the manufacturer of the instrument. It is used to align the torch after replacement.
- 6.24** Type II deionized water.
- 6.25** Standard 3 Mix: La at 1000 ug/mL and Sc ug/mL at 200 in 2% HNO<sub>3</sub>, High-Purity Standards SM-150-061. Expiration date as stated by the manufacturer.
- 6.26** Low Level RLCS Stock Fe and Al: Add deionized water (6.25) to a 500 mL volumetric flask. Carefully add 10 mL concentrated nitric acid (6.3) and 25 mL concentrated hydrochloric acid (6.4). Add 0.2 mL of the aluminum and 0.25 mL of the iron single element stock solutions (6.4). Dilute to mark and mix well.

<b>Stock Solution</b>	<b>Volume of Stock Solution (mL)</b>	<b>Final Concentration (mg/L)</b>
10000 Al	0.20	4.00
10000 Fe	0.25	5.00

## **7.0 INSTRUMENT SETTINGS**

- 7.1** Ensure that the exhaust vent is ON.

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- 7.2** Ensure that the argon tank has enough argon, and that the tank valve is open.
- 7.3** Ensure that the water chiller is ON, and that it has enough water.
- 7.4** Ensure that the peristaltic pump tubing is in good condition.
- 7.5** Ensure that the drain tubing on the spray chamber is in good condition.
- 7.6** Ensure that the drain bottle has enough empty volume to collect waste.
- 7.7** Ensure that the autosampler rinse bottle has enough water, and that the lutetium internal standard bottle is full.
- 7.8** Ensure that the sampling probe is installed at the correct height, and that the probe capillary is attaching to the pump tubing.
- 7.9** Ensure that the printer has an adequate supply of paper.
- 7.10** Ensure that the “Main Instrument” switch is ON. Leave the “Main Instrument” switch on, even when the instrument is not in use.
- 7.11** Ensure that the autosampler is ON.
- 7.12** Ensure that the computer, monitor, and printer are ON.

**8.0 CALIBRATION**

- 8.1** Use a calibration blank (6.10) and one calibration standard for each metal. Because of chemical interactions, the metals are divided into four combination standards.
  - 8.1.1** Load S0 (6.10)
  - 8.1.2** Load S1 (6.11)
  - 8.1.3** Load S2 (6.12)
  - 8.1.4** Load S3 (6.13)
  - 8.1.5** Load S4 (6.14)
- 8.2** Analyze an ICV (6.20).
- 8.3** Analyze a LLRLCS (6.20.1).

- 8.4** Analyze an ICB (6.10).
- 8.5** Analyze a RLCS (6.19).
- 8.6** Analyze an ICSA (6.17).
- 8.7** Analyze an ICSAB (6.18).
- 8.8** Analyze a CCV (6.20).
- 8.9** Analyze a CCB (6.10).

## **9.0 SAMPLE HANDLING AND PRESERVATION**

- 9.1** Samples for total recoverable metals should have been preserved by acidification with nitric acid to a pH < 2 or lower immediately upon collection in the field or upon being accepted by Sample Receiving as per SVL 2001.
  - 9.1.1** Samples that have been put on hold due to the 24-hour desorb period after preservation, may not be removed for analysis prior to Sample Receiving releasing the container(s) as per SVL 2001 section 8.15.
  - 9.1.2** Samples for dissolved analysis are to be filtered through a 0.45 µm filter upon collection or as soon thereafter as practically possible. After filtration preserve sample by acidification with nitric acid to a pH < 2.
- 9.2** Soil samples are preserved by refrigeration (held between 0-6 °C) when the client instructs. There is no published holding time for soils.

## **10.0 SAMPLE PREPARATION AND ANALYSIS**

- 10.1** If a preserved aqueous sample has a turbidity of less than 1 NTU, it may be analyzed directly, without digestion. Direct analysis requires some bench prep:
  - 10.1.1** Each batch must be accompanied by a preparation blank, LCS, matrix spike and matrix spike duplicate.

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- 10.1.2** Add 0.1 mL of concentrated nitric acid and 10 mL of sample to each vial.
- 10.1.3** The matrix spike and LCS are prepared as above with the addition of 100 µL of each of the five spike solutions (6.6) as needed for the batch.
- 10.2** Samples for dissolved metals must be filtered and preserved prior to analysis (acidify with nitric acid to a 1% (v/v)). These samples do not require digestion.
- 10.2.1** Each batch must be accompanied by a preparation blank, LCS, matrix spike and matrix spike duplicate. The preparation blank and LCS must also be filtered.
- 10.3** All other aqueous samples must be digested prior to analysis. SOPs covering digestive procedures are as follows: If the analytical method is 6010C, SOP SVL 4079 (EPA method 3010A) should be used for total metals, SOP SVL 4080 (EPA method 3005A) should be used for total recoverable metals, if the analytical method is 200.7, SOP SVL 4106 should be used for total recoverable metals.
- 10.3.1** For aqueous samples containing silver at concentrations  $\geq 0.1$  mg/L the sample will need to be re-digested to a level below 0.1 mg/L and re-analyzed. The diluted silver sample results shall be reported.
- 10.4** Soil samples by EPA 6010C must be digested by SOP SVL 4094 prior to analysis.
- 10.4.1** Soil samples containing  $> 50$  mg/kg of silver will also need to be diluted and re-digested below 50 mg/kg and re-ran on the ICP. The diluted silver sample results shall be reported.
- 10.4.2** If difficulties are noticed with the recovery of antimony, barium, lead or silver an alternative preparation method following section 7.5 of EPA 3050B may be used.
- 10.5** Soil samples by EPA 200.7
- 10.5.1** Transfer a portion of the sample to a weigh dish and perform a percent solid test on it by SVL SOP 4022.
- 10.5.2** To achieve homogeneity, take a dried portion of the sample and ground it up using a mortar and pestle.

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**10.5.3** To a 100 ml snap-top digestion container add 4 mL of (1+1) HNO<sub>3</sub> and 10 mL of (1+4) HCL. Cover the lip of the container with a watch glass. Place the snap-top in the digestion block. Reflux at 85 °C.

**10.5.3.1** Heat the sample and gently reflux for 30 minutes. Avoid vigorous boiling of the sample. Allow the sample to cool and bulk it up to 100 mL with de-ionized water.

**10.5.4** Allow samples to stand overnight or filter using a 45 µm filter before running on the ICP.

**10.6** Double-click on the “WinLab” icon to start the program.

**10.7** Ignite the plasma.

**10.7.1** Click “Tools” at the top of the screen and click “Plasma Control,” or click the “Plasma” icon on the Toolbar. The “Plasma Control” window will appear.

**10.7.2** Click the “Plasma” switch in the “Plasma Control” window to ON.

**10.7.3** Immediately examine the plasma through the viewing window to ensure that it is stable.

**10.7.3.1** If the plasma is not stable, click the “Plasma” switch to OFF, or press F9. Address any potential problems and restart the plasma, observing its stabilization again.

**10.7.4** Allow the plasma to stabilize for about thirty minutes before analyzing samples.

**10.8** Perform a mercury realignment.

**10.8.1** Click “Tools” at the top of the WinLab window. A drop-down menu will appear.

**10.8.2** Click “Spectrometer Control.” The “Spectrometer Control” window will appear.

**10.8.3** Select “Axial.”

**10.8.4** Click the “Hg Realign” button at the bottom of the “Spectrometer Control” window. The “Hg Re-alignment” dialog box will appear.

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**10.8.5** Click "OK." The alignment is finished when "Shutter Closed" appears in the spectrometer box.

**10.9** Align the torch viewing position.

**10.9.1** Click "Tools" at the top of the "WinLab" window; then click "Spectrometer Control." The "Spectrometer Control" window will appear.

**10.9.2** Select "Radial."

**10.9.3** Click "Align View" in the "Spectrometer Control" window. A dialog box will appear.

**10.9.4** Select "Manganese."

**10.9.5** Set the "Read Delay" time to 45 seconds.

**10.9.6** Aspirate a 10-ppm solution of manganese (6.23).

**10.9.7** The instrument will automatically position the torch viewing position.

**10.9.8** The alignment is finished when "Shutter Closed" appears in the spectrometer box.

**10.9.9** Select "Axial" on the "Spectrometer Control" window.

**10.9.10** Click "Align View." The dialog box will appear.

**10.9.11** Select "Manganese."

**10.9.12** Set the "Read Delay" time to 45 seconds.

**10.9.13** Aspirate a 1-ppm solution of manganese (6.24).

**10.9.14** The instrument will automatically position the torch viewing position. The alignment is finished when "Shutter Closed" appears in the spectrometer box.

**10.9.15** Click "File." Select "Print," then click on "New Page." This will print the rest of the alignment and start the run data on a new page.

**10.10** Open the method to be used for analysis.

**10.10.1** Click "File" at the top of the window. A drop-down menu will appear.

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**10.10.2** Click “Open Method...” A dialog box will appear.

**10.10.3** Click the name of the method to be used as appropriate for the analysis.

**10.10.4** Click “OK.”

**10.11** Prepare a Sample Information File for the job.

**10.11.1** Click “File” at the top of the window. A drop-down menu will appear.

**10.11.2** Click “Open Sample Info File...” The “Open Sample Information” dialog box will appear.

**10.11.3** Select the pattern Sample Info File.

**10.11.4** Click “Open.”

**10.11.5** Click the “SamInfo” icon, or click “Tools” at the top of the window, then “Sample Information Editor.” The “Sample Information File” will appear.

**10.11.6** Type the initials of the operator next to “Analyst Name” in the “Parameters Common to All Samples” section.

**10.11.7** Type the SVL job number next to “Batch ID” in the “Parameters Common to All Samples” section and next to “JOB#.”

**10.11.8** Type the EPA method number next to “EPA METHOD#” in the “Parameters Common to All Samples” section.

**10.11.9** Type the sample number of each sample to be analyzed in the “Sample ID” column. Do not enter calibration standards, blanks, RLCS, ICSA, ICSAB, ICV, ICB, CCV, or CCB. These have already been listed in the “Calibration” and “QC” pages of the method.

**10.11.9.1** If a sample requires a dilution it should be entered as the sample ID followed by “@xxX.” For example, a 10X dilution of sample 1234567 would be 1234567@10X.

**10.11.10** Type the autosampler location for each sample in the “A/S Location” column.

**10.11.11** Enter matrix spike recovery checks as necessary.

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**10.11.12** Add PB and LCS using the QC page of the method. Open "Schedule QC's" and select the appropriate PB or LCS. Open "QC Sample Definition", scrolling to the right until the PB/LCS locations are found, and enter the PB and LCS ID labels into the appropriate positions.

**10.12** Prepare the "Automated Analysis Control" window for the run.

**10.12.1** Click the "Auto" icon. The "Automated Analysis Control Set Up" page will appear.

**10.12.2** The selected method will already appear in the "Method" column.

**10.12.3** The number of minutes the system should wait before it starts the method will already appear in the "Delay (min)" column. This may be 0.0.

**10.12.4** The name of the sample information file to be used will already appear. If not, click the "Open" tab next to "Sample Information File."

**10.12.5** "All Defined" will already appear in the "Sample Info File" column. If not, check "Use Sample Info" under the name of the sample information file. A drop-down menu will appear. To analyze all samples according to the specified sample information file, select "All Defined" from the drop-down menu. To analyze only certain samples, select "Sample Nos.," then list the sample numbers in the "Sample Nos." column.

**10.12.6** Click "Use Method in Memory" to use the method you specified.

**10.12.7** Click the "Open" button next to "Results Data Set Name." The "Select Results Data Set" dialog box will appear.

**10.12.8** Type a file name for the results to be generated. This will be the last two digits of the year and the day number of that year, followed by a letter designation coinciding with the run for the day (A for the first calibration/run, B for the second, C for the third, etc. For example the first calibration/run for January 3, 2009 would be 09003A. Then click "OK."

**10.12.9** If desired to automatically shut down after the run is complete, click the "Set" tab next to "Auto Shutdown."

**10.12.10** To print a report of data during analysis, check the box next to "Print Log During Analyses."



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- 10.12.11** Click the “Analyze” tab at the bottom of the “Automated Analysis Control” window. The “Analyze” page will appear.
- 10.12.12** Click “Rebuild List” to transfer the contents of the Sample Info File to the “Analyze” page.
- 10.13** Load samples and QC standards in the autosampler tray.
- 10.14** Place the loaded tray on the autosampler.
- 10.15** Click “Analyze All” on the “Automated Analysis Control: Analyze” page to analyze the full sequence of calibration standards, QC, and samples. The instrument will automatically analyze in the following order: zero-standard, calibration standards, ICV, ICB, RLCS, ICS-A, ICS-AB, CCV, CCB, samples.
- 10.16** To stop an analysis in the course of a run, click “Analyze All” button again. The “Stopping an Analytical Sequence” dialog box will appear. Select when the analysis will stop, either “Stop immediately” or “Complete current replicate,” or “complete all replicates for current sample.” Then click “OK.”
- 10.17** To re-start the analysis where it was stopped, click “Analyze All” again. The “Continuing an Analytical Sequence” dialog box will appear. Select “Continue with next sequence#,” “Re-analyze previous sequence # and continue,” or “Continue with sequence # n.” Then click “OK.”
- 10.18** To add another sample to the analytical sequence, click the “Priority” button at the bottom of the “Automated Analysis Control Analyze” page. A dialog box will appear. Enter the sample information. Select an option from the “When to Analyze” menu, then click “Add Sample.”
- 10.19** To re-analyze a calibration standard manually during the run:
  - 10.19.1** Click the “Reset Sequence” button at the top of the “Automated Analysis Control: Analyze” page. Then close the “Automated Analysis Control” window.
  - 10.19.2** Click “Tools” at the top of the window and then click “Manual Analysis Control.” Or click the “Manual” icon. The “Manual Analysis Control” window will appear.
  - 10.19.3** Select the standard to be re-analyzed.
  - 10.19.4** Click the “Analyze Standard” tab.
- 10.20** To re-analyze a QC sample (like a CCV or a CCB) during the course of an analysis, click the “Analyze Samples” button. The “Stopping an Analytical

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Sequence” dialog box will appear. Select “Reanalyze previous number.”  
Then click “OK.”

**10.21 Instrument maintenance.**

- 10.21.1** Each day, flush the sample introduction system with 2% nitric acid for five minutes with the plasma on.
- 10.21.2** Each day check the chiller to make sure it has been filled with distilled (not deionized!) water. On the advice of Perkin-Elmer technicians, SVL does not add 1.8 grams chloramine-T to prevent algae growth as stated in the operation manual.
- 10.21.3** Each day check the torch to make sure there are no deposits or signs of melting.
- 10.21.4** Change the peristaltic pump tubing after about 24 hours of operation, but not in the middle of a calibration sequence.
- 10.21.5** Clean the torch at least once a week, or as necessary. Remove the copper foil igniter and soak the torch in 5% (no more than 20%) nitric acid or aqua regia. Rinse thoroughly. Then dry it with clean air or nitrogen.
- 10.21.6** Inspect the torch O-rings when cleaning the torch. If necessary, clean them with soap and water. Replace them if they are cracked or worn.
- 10.21.7** Check the nebulizer spray pattern with deionized water at least once a week. If necessary, clean the nebulizer with soap and water, then rinse thoroughly.
- 10.21.8** Clean the spray chamber when necessary. Soak the chamber in 5% (no more than 20%) nitric acid or aqua regia. Rinse thoroughly.
- 10.21.9** Clean the spectrometer and generator air filters every month. Replace them if necessary.
- 10.21.10** Flush the chiller every six months. Replace the water filter every three months, or as needed.
- 10.21.11** Log maintenance in the appropriate maintenance log.

## 11.0 DATA REDUCTION

- 11.1 The software creates a linear calibration curve and calculates results from that curve. Results must be corrected manually for sample dilution.
- 11.2 Check the instrument calculated percent recoveries of the matrix spikes and the LCS, ensuring they are within control parameters. If needed re-run or post spike a QC sample.
- 11.3 Check and flag as necessary the relative percent differences (RPDs) of the sample duplicates when processing the data in Element.

## 12.0 DATA AND RECORDS MANAGEMENT

- 12.1 The raw data will print to a PDF file as the run proceeds. IT IS IMPORTANT TO NOT CLOSE THIS APPLICATION WINDOW WHILE THE RUN IS STILL ACTIVE. The software overwrites the file with the new complete file (including the newest page) with each new page. IF YOU CLOSE THE "PDF PRO SOFTWARE" WHILE IT IS SAVING, THE FILE WILL BE LOST.
  - 12.1.1 The PDF Pro software will open automatically when the instrument software sends the first page to the printer during the startup alignments. After the alignments are complete go to "File," "Print" and "New Page." This will start the run data on a new page. The PDF Pro software will open automatically after the first standard (blank) runs for second and subsequent analytical runs.
  - 12.1.2 Change the file name in the "Doc Info" tab of the PDF Pro window. The "Title" will be the same as the instrument file name (IE 09001A). *It is very important this name be correct. If the file is misnamed the same as an existing file it (the previous file) will be overwritten with the new data and the previous file will be lost.*
  - 12.1.3 The previous day's file should be moved into the appropriate folder (i.e. folder Jan 2009 for file 09001A) either at the end of the run or before the beginning of the next run (or start of the day). This will prevent an accidental overwrite as mentioned above.
- 12.2 Print the run log to the same PDF file after the run is complete by clicking on "Close" after running the macro for run logs. An additional paper copy

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will need to be printed for the run log book. After clicking "Close" the preview window will close and the paper copy can be obtained by going to the "File," then "Print." The "Printer" selection will need to be changed to the appropriate printer (i.e. HP Laserjet Series 4050 PCL for the Optima 1). Click "Print" after changing the printer selection.

**12.2.1** If a paper printout is required for any application it must be printed from the "File," then the "Print" window and then under "Printer," the "Name" must be changed to the appropriate printer.

**12.3** File the run log in the run log book.

**12.4** Print the Standards Log page associated with that run to the appropriate PDF file. Also print the ICP Control Sheet associated with that run to the same PDF file.

**12.5** When analysis is complete, create a transfer file and upload ICP data to Element.

**12.5.1** Click "File" at the top of the WinLab window. A drop-down menu will appear.

**12.5.2** Click "Utilities Data Manager." The "Data Manager" window will appear with a list of data files in the "D:\pe\Default User\Results..." section. If not, click the "Library Category" button to ensure that "Results" appears in the box to its right. Then click the "Library Name" button to ensure that "D:\pe\Default User\Results..." appears in the box to its right.

**12.5.3** Click on the appropriate "Results Data" set name.

**12.5.4** Click the "Export" icon on the tool bar. The "Data Export Wizard" will appear.

**12.5.5** Click the button next to "Use Existing Design."

**12.5.6** Click the "Browse..." button.

**12.5.7** For level 1 and 2 jobs use the "LIMS\_Export.xpt" template. See section 12.8 for Level 3 data upload instructions.

**12.5.8** Click the "Open" button. The "Data Export Wizard" window will re-appear.

**12.5.9** Click the "Next" button. The "Select Samples to Export" window will appear.

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- 12.5.10** Select “Enable/Disable Samples.” The “Enable/Disable Samples” window will appear.
  - 12.5.11** Double-click the column header “Enabled.” The “Enabled Column Fill” menu will appear.
  - 12.5.12** Click “OK.”
  - 12.5.13** Click the box to the left of each sample necessary for the job.
  - 12.5.14** Click the “Next” button. The “Select Analytes to Export” window will appear.
  - 12.5.15** All analytes should be selected, if not select “All Analytes.”
  - 12.5.17** Click the “Next” button. The “Select Export Options” window will appear.
  - 12.5.18** Check the second of the “Name:” choices (**not** “Same as Data Set”) and fill the field with the appropriate batch number.
  - 12.5.19** Click the “Next” button. The “Select Sample Parameters” window will appear.
  - 12.5.20** Click the “Next” button. The “Select Mean-Related Parameters” window will appear.
  - 12.5.21** Click the “Next” button. The “Select Replica-Related Parameters” window will appear.
  - 12.5.22** Click the “Next” button. The “Export Data Set” window will appear.
  - 12.5.23** Click the “Export Data” button. A pop-up window briefly appears, showing a file folder.
  - 12.5.24** Click the “Finish” button.
  - 12.5.25** Click the “Export” button.
  - 12.5.26** Close the Data Manager.
- 12.6** Uploading Level 3 Data
- 12.6.1** Click “File” at the top of the WinLab window. A drop-down menu will appear.

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- 12.6.2** Click “Utilities Data Manager.” The “Data Manager” window will appear with a list of data files in the “D:\pe\Default User\Results...” section. If not, click the “Library Category” button to ensure that “Results” appears in the box to its right. Then click the “Library Name” button to ensure that “D:\pe\Default User\Results...” appears in the box to its right.
- 12.6.3** Click on the appropriate “Results Data” set name.
- 12.6.4** Click the “Export” icon on the tool bar. The “Data Export Wizard” will appear.
- 12.6.5** In window “1. Select Export Design” click on the “Browse” button.
- 12.6.6** Select the “MARRS.xpt” by double clicking in it. Click “Next” after ensuring the MARRS.xpt path is in the window and the “Use Existing Design” choice is selected. This should happen automatically.
- 12.6.7** In window “2. Select Samples to Export” click the “Enable/Disable” button. Disable any samples not in the relevant batch/work order. This is done by clicking the boxes in the “Enabled” column, making the red check mark disappear from the grey box. **All Calibration and QC samples must be included in the export.** Click “OK” followed by “Next” once the “2. Select Samples to Export” window reopens.
- 12.6.8** In window “3. Select Analytes to Export” add or remove analytes as is appropriate for the batch/work order at hand. **Lu must be included in addition to any target analytes.** Click “Next.”
- 12.6.9** In window “4. Select Export Options” type the batch number in the ‘Exported File: Name: box. The “Overwrite” choice should be selected for the “If File Already Exists:” selection.
- 12.6.10** For subsequent data files for reanalysis (e.g. in the case of all the required analysis not being valid as a result of failed QC, instrument failure or any other cause) the appropriate samples must be reanalyzed and re-exported. These data files will be labeled with the batch number followed by a letter designation starting with ‘A’ for the second analytical run (‘B’ for the third, etc.)
- 12.6.11** Click “Finish.” From window “8. Export Data Set” click the “Export Data” Button. This exports the file to the “H:” drive. Click “Finish.”

- 12.7** Uploading ICP data into Element.
- 12.7.1** Open Element, click on “Laboratory” and then select “Data Entry / Review.”
  - 12.7.2** Locate and select the batch number from the list on the left of the “Laboratory-Enter/Edit Data (Metals Batches)” Element window. Click “Create” followed by “Data Tool.”
  - 12.7.3** The “Select Data System Files” Data Tool window will open automatically. Select the file number by double clicking on the relevant file name (e.g. W701001.pm) from the “PE Winlab” window at the lower right. Sample ID numbers will appear in the ‘File Name/Sample Information’ window to the upper left. Click “Auto Select” and verify the necessary samples have moved down to the field directly below. If any samples didn’t move down that are needed (primarily because of a labeling issue) highlight the sample numbers and click the “Include” button. Click the “Done” button.
  - 12.7.4** From the “Data Tool – Main” window click on “Merge Files.” Check that the “Merged Upload” and “Empty Upload” tabs should have matching sample ID numbers. If sample ID numbers need to be changed click the “Instrument Data” tab followed by selection of the “Lab\_Number” column. To change the sample IDs to the correct number(s) right click on the highlighted “Lab\_Number” column and select “Replace.” Choose the incorrect sample label in the “Search for:” field then choose the correct label from the “Replace with:” field. Click “Replace” followed by “Done.” Next click “Refresh” then “Save.” A “Save As” box will pop up. Click on a file (e.g. AAA) and “Save.” Return to “Data Entry / Review” screen and click “Save,” “OK” in the “Element Data System – Data Post Results” pop-up then “Query.”
  - 12.7.5** The analyst shall perform all reviews on the “Data Entry/Review” page in Element and verify their data uploads.
  - 12.7.6** If an input comes up color coded apply the appropriate data flags and/or undertake any corrective actions.
  - 12.7.7** The analyst shall assign any qualifiers as necessary, which include but are not limited to: dilutions, RPD/RSD and/or spike recovery issues.
  - 12.7.8** The analyst will then lock the results so that any future imports will not overwrite acceptable results.

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- 12.7.9** The analyst will then update the status of the batch to “Analyzed.”
- 12.7.10** The raw data is stored both on the instrument and on the network as a PDF file, which is located in a folder on the H: drive named for the respective instrument on which it was analyzed (H:\Optima1\PDF FILES for example).
- 12.7.11** Data review is outlined in SVL SOP 2009.
- 12.8** Procedures for constructing bench sheets can be found at R:\Promium Stuff\How to’s\Batching.doc. Make sure that the bench sheet is initialed and dated when the actual preparation of the samples began.
- 12.8.1** Indicate all reagents used in the batch by including them in the reagent section of the “Batch” screen.
- 12.9** Procedures for constructing sequences can be found at R:\Promium Stuff\How to’s\Sequences.doc.
- 12.10** Corrective action is governed by SOP SVL 1019.

**13.0 QUALITY CONTROL**

<b>Method</b>	<b>LCS</b>	<b>RPD</b>	<b>Spike</b>	<b>Prep Blank</b>
<b>200.7</b>	Limits: 85 to 115%	Limit: 20%	Limits: 70 to 130%	Limit: less than half of the reporting limit <sup>1</sup>
	Action: re-run, then re-prep if outside limits	Action: flag if higher	Action: flag if outside recovery limits	Action: re-run, then re-prep if outside limits

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<sup>1</sup> For California 200.7 samples, the method blank must be less than 2.2 x MDL or 10% of the lowest sample concentration.



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Method	LCS	RPD	Spike	Prep Blank
<b>6010C</b>	Limits: 80 to 120%	Limit: 20%	Limits: 75% To 125%	Limit: less than half of the reporting limit
	Action: re-run, then re-prep if outside limits	Action: flag if higher	Action: flag, then run analytical (post) spike; failed post spike requires serial dilution	Action: re-run, then re-prep if outside limits

Method	ICV	ICB	CCV	CCB
<b>200.7</b>	Limit: within 5% of expected; 3% RSD between replicates	Limit: less than half of the reporting limit	Limit: within 10% of expected	Limit: less than half of the reporting limit
	Action: re-run then re-calibrate	Action: re-run, then re-calibrate and re-analyze	Action: re-run, then re-calibrate and re-analyze	Action: re-run, then re-calibrate and re-analyze
<b>6010C</b>	Limit: within 10% of expected; 5% RSD between replicates	Limit: less than half of the reporting limit	Limit: within 10% of expected	Limit: less than half of the reporting limit
	Action: re-run then re-calibrate	Action: re-run, then re-calibrate	Action: re-run, then re-calibrate and re-analyze	Action: re-run, then re-calibrate and re-analyze

**13.1** For method 200.7, the recovery of the ICV (6.20) must be within 5% of the expected value for target analytes. For method 6010C, the recovery must be within 10% of the expected value for target analytes. In addition, the relative percent difference of the ICV replicate integrations must be 3% or less for method 200.7 and 5% or less for method 6010C. If the recovery falls outside these criteria, re-analyze the ICV. If the recovery still falls outside these criteria, re-calibrate the instrument before proceeding with the analysis. Additionally, for 200.7, a LLRLCS will be analyzed for silver

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at a concentration of 0.4 mg/L that must meet the same 200.7 criteria as above.

- 13.2** Recovery of the ICB (6.10) must be less than one-half the reporting limit, if the recovery exceeds this criterion, re-run the ICB. If the recovery still exceeds this limit, re-calibrate the instrument before proceeding with analysis. This is a deviation from method 200.7, which requires that the ICB be less than the IDL, but greater than the lower 3-sigma control limit of the calibration blank (section 9.3.4 of method 200.7).
- 13.3** Analyze an RLCS (required for 200.7 drinking water and 6010C samples) (6.21) after the calibration. The acceptance limits are plus or minus 50% of the expected value. If the recovery exceeds the criteria re-run immediately, if it fails again re-calibrate the instrument before proceeding with analysis or do not report failed analytes from the run.
- 13.4** Analyze an ICSA (6.17) and an ICSAB (6.18) immediately after the RLCS standard. For the ICSA and ICSAB, the recovery of the target analytes must be within 20% of the expected value. If the recovery of any analyte exceeds the 20% criteria, re-run the solution with the failed analytes. For the ICSA all non target analytes should be within  $\pm 2x$  the EPA's CRQL; if not, adjust IECs to minimize interferences. If the recovery still exceeds the criteria, fix the problem and re-calibrate the instrument before proceeding with analysis.
- 13.4.1** For 200.7 Drinking Waters the ICSA and ICSAB must also be run at the end of the batch, before the last CCV and CCB.
- 13.5** Analyze a prep blank at a frequency of one per batch of 20 or fewer samples. The concentration of each target analyte in the prep blank must be less than half the reporting limit, or less than 10% of the concentration of the analyte in all samples. If the recovery of a target analyte in the prep blank exceeds these criteria, re-analyze the prep blank. If the recovery in the prep blank still exceeds the criteria, re-digest and re-analyze the samples associated with the prep blank.
- 13.5.1** For California method 200.7 samples, the prep blank must be less than 2.2 x MDL or 10% of the lowest sample concentration.
- 13.6** Analyze an LCS (6.6) at a frequency of one per batch of 20 or fewer samples. For method 200.7, the recovery for the LCS must be between 85 and 115% of the expected value. The recovery of the LCS for method 6010C must be within 80 to 120% of the expected value, if the recovery falls outside the above criteria, re-run the LCS. If the recovery still falls outside these criteria, re-digest and re-analyze the samples associated with the LCS.

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The expected concentration of the LCS will vary depending on the stock solutions used to prepare it. If several stock solutions are used, each may contribute to the final concentration. Make sure all spikes are accounted for on the benchsheet so that an accurate summation of the analytes spiked takes place.

**13.6.1** If QC19 is used, the expected concentrations are:

As 1.0 mg/L  
Be 1.0 mg/L  
Ca 1.0 mg/L  
Cd 1.0 mg/L  
Co 1.0 mg/L  
Cr 1.0 mg/L  
Cu 1.0 mg/L  
Fe 1.0 mg/L  
Mg 1.0 mg/L  
Mn 1.0 mg/L  
Mo 1.0 mg/L  
Ni 1.0 mg/L  
Pb 1.0 mg/L  
Sb 1.0 mg/L  
Se 1.0 mg/L  
Ti 1.0 mg/L  
Tl 1.0 mg/L  
V 1.0 mg/L  
Zn 1.0 mg/L

**13.6.2** If SVL 11 is used, the expected concentrations are:

Al 1.0 mg/L  
B 1.0 mg/L  
Ba 1.0 mg/L  
Bi 1.0 mg/L  
Ga 1.0 mg/L  
La 1.0 mg/L  
Li 1.0 mg/L  
P 1.0 mg/L  
Si 5.0 mg/L  
Ag 0.05 mg/L  
Sr 1.0 mg/L

**13.6.3** If N5 is used, the expected concentrations are:

Ca 19 mg/L

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Fe 9 mg/L  
K 20 mg/L  
Mg 19 mg/L  
Na 19 mg/L

**13.6.4** If QC-Sc is used, the expected concentration is:

Sc 0.5 mg/L

**13.6.5** If QC-Sn is used, the expected concentration is:

Sn 1.0 mg/L

**13.6.6** If TCLP7 is used, the expected concentration is:

Ag 1.0 mg/L  
As 1.0 mg/L  
Ba 20 mg/L  
Cd 0.2 mg/L  
Cr 1.0 mg/L  
Pb 1.0 mg/L  
Se 0.2 mg/L

**13.7** For method 200.7 analyze a Matrix Spike (MS) at a frequency of one per batch of 10 or fewer samples. Analyze a Matrix Spike Duplicate (MSD) at a frequency of 1 per batch of 20 or fewer samples. Acceptance limits for spike recoveries for both the MS and MSD is 70 to 130% of the expected value. Recovery calculations are not required if the concentration added is less than 30% of the sample background concentration.

**13.7.1** The spike concentration for the MS will mirror the LCS concentration (see 13.6).

**13.7.2** The control limit for the relative percent difference (RPD) between the MS and MSD is 20%.

**13.7.3** If the spike recovery or RPD exceed the above criterion, flag the data on the client's report.

**13.8** For method 6010C analyze an MS and MSD at a frequency of 1 per batch of 20 or fewer samples. Acceptance limits for spike recoveries for both the MS and MSD is 75 to 125% of the expected value, if recovery is outside this range flag the client's report. The control limit for the relative percent difference (RPD) between the MS and MSD is 20%, if the RPD

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is outside the 20% criteria flag the clients' report. If the spike recovery is outside the 75 to 125% do the following before reporting results out to clients.

- 13.8.1** If the added analyte concentration is less than 30% of the sample concentration then a post spike will not be required. The results will be flagged accordingly.
- 13.8.2** Analyze a post spike (analyte spike) by spiking to a portion of a prepared sample, or its dilution. Acceptance limits for the post spike should be between 80 to 120%. The level of this spike will be between 10 and 100 times the reporting limit (post spikes will be made from single analyte standards (6.5)). If the post spike fails then run a dilution test. Failure of the post spike will result in flagging the client's report.
- 13.8.3** If the failing analyte concentration is above 10 times the reporting limit, then a dilution test at a 1:5 ratio of the original sample will be run. If the results of the dilution and the original sample don't agree within  $\pm 10\%$  then a matrix effect is confirmed and will be indicated via a case narrative or a qualifier on the report. If the dilution test passes then there is no confirmation of matrix interference and the qualifiers for the spikes indicate to the client the recovery problems.
- 13.9** Method 6010C requires a RLCS (6.21) be analyzed at the end of each analysis run (prior to the CCV and CCB) with a recovery limit of 50 to 150%. If the calibration cannot be verified within these specified limits, the analysis of samples containing the affected analytes at less than 5 times the reporting limit must be reanalyzed. Sample results greater than 5 times the reporting limit can be reported.
- 13.10** Analyze a CCV (6.20) at a frequency of one per 10 samples or fewer, and at the end of an analytical run. The recovery must be within 10% of the expected value for target analytes. Reportable analytes must be bracketed by acceptable CCVs, if the recovery falls outside these criteria, determine the cause, perform corrective action, and re-analyze it. If the recovery still falls outside the criteria, re-analyze all samples run since the last successful CCV.
- 13.11** Analyze a CCB (6.10) immediately after each CCV. The recovery must be less than one-half the reporting limit. Reportable analytes must be bracketed by acceptable CCBs, if the recovery exceeds this criterion, determine the cause, perform corrective action, and re-analyze the CCB. If the recovery still exceeds the limit, re-analyze all samples run since the last successful CCB. This is a deviation from method 200.7, which

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requires that the CCB be less than the IDL, but greater than the lower 3-sigma control limit of the calibration blank (section 9.3.4 of method 200.7).

- 13.12** Monitor the response from the internal standard throughout the sample set being analyzed. The ratio of the response of the internal standard in any sample to the response in the zero calibration standard must be between 60 and 125%. If the ratio lies outside this range, dilute the sample by a factor of two to five and re-analyze it.
- 13.13** Perform an aqueous and a soil MDL study annually. See SOP 1011 "Performing an MDL Study".
- 13.14** Determine the linear dynamic range every 12 months for all elements and every six months for Ca, Cu, Fe, Mg, Mn, Na, Pb, and Zn.
- 13.14.1** Calibrate the instrument in the normal manner.
- 13.14.2** Analyze three successively higher standards with the highest near the upper limit.
- 13.14.3** If the recovery of the higher standard is within 10% of the expected concentration, it is within the linear range of the instrument.
- 13.14.4** If the recovery of the higher standard deviates more than 10% from the expected value, analyze a lower-concentration standard to determine the linear range.
- 13.15** Determine inter-element correction factors for each element every quarter using single-element standards.
- 13.16** Trend analysis can be found in SOP SVL 1033.

**14.0 REFERENCES**

- 14.1** Method 6010C, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Third Edition, Update III, December 1996.
- 14.2** Method 200.7, Revision 4.4, Methods for the Determination of Metals in Environmental Samples—Supplement I, Method Update Rule, May, 18 2012.
- 14.3** Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition.

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**14.4** Perkin-Elmer Instruments, Optima 4000 Series Hardware Guide, Part No. 0993-6373, July 2000.

**14.5** Perkin-Elmer Instruments, WinLab32 Instrument Control Software Guide, Part No. 0993-6335, January 2000.

**15.0 POLLUTION PREVENTION**

**15.1** All standards are prepared and reagents used in volumes consistent with good laboratory practice to minimize the volume of disposable waste.

**16.0 WASTE MANAGEMENT**

**16.1** Most chemicals used during digestion and/or analysis are neutralized and/or diluted prior to disposal by permit to the public sewer. Any hazardous chemicals and/or residues are disposed of through SVL's hazardous waste disposal system (see SOPs SVL 1001 & 1008).

**17.0 CHANGE HISTORY**

<b>DATE</b>	<b>VER.</b>	<b>CHANGE</b>
08/26/10	10.0	6.9 changed to "Standard 1 Mix: Bi, Ga, Li, Sn and Sr at 500 µg/mL as well as P at 1000 µg/mL, in 10% HCl and Tr HNO <sub>3</sub> . High-Purity Standards SM-150-060. Expiration date as stated by the manufacturer". 6.10 added "(Seq-Cal1@S0)". 6.11 added "(Seq-Cal 2@S)" and "Standard 1 Mix". 6.12 added "(Seq-Cal 3@S)". 6.13 added "(Seq-Cal 4@S)" and "10 mL of Standard 3 Mix (6.26) and 2.5 mL Si stock solution". 6.14 added "(Seq-Cal 5@S)". 6.26 added "Standard 3 Mix: La at 1000 ug/mL and Sc ug/mL at 200 in 2% HNO <sub>3</sub> , High-Purity Standards SM-150-061. Expiration date as stated by the manufacturer". 9.1.1 added " Samples that have been put on hold due to the 24-hour desorb period after preservation, may not be removed for analysis prior to Sample Receiving releasing the container(s) as per SVL 2001 section 8.15". 12.8 Added "Procedures for constructing bench sheets can be found

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<b>DATE</b>	<b>VER.</b>	<b>CHANGE</b>
		at R:\Promium Stuff\How to's\Batching.doc. Make sure that the bench sheet is initialed and dated when the actual preparation of the samples began". 12.8.1 Added "Indicate all reagents used in the batch by including them in the reagent section of the "Batch" screen". 12.9 Added "Procedures for constructing sequences can be found at R:\PromiumStuff\Howto's\Sequences.doc". 13.4 changed to "For the ICSEA all non target analytes should be within $\pm 2x$ the EPA's CRQL".
8/26/11	11.0	10.4.2 added "If difficulties are noticed with the recovery of antimony, barium, lead or silver an alternative preparation method following section 7.5 of EPA 3050B may be used".



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DATE	VER.	CHANGE
03/01/13	12.0	<p>2.0 added "6010B may also be run under this SOP." 6.20.1 added "Low Level Initial Check Verification (LLICV) solution: Add deionized water (6.25) to a 1000 mL volumetric flask. Carefully add 20 mL concentrated nitric acid (6.4) and 50 mL concentrated hydrochloric acid (6.3). Add 0.4 mL of 1000 ppm silver stock solution (6.5). Dilute to the mark with deionized water and mix well. Make sure to use a secondary source or a different lot number than was used for the calibration standards." 6.20.2 added " Note: The lower silver concentration (0.4 mg/L) is for the LLICV." 9.1 inserted "total recoverable." 9.1.2 changed to "Samples for dissolved analysis are to be filtered through a 0.45 µm filter upon collection or as soon thereafter as practically possible. After filtration preserve sample by acidification with nitric acid to a pH &lt; 2." 10.2 inserted "acidify with nitric acid to a 1% (v/v)." 10.2.1 added "Each batch must be accompanied by a preparation blank, LCS, matrix spike and matrix spike duplicate. The preparation blank and LCS must also be filtered." 13.1 added "Additionally, for 200.7, a LLICV will be analyzed for silver at a concentration of 0.4 mg/L that must meet the same 200.7 criteria as above." 13.3 changed to "Analyze an RLCS (required for 200.7 drinking water and 6010C samples) (6.21) after the calibration. The acceptance limits are plus or minus 50% of the expected value for 200.7 and plus or minus 30% of the expected value for 6010C." 13.6 added "6010C must be within 80 to 120% of the expected value". Re-wrote 13.7 thru 13.9 please read the text of the document for current instructions.</p>
03/27/13	12.1	<p>13.3 changed to "The acceptance limits are plus or minus 50% of the expected value." 13.9 changed to "Method 6010C requires an RLCS (6.21) be analyzed at the end of each analysis run (prior to the CCV and CCB) with an expected recovery between 50% and 150%. If the recovery exceeds these limits, one of two possibilities will occur. Samples with similar values (those that are less than 5 times the reporting limit) will be re-analyzed</p>

**ANALYSIS OF METALS BY METHODS 6010C and 200.7 USING THE PERKIN-ELMER  
OPTIMA ICP**

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<b>DATE</b>	<b>VER.</b>	<b>CHANGE</b>
		with passing QC. Samples that are not similar (meaning greater than 5 times the reporting limit) shall be reported as run.”
01/15/14	13.0	Added in all of the requirements for running mercury by 6010 C. 12.10 added “Corrective action is governed by SOP SVL 1019.” 13.6 added “Trend analysis can be found in SOP SVL 1033.” 13.7 added “Demonstration of capability requirements can be found in SOP SVL 1010.”
01/14/15	13.1	No changes.
10/21/15	14.0	Removed verbiage related to the running of gold and mercury by EPA 200.7. Changed CRI to RLCS throughout document.

## Appendix E Quality Assurance Manual for ESC Laboratory

# Quality Assurance Manual



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**Version 14.0 9/1/15**

**COMPREHENSIVE QUALITY ASSURANCE MANUAL**

for

**ESC LAB SCIENCES  
12065 LEBANON ROAD  
MT. JULIET, TENNESSEE 37207  
(615)758-5858**

Prepared by

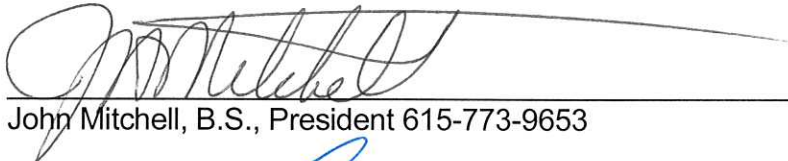
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**NOTE: The QAM has been approved by the following people.**




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*The ESC QAM has been prepared in accordance with the following standards: AIHA (LQAP), A2LA (Env. Prog. Req.), ANSI/ISO 17025-2005, 2003 NELAC Standard, 2009 TNI Standard, and DOD QSM.*

## **Disclaimer**

The ESC Lab Sciences Quality Assurance Manual is a living document. It is reviewed at least annually and revised when needed. The information stated herein is subject to change at any time due to updates to QC Limits, methods, operations, equipment, staff, etc. At the time of distribution the requestor will receive the most recent version of the manual and will be assigned a control number. The control number will help ESC to track what version is sent. The revision number is stated on the cover page of the manual.

## **Expiration**

This manual expires 1 year from the date listed at the front of the manual on the “Approvals” page. If you have a copy that is not dated within this time period, please contact the laboratory and obtain the most recent version.

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## **1.0 GENERAL**

### **1.1 INDEX AND REVISION STATUS**

The numbering of this quality manual corresponds directly to the numbering of ISO 17025:2005 with cross-references to the 2003 National Environmental Laboratory Accreditation Conference (NELAC) Standard and the 2009 standard of The NELAC Institute (TNI).

This quality manual is only valid if all pages are at the same issue level as shown in the index of the quality manual.

Updates to this manual are made by re-issuing the relevant section of this manual and adapting the issue level in the index. New version numbers are assigned upon revision.

**NOTE: This manual expires 1 year from the date listed at the beginning of the manual on the “Approvals” page.**

### **1.2 PURPOSE**

This quality manual documents the laboratory’s management system and demonstrates the ability to execute the indicated tests and/or procedures and to meet regulatory requirements.

This manual establishes compliance with ISO (International Organization for Standardization) 17025, The NELAC Institute (TNI), Department of Defense Quality Systems Manual (DOD QSM), and the American Industrial Hygiene Association (AIHA).

## **2.0 LABORATORY BACKGROUND**

### **2.1 ACTIVITIES**

#### **2.1.1 Analytical Support and Service Areas**

ESC Lab Sciences is an environmental analytical firm providing technical and support services to clients nationwide. Specific service areas include the following:

- drinking water analysis
- industrial wastewater analysis
- hazardous waste characterization and identification
- groundwater analysis
- air analysis
- regulatory document guidance
- biological assessments
- mold identification
- solid/soil analysis and characterization
- industrial hygiene/environmental lead
- aquatic toxicity analysis
- cryptosporidium/giardia

#### **2.1.2 Regulatory Compliance and Quality Standards**

ESC is devoted to providing reliable and accurate data recognizing the necessity to establish sound, objective, and legally defensible positions or opinions for clients regarding compliance with governing regulations. ESC maintains quality systems that are compliant with the following Quality Standards: AIHA LQAP, A2LA, ANSI/ISO/IEC 17025, The TNI Standard, DOD QSM. The effectiveness of the quality system is measured by accreditation maintenance, internal and external audits, management reviews, proficiency sample testing, and an active preventive/corrective action system.

#### **2.1.3 Analytical Capabilities:**

Where mandated, only approved procedures are used for environmental analyses. ESC utilizes a number of method sources to accomplish project requirements. For NPDES and SDWA, methodologies are taken directly from 40 CFR parts 136 and 141.

For industrial hygiene analytical procedures, ESC utilizes guidance from NIOSH and OSHA published methods.



The following list is an example of the methodology ESC routinely performs:

<i>Routine Methodology and Programs</i>	
<b>PROGRAM</b>	<b>METHOD SOURCE</b>
NPDES	EPA 821/R-93-010-A <i>Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume I. Revision 1, August 1993.</i>
	40 CFR part 136
	<i>Methods for Chemical Analysis of Water and Wastes (March 1983)</i>
AQUATIC TOXICITY	<i>Standard Methods for the Examination of Water and Wastewater (20<sup>th</sup> through 22<sup>nd</sup> editions)</i>
	7-Day Fathead Minnow ( <i>Pimephales promelas</i> ) Larval Survival and Growth Test; Test Method 1000.0 from "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (EPA 821-R-02-013).
	3-Brood <i>Ceriodaphnia dubia</i> Survival and Reproduction Test; Test Method 1002.0 from "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (EPA 821-R-02-013).
	Fathead Minnow ( <i>Pimephales promelas</i> ) Acute Toxicity Test (24, 48 or 96 hour duration); referenced in "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms" (EPA 821-R-02-012, 10-02).
	<i>Ceriodaphnia dubia</i> Acute Toxicity Test (24, 48 or 96 hour duration); referenced in "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms" (EPA 821-R-02-012, 10-02).
SDWA	40 CFR parts 141
	<i>Methods for Chemical Analysis of Water and Wastes (March 1983)</i>
	<i>Standard Methods for the Examination of Water and Wastewater (20<sup>th</sup> through 22<sup>nd</sup> editions)</i>
	<i>Methods for the Determination of Organic Compounds in Drinking Water - EPA/600/4-88/039 - December 1988 (Revised July 1991)</i>
	<i>Methods for the Determination of Organic Compounds in Drinking Water Supplement I, EPA/600/4-90/020 - July 1990</i>
	<i>Methods for the Determination of Organic Compounds in Drinking Water Supplement II, EPA/600/R-92/129 - August 1992</i>
	EPA. Method 1622: <i>Cryptosporidium</i> in Water by Filtration/IMS/FA, December 2005.
	EPA. Method 1623: <i>Cryptosporidium</i> and <i>Giardia</i> in Water by Filtration/IMS/FA, December 2005.
RCRA	SW-846, <i>Test Methods for Evaluating Solid Wastes (3<sup>rd</sup>, 4<sup>th</sup> and online editions)</i>

<i>Routine Methodology and Programs</i>	
<b>PROGRAM</b>	<b>METHOD SOURCE</b>
<i>AIR</i>	<i>Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air</i>
	<i>Emission Measurement Center (Air Emissions Methods)</i>
	<i>NIOSH Manual of Analytical Methods (4<sup>th</sup> edition)</i>
	<i>Journal of Chromatographic Science, Vol. 36, May 1998.</i>
	<i>OSHA Sampling and Analytical Methods (online)</i>
<i>CLP</i>	<i>USEPA CONTRACT LABORATORY PROGRAM - STATEMENT OF WORK FOR ORGANICS ANALYSIS Multi-Media, Multi-Concentration OLM04.3</i>
	<i>USEPA CONTRACT LABORATORY PROGRAM - STATEMENT OF WORK FOR INORGANIC ANALYSIS Multi-Media, Multi-Concentration ILM05.3</i>
<i>MOLD</i>	<i>American Industrial Hygiene Association</i>
<i>Miscellaneous</i>	<i>American Society for Testing and Materials (ASTM)</i>
	<i>State Specific Methodologies from the following: Florida, Oregon, Iowa, Washington, Texas, Arizona, Massachusetts, North Carolina, Louisiana, Missouri, Kansas, Wisconsin, Ohio</i>
<i>Miscellaneous</i>	<i>Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewater, Revision A EPA-821-B-98-016 - July 1998 (Approved at 40 CFR Part 136, Not Approved at Part 141)</i>

## 2.2 HISTORY

ESC Lab Sciences was founded in 1970 by Dr. Arthur Schulert, a professor of Biochemistry at Vanderbilt University Medical School. The laboratory's first location was a 2,000 square foot building located in Mt. Juliet, TN.

ESC initially conducted several research contracts for the National Science Foundation. EPA Clean Water and Safe Drinking Water legislation of the early 1970s provided an additional market of Tennessee utilities and industries. ESC grew slowly for several years by increasing the share of the drinking and wastewater markets in Tennessee. In the late 1980s, ESC expanded its capabilities to include Underground Storage Tank testing and Biomonitoring/Toxicity testing.

Strategic expansion of the laboratory allowed ESC to provide support to large RCRA sites and add capabilities to offer analytical support for air and mold analyses. ESC is currently the nation's largest, single-location environmental laboratory operating in all US states. Our staff of over 250 employees works out of our 87,000 square feet, nine-building facility approximately 20 minutes east of Nashville International Airport.

### 3.0 INTRODUCTION, SCOPE, AND DEFINITIONS

#### 3.1 SCOPE OF CAPABILITIES

A list of approved and certified analytical capabilities can be found at the end of this section in Table 3.3b.

#### 3.2 TABLE OF CONTENTS, REFERENCES AND APPENDICES

The table of contents is found at the beginning of this Manual. This Quality Manual uses the references from the 2003 NELAC Standard, Chapter 5, Appendix A and the 2009 TNI Standard (EL-V1M2-ISO-2009, Section 3.0).

#### 3.3 DEFINITIONS AND TERMINOLOGY

The source of some of the definitions is indicated previous to the actual definition.

<b>Table 3.3a Definitions</b>	
Acceptance Criteria	TNI and DoD- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI and DoD- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.
Accrediting Authority	DoD- The Territorial, State or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation.
Accrediting (or Accreditation) Body	DoD- Authoritative body that performs accreditation.
Accuracy	TNI and DoD- 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 that are due to sampling and analytical operations; a data quality indicator.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI and DoD- 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.
Analyte	DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
Analytical Reagent Grade	Designation for the high purity of certain chemical reagents and solvents assigned by the American Chemical Society.

Analytical Sensitivity	The lowest concentration that can be detected by the method. (e.g., for methods involving a count = 1 raw count calculated to the reporting units). Analytical sensitivity is commonly used in Mold analysis.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- The evaluation process used to measure the performance or effectiveness of a system and its elements against specific criteria. Note: In this standard (DoD), assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	DoD- A process in which a sample is converted to free atoms.
Audit	TNI- 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. DoD- A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.
Batch	TNI and DoD- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A <b>preparation batch</b> is composed of one to 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 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 20 samples.
Bias	TNI- 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).
Blank	TNI and DoD- 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.
Blind Sample	DoD- A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.

Calibration	TNI and DoD- 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. 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 test 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	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. DoD- The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Factor	The ratio of the detector response (peak areas or peak heights) to the amount (mass) of analyte in the calibration standard.
Calibration Method	DoD- A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration. DoD- A substance or reference material used to calibrate an instrument.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. DoD- A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.
Chain of Custody	DoD- An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of custody Form (COC)	TNI and DoD- 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 type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	DoD- Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.

Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is:  $\% \text{ Completeness} = (\text{Valid Data Points} / \text{Expected Data Points}) * 100$
Confirmation	TNI and DoD- 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.
Conformance	DoD- 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.
Congener	DoD- A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a CVS in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence.

Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Data Audit	DoD- A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e. that they meet specified acceptance criteria).
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form. DoD- The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.
Definitive Data	DoD- Analytical data of known quality, concentration and level of uncertainty. The levels of quality and uncertainty of the analytical data are consistent with the requirements for the decision to be made. Suitable for final decision-making.
Demonstration of Capability	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate acceptable accuracy.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate is 1%.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat) to convert the sample to a more easily measured form.
Document Control	DoD- 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 is performed.
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	DoD- The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	DoD- A solvent used to carry the components of a mixture through a stationary phase.
Elute	DoD- To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	DoD- A process in which solutes are washed through a stationary phase by movement of a mobile phase.

Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	DoD- The process of measuring or collecting environmental data.
Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: <ul style="list-style-type: none"> <li>• Non Potable Water ( Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts)</li> <li>• Drinking Water - Delivered (treated or untreated) water designated as potable water</li> <li>• Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents</li> <li>• Sludge - Municipal sludges and industrial sludges.</li> <li>• Soil - Predominately inorganic matter ranging in classification from sands to clays.</li> <li>• Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes</li> </ul>
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
External Calibration Model	Comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the corresponding analytes in calibration standards.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.
False Positive	DoD- An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.



Finding	<p>TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.</p> <p>DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative and is normally accompanied by specific examples of the observed condition. Note: For DoD, the finding must be linked to a specific requirement.</p>
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	<p>TNI- The maximum time that can elapse between two specified activities.</p> <p>40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised.</p> <p>For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure.</p> <p>DoD- The time elapsed from the time of sampling to the time of extraction or analysis, or from extraction to analysis, as appropriate.</p>
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	DoD- One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.

Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP-AES that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- A standard obtained or prepared from a source independent of the source of the standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.
Inspection	DoD- An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic.
Instrument Blank	DoD- A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	DoD- Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	DoD- Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.
Interference Check Sample (ICS)	A series of two solutions, used in ICP and ICPMS analysis, to verify that inter-element interferences are compensated for correctly. This standard is referred to as the Spectra Interference Check (SIC) in EPA Method 200.7 <ul style="list-style-type: none"> <li>• ICSA – A solution containing only the interfering analytes at high concentrations.</li> <li>• ICSAB – A solution containing interferences plus other method analytes at the level of concern, which corresponds to the project specific action limits.</li> </ul> ICSA and ICSAB provide an adequate test of inter-element correction (IEC) factors.
Internal Calibration Model	Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific internal standard analytes added to the sample or sample extract prior to injection.
Internal Standards	TNI and DoD- 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 method.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.

International System of Units (SI)	DoD- The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	DoD- One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties. For example, hexane (C <sub>6</sub> H <sub>14</sub> ) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.
Laboratory	DoD- A body that calibrates and/or tests.
Laboratory Control Sample (LCS)	TNI and DoD- (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 and taken through all sample preparation and analytical 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 evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	DoD- Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	A computer system that is used to maintain all sample information from sample receipt, through preparation and analysis and including sample report generation.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.
Limit(s) of Detection (LOD)	TNI- 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. DoD- The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate is 1%.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.
Lot	A quantity of bulk material of similar composition processed or manufactured at the same time.
Management	DoD- Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	DoD- System to establish policy and objectives and to achieve those objectives.
Manager (however named)	DoD- The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.

Matrix	TNI and DoD- The substrate of a test sample. For information is provided in the definition of Quality System Matrix below.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- 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. DoD- A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate 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 (MSD) (spiked sample or fortified sample duplicate)	TNI and DoD- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurement System	TNI and DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI and DoD- 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 (MDL)	DoD- One way to establish a Detection Limit; defined as the minimum concentration of a substance 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.
Method of Standard Additions	DoD- A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.
MintMiner	Software used to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.

Negative Control	DoD- Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	DoD- An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Percent Recovery	A comparison between the observed value and the true value of a known spiked concentration, represented as a percentage. This evaluation applies to the calculation of ICV, CCV, LCS, MS/MSD, Surrogates, etc.
Performance Audit	DoD- 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.
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	DoD- Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Detection Limit (PDL)	Another term for method detection limit (MDL) or limit of detection (LOD). However, a PDL might not be statically derived and could be set using an in-house protocol.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit or limit of quantitation (LOQ). The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI and DoD- 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	TNI- Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. DoD- Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.

Proficiency Testing	TNI and DoD- 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.
Proficiency Testing Program	TNI and DoD- 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.
Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria. DoD- A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.
Protocol	TNI and DoD- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Quality Assurance (QA)	TNI- 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 and quality needed and expected by the client. DoD- An integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.
Quality Assurance Manual (QAM)	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.
Quality Assurance Project Plan (QAPP)	DoD- 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.
Quality Control (QC)	TNI- 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. DoD- The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of the users.
Quality Control Sample (QCS)	TNI- 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. DoD- 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.

Quality Manual	TNI and DoD- 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.
Quality System	TNI and DoD- 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 quality assurance and quality control activities.
Quality System Matrix	<p>TNI and DoD- These matrix definitions are to be used for purposes of batch and quality control requirements:</p> <ul style="list-style-type: none"> <li>• <b>Air and Emissions:</b> 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</li> <li>• <b>Aqueous:</b> Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts.</li> <li>• <b>Biological Tissue:</b> Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin.</li> <li>• <b>Chemical Waste:</b> A product or by-product of an industrial process that results in a matrix not previously defined.</li> <li>• <b>Drinking Water:</b> Any aqueous sample that has been designated a potable or potentially potable water source.</li> <li>• <b>Non-aqueous liquid:</b> Any organic liquid with &lt;15% settleable solids</li> <li>• <b>Saline/Estuarine:</b> Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.</li> <li>• <b>Solids:</b> Includes soils, sediments, sludges, and other matrices with &gt;15% settleable solids.</li> </ul>
Quantitation Range	DoD- The range of values in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard. The quantitation range lies within the calibration range.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.

Raw Data	<p>TNI- 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.</p> <p>DoD- Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.</p>
Reagent Blank (method reagent blank)	<p>DoD- A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.</p>
Reagent Grade	<p>Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.</p>
Reference Material	<p>TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.</p> <p>DoD- A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.</p>
Reference Standard	<p>TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.</p> <p>DoD- A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.</p>
Reference Toxicant	<p>DoD- The toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the test correctly and obtain consistent results.</p>
Relative Percent Difference (RPD)	<p>A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.</p>
Replicate Sample	<p>The analytical measurement of a sample that has been split after it has been processed through the preparation stage. A replicate can also originate from a single sample that has been sub-sampled two or more times during the same analytical process time.</p>
Reporting Limit (RL)	<p>The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e. statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL.</p> <p>DoD- A client-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.</p>



Reporting Limit Verification Standard (or otherwise named)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	DoD- Denotes a mandatory specification; often designated by the term "shall".
Response Factor (RF)	A measure of the relative response area of an analyte compared to its internal standard. The response factor is determined by the equation below, and if the calculated value meets the method guidelines it can be used to determine concentration for organic analyses.
Retention Time	DoD- The time between sample injection and the appearance of a solute peak at the detector.
Sample	DoD- Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Blank (or Turbidity Blank)	The purpose of a sample blank is to account for spectrophotometric interferences such as sample color, cloudiness, viscosity, etc. The sample blank must be analyzed at the same dilution as the sample. The sample blank is analyzed without any addition of reagents.
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain of custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Secondary Source Calibration Verification (SSCV)	A mid-point or low standard made from the secondary source (lot or manufacturer) that is not used to construct the calibration curve. The SSCV is used to represent the calibration accuracy of the instrument and must perform within method stated guidelines. This sample is used to document calibration accuracy. The SSCV can be the same solution as the LCS, but is analyzed as an instrument standard, rather than a method prepared standard.
Selective Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector.
Selectivity	TNI- 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. DoD- The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.

Sensitivity	TNI and DoD- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled.
Should	Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
Signal-to-Noise Ratio	DoD- The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude.
Spike	DoD- 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 (Document)	TNI and DoD- The document describing the elements of a 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.
Standard (Chemical)	DoD- Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	DoD- A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that 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. DoD- A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.
Standard Reference Material (SRM)	DoD- A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.

Supervisor	DoD- The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes specifically named by a client (also called project-specific analytes).
Technical Director	DoD- Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Tentatively Identified Compound (TIC)	Compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. TICs can be tentatively identified using mass spectrometers in spectral comparisons with NBS library searches. Quantitation of TICs provides a rough approximation of the concentration of these non-target analytes.
Test	DoD- A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	DoD- An adoption of a scientific technique for performing a specific measurement as documented in a laboratory SOP or as published by a recognized authority.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- 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 conditions 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. DoD- The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.

Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	DoD- A check and/or adjustment of instrument performance for mass spectrometry as required by the method.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty Measurement	The parameter associated with the result of a measurement that characterized the dispersion of the values that could be reasonably attributed to the measurand (i.e. the concentration of an analyte).
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	TNI and DoD- Confirmation by examination and objective evidence that specified requirements have been met. Note: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

**Table 3.3b**  
**Analytical Capabilities**

*AE=Air Emissions, DW=Drinking Water, NPW=Non-potable Water, SCM=Solid Chemical Materials*

*The information listed is subject to change.*

*Always check with the laboratory for the most updated information.*

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
AE	EPA TO-15	Ethanol
AE	EPA TO-15	Gasoline range organic
AE	EPA TO-15	Naphthalene
AE	EPA TO-15	Allyl chloride
AE	EPA TO-15	Chlorotoluene (2-)
AE	EPA TO-15	Isopropylbenzene
AE	EPA TO-15	Methyl methacrylate
AE	EPA TO-15	Tetrahydrofuran
AE	EPA TO-15	Vinyl bromide
AE	EPA TO-15	Dibromoethane (1,2-) (EDB)
AE	EPA TO-15	Dichloroethene (1,1-)
AE	EPA TO-15	Hexachlorobutadiene (1,3-)
AE	EPA TO-15	Hexanone (2-)
AE	EPA TO-15	Acetone
AE	EPA TO-15	Chloromethane
AE	EPA TO-15	Dibromochloromethane
AE	EPA TO-15	Dichlorodifluoromethane
AE	EPA TO-15	Dichloroethene (cis-1,2-)
AE	EPA TO-15	Dichloroethene (trans-1,2-)
AE	EPA TO-15	Dichloropropene (trans-1,3-)
AE	EPA TO-15	Dichlorotetrafluoroethane (1,2-)
AE	EPA TO-15	Ethylbenzene
AE	EPA TO-15	Ethyltoluene (4-)
AE	EPA TO-15	Isopropanol
AE	EPA TO-15	Trichlorofluoromethane
AE	EPA TO-15	Trimethylpentane (2,2,4-)
AE	EPA TO-15	Vinyl chloride
AE	EPA TO-15	Benzene
AE	EPA TO-15	Benzyl chloride
AE	EPA TO-15	Bromodichloromethane
AE	EPA TO-15	Bromoform
AE	EPA TO-15	Bromomethane

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
AE	EPA TO-15	Butadiene (1,3-)
AE	EPA TO-15	Carbon disulfide
AE	EPA TO-15	Carbon tetrachloride
AE	EPA TO-15	Chlorobenzene
AE	EPA TO-15	Chloroethane
AE	EPA TO-15	Chloroform
AE	EPA TO-15	Cyclohexane
AE	EPA TO-15	Dichlorobenzene (1,2-)
AE	EPA TO-15	Dichlorobenzene (1,3-)
AE	EPA TO-15	Dichlorobenzene (1,4-)
AE	EPA TO-15	Dichloroethane (1,1-)
AE	EPA TO-15	Dichloroethane (1,2-)
AE	EPA TO-15	Dichloropropane (1,2-)
AE	EPA TO-15	Dichloropropene (cis-1,3-)
AE	EPA TO-15	Dioxane (1,4-)
AE	EPA TO-15	Heptane (n-)
AE	EPA TO-15	Hexane (n-)
AE	EPA TO-15	Methyl ethyl ketone
AE	EPA TO-15	Methyl isobutyl ketone (MIBK)
AE	EPA TO-15	Methyl tert-butyl ether
AE	EPA TO-15	Methylene chloride (Dichloromethane)
AE	EPA TO-15	Styrene
AE	EPA TO-15	Trichlorobenzene (1,2,4-)
AE	EPA TO-15	Trimethylbenzene (1,3,5-)
AE	EPA TO-15	Trimethylbenzene (1,2,4-)
AE	EPA TO-15	Tetrachloroethane (1,1,2,2-)
AE	EPA TO-15	Tetrachloroethene
AE	EPA TO-15	Toluene
AE	EPA TO-15	Trichloroethane (1,1,1-)
AE	EPA TO-15	Trichloroethane (1,1,2-)
AE	EPA TO-15	Trichloroethene
AE	EPA TO-15	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
AE	EPA TO-15	Vinyl acetate
AE	EPA TO-15	Xylene (m-)
AE	EPA TO-15	Xylene (o-)
AE	EPA TO-15	Xylene (p-)
AE	EPA TO-15	Xylenes (total)
AE/NPW	8015M/ RSK-175	Ethane
AE/NPW	8015M/ RSK-175	Ethene
AE/NPW	8015M/ RSK-175	Methane

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
AE/NPW	8015M/ RSK-175	Propane
AE/NPW	8015M/ RSK-175	Acetylene
DW	EPA 150.1	pH
DW	EPA 1622	Cryptosporidium
DW	EPA 1623	Cryptosporidium
DW	EPA 1623	Giardia
DW	EPA 180.1	Turbidity
DW	EPA 200.7	Aluminum
DW	EPA 200.7	Antimony
DW	EPA 200.7	Arsenic
DW	EPA 200.7	Barium
DW	EPA 200.7	Beryllium
DW	EPA 200.7	Boron
DW	EPA 200.7	Cadmium
DW	EPA 200.7	Calcium
DW	EPA 200.7	Calcium-hardness
DW	EPA 200.7	Total hardness
DW	EPA 200.7	Chromium
DW	EPA 200.7	Cobalt
DW	EPA 200.7	Copper
DW	EPA 200.7	Iron
DW	EPA 200.7	Lead
DW	EPA 200.7	Magnesium
DW	EPA 200.7	Manganese
DW	EPA 200.7	Molybdenum
DW	EPA 200.7	Nickel
DW	EPA 200.7	Potassium
DW	EPA 200.7	Selenium
DW	EPA 200.7	Silica
DW	EPA 200.7	Silver
DW	EPA 200.7	Sulfur
DW	EPA 200.7	Sodium
DW	EPA 200.7	Strontium
DW	EPA 200.7	Thallium
DW	EPA 200.7	Tin
DW	EPA 200.7	Titanium
DW	EPA 200.7	Vanadium
DW	EPA 200.7	Zinc
DW	EPA 200.8	Aluminum
DW	EPA 200.8	Antimony

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
DW	EPA 200.8	Arsenic
DW	EPA 200.8	Barium
DW	EPA 200.8	Beryllium
DW	EPA 200.8	Boron
DW	EPA 200.8	Cadmium
DW	EPA 200.8	Calcium
DW	EPA 200.8	Chromium
DW	EPA 200.8	Cobalt
DW	EPA 200.8	Copper
DW	EPA 200.8	Iron
DW	EPA 200.8	Lead
DW	EPA 200.8	Magnesium
DW	EPA 200.8	Manganese
DW	EPA 200.8	Molybdenum
DW	EPA 200.8	Nickel
DW	EPA 200.8	Potassium
DW	EPA 200.8	Selenium
DW	EPA 200.8	Silver
DW	EPA 200.8	Sodium
DW	EPA 200.8	Strontium
DW	EPA 200.8	Thallium
DW	EPA 200.8	Thorium
DW	EPA 200.8	Tin
DW	EPA 200.8	Titanium
DW	EPA 200.8	Uranium
DW	EPA 200.8	Vanadium
DW	EPA 200.8	Zinc
DW	EPA 218.6	Chromium (VI)
DW	EPA 218.7	Chromium (VI)
DW	EPA 245.1	Mercury
DW	EPA 300.0	Nitrite
DW	EPA 300.0	Nitrate
DW	EPA 300.0	Fluoride
DW	EPA 300.0	Sulfate
DW	EPA 300.0	Bromide
DW	EPA 300.0	Chloride
DW	EPA 314.0	Perchlorate
DW	EPA 335.4	Cyanide
DW	EPA 350.1	Ammonia
DW	EPA 353.2	Nitrate



<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
DW	EPA 353.2	Nitrite
DW	EPA 504.1	Dibromoethane (1,2-) (EDB)
DW	EPA 504.1	Dibromo-3-chloropropane (1,2-)
DW	EPA 507	Alachlor
DW	EPA 507	Butachlor
DW	EPA 507	Metolachlor
DW	EPA 507	Metribuzin
DW	EPA 507	Atrazine
DW	EPA 507	Simazine
DW	EPA 524.2	Tetrahydrofuran
DW	EPA 524.2	Dichloro-2-butene (trans-1,4-)
DW	EPA 524.2	Hexachloroethane
DW	EPA 524.2	Acetone
DW	EPA 524.2	Butanone (2-)
DW	EPA 524.2	Carbon disulfide
DW	EPA 524.2	Hexanone (2-)
DW	EPA 524.2	Pentanone (4-methyl-2-) (MIBK)
DW	EPA 524.2	Trichlorobenzene (1,3,5-)
DW	EPA 524.2	Bromochloromethane
DW	EPA 524.2	Bromoform
DW	EPA 524.2	Chloroform
DW	EPA 524.2	Dibromochloromethane
DW	EPA 524.2	Bromodichloromethane
DW	EPA 524.2	Benzene
DW	EPA 524.2	Carbon tetrachloride
DW	EPA 524.2	Chlorobenzene
DW	EPA 524.2	Dichlorobenzene (1,2-)
DW	EPA 524.2	Dichlorobenzene (1,3-)
DW	EPA 524.2	Dichlorobenzene (1,4-)
DW	EPA 524.2	Dichloroethane (1,1-)
DW	EPA 524.2	Dichloroethane (1,2-)
DW	EPA 524.2	Dichloroethene (cis-1,2-)
DW	EPA 524.2	Dichloroethene (trans-1,2-)
DW	EPA 524.2	Methylene chloride (Dichloromethane)
DW	EPA 524.2	Dichloropropane (1,2-)
DW	EPA 524.2	Ethylbenzene
DW	EPA 524.2	Methyl tert-butyl ether
DW	EPA 524.2	Naphthalene
DW	EPA 524.2	Styrene
DW	EPA 524.2	Tetrachloroethane (1,1,2,2-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
DW	EPA 524.2	Tetrachloroethene
DW	EPA 524.2	Trichloroethane (1,1,1-)
DW	EPA 524.2	Trichloroethene
DW	EPA 524.2	Toluene
DW	EPA 524.2	Trichlorobenzene (1,2,4-)
DW	EPA 524.2	Dichloroethene (1,1-)
DW	EPA 524.2	Trichloroethane (1,1,2-)
DW	EPA 524.2	Vinyl chloride
DW	EPA 524.2	Xylenes (total)
DW	EPA 524.2	Bromobenzene
DW	EPA 524.2	Bromomethane
DW	EPA 524.2	Butyl benzene (n-)
DW	EPA 524.2	Sec-butylbenzene
DW	EPA 524.2	Tert-butylbenzene
DW	EPA 524.2	Chloroethane
DW	EPA 524.2	Chloromethane
DW	EPA 524.2	Chlorotoluene (2-)
DW	EPA 524.2	Chlorotoluene (4-)
DW	EPA 524.2	Dibromo-3-chloropropane (1,2-)
DW	EPA 524.2	Dibromoethane (1,2-) (EDB)
DW	EPA 524.2	Dibromomethane
DW	EPA 524.2	Dichlorodifluoromethane
DW	EPA 524.2	Dichloropropane (1,3-)
DW	EPA 524.2	Dichloropropane (2,2-)
DW	EPA 524.2	Dichloropropene (1,1-)
DW	EPA 524.2	Dichloropropene (cis-1,3-)
DW	EPA 524.2	Dichloropropene (trans-1,3-)
DW	EPA 524.2	Hexachlorobutadiene (1,3-)
DW	EPA 524.2	Isopropylbenzene
DW	EPA 524.2	Isopropyltoluene (4-)
DW	EPA 524.2	Propylbenzene (n-)
DW	EPA 524.2	Tetrachloroethane (1,1,1,2-)
DW	EPA 524.2	Trichlorobenzene (1,2,3-)
DW	EPA 524.2	Trichlorofluoromethane
DW	EPA 524.2	Trichloropropane (1,2,3-)
DW	EPA 524.2	Trimethylbenzene (1,2,4-)
DW	EPA 524.2	Trimethylbenzene (1,3,5-)
DW	EPA 552.2	Bromochloroacetic acid
DW	EPA 552.2	Dibromoacetic acid
DW	EPA 552.2	Dichloroacetic acid

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
DW	EPA 552.2	Monobromoacetic acid (MBAA)
DW	EPA 552.2	Monochloroacetic acid (MCAA)
DW	EPA 552.2	Trichloroacetic acid
DW	SM 2120 B	Color
DW	SM 2130 B	Turbidity
DW	SM 2150 B	Odor
DW	SM 2320 B	Alkalinity
DW	SM 2340 B	Total hardness
DW	SM 2340 C	Total hardness
DW	SM 2510 B	Conductivity
DW	SM 2540 C	Total dissolved solids (TDS)
DW	SM 3120 B	Total hardness
DW	SM 4110 B	Bromide
DW	SM 4110 B	Nitrite
DW	SM 4110 B	Nitrate
DW	SM 4110 B	Fluoride
DW	SM 4110 B	Sulfate
DW	SM 4110 B	Chloride
DW	SM 4500-Cl G	Chlorine - residual
DW	SM 4500-CN C,E	Cyanide
DW	SM 4500-CN C,G	Cyanide
DW	SM 4500-H B	pH
DW	SM 4500-NH3 G	Ammonia
DW	SM 4500-NO3 F	Nitrate
DW	SM 4500-NO3 F	Nitrite
DW	SM 4500-P E	Orthophosphate
DW	SM 5310 B	Total organic carbon (TOC)
DW	SM 5310 C	Dissolved organic carbon (DOC)
DW	SM 5310 C	Total organic carbon (TOC)
DW	SM 5320 B	Total organic halides (TOX)
DW	SM 5540 C	Foaming agents
DW	SM 5910 B	UV-absorbing compounds
DW	SM 9215B (Pour Plate)	Heterotropic Bacteria
DW	SM 9223 B (Colilert)	Total coliform / E. coli
DW	User Defined 524.2	Diisopropyl Ether [DIPE]
NPW	ASTM D6503	Enterococci
NPW	ASTM F1647-02A	Total organic carbon (TOC)
NPW	EPA 1000.0	Toxicity - chronic, FW organism
NPW	EPA 1002.0	Toxicity - chronic, FW organism

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	EPA 120.1	Specific conductance
NPW	EPA 130.1	Hardness - total as CaCO <sub>3</sub>
NPW	EPA 160.4	Residue - volatile
NPW	EPA 1657	Phorate
NPW	EPA 1657	Bolstar
NPW	EPA 1657	Chlorpyrifos
NPW	EPA 1657	Coumaphos
NPW	EPA 1657	Dichlorvos
NPW	EPA 1657	Dimethoate
NPW	EPA 1657	EPN
NPW	EPA 1657	Fensulfothion
NPW	EPA 1657	Fenthion
NPW	EPA 1657	Naled
NPW	EPA 1657	Parathion ethyl
NPW	EPA 1657	Parathion methyl
NPW	EPA 1657	Ronnel
NPW	EPA 1657	Stirofos
NPW	EPA 1657	Sulfotepp
NPW	EPA 1657	TEPP
NPW	EPA 1657	Tokuthion [Protothiofos]
NPW	EPA 1657	Trichloronate
NPW	EPA 1658	D (2,4-)
NPW	EPA 1658	Dalapon
NPW	EPA 1658	Dichlorprop
NPW	EPA 1664A & B	Oil & grease - hem-SPE
NPW	EPA 1664A & B	Oil & grease - non polar
NPW	EPA 1664A & B	Oil & grease - hem-LL
NPW	EPA 1664A & B	Oil & grease - sgt-non polar-SPE
NPW	EPA 180.1	Turbidity
NPW	EPA 200.7	Aluminum
NPW	EPA 200.7	Antimony
NPW	EPA 200.7	Arsenic
NPW	EPA 200.7	Barium
NPW	EPA 200.7	Beryllium
NPW	EPA 200.7	Boron
NPW	EPA 200.7	Cadmium
NPW	EPA 200.7	Calcium
NPW	EPA 200.7	Calcium-hardness
NPW	EPA 200.7	Total hardness
NPW	EPA 200.7	Chromium

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	EPA 200.7	Cobalt
NPW	EPA 200.7	Copper
NPW	EPA 200.7	Iron
NPW	EPA 200.7	Lead
NPW	EPA 200.7	Lithium
NPW	EPA 200.7	Magnesium
NPW	EPA 200.7	Manganese
NPW	EPA 200.7	Molybdenum
NPW	EPA 200.7	Nickel
NPW	EPA 200.7	Potassium
NPW	EPA 200.7	Selenium
NPW	EPA 200.7	Silica
NPW	EPA 200.7	Silver
NPW	EPA 200.7	Sulfur
NPW	EPA 200.7	Sodium
NPW	EPA 200.7	Strontium
NPW	EPA 200.7	Thallium
NPW	EPA 200.7	Tin
NPW	EPA 200.7	Titanium
NPW	EPA 200.7	Vanadium
NPW	EPA 200.7	Zinc
NPW	EPA 200.8	Aluminum
NPW	EPA 200.8	Antimony
NPW	EPA 200.8	Arsenic
NPW	EPA 200.8	Barium
NPW	EPA 200.8	Beryllium
NPW	EPA 200.8	Boron
NPW	EPA 200.8	Cadmium
NPW	EPA 200.8	Calcium
NPW	EPA 200.8	Chromium
NPW	EPA 200.8	Cobalt
NPW	EPA 200.8	Copper
NPW	EPA 200.8	Iron
NPW	EPA 200.8	Lead
NPW	EPA 200.8	Magnesium
NPW	EPA 200.8	Manganese
NPW	EPA 200.8	Molybdenum
NPW	EPA 200.8	Nickel
NPW	EPA 200.8	Potassium
NPW	EPA 200.8	Selenium

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	EPA 200.8	Silver
NPW	EPA 200.8	Sodium
NPW	EPA 200.8	Strontium
NPW	EPA 200.8	Thallium
NPW	EPA 200.8	Thorium
NPW	EPA 200.8	Tin
NPW	EPA 200.8	Titanium
NPW	EPA 200.8	Uranium
NPW	EPA 200.8	Vanadium
NPW	EPA 200.8	Zinc
NPW	EPA 2000.0	Toxicity - acute, FW organism
NPW	EPA 2002.0	Toxicity - acute, FW organism
NPW	EPA 218.6	Chromium (VI)
NPW	EPA 245.1	Mercury
NPW	EPA 300.0	Guanidine nitrate
NPW	EPA 300.0	Bromide
NPW	EPA 300.0	Chloride
NPW	EPA 300.0	Fluoride
NPW	EPA 300.0	Nitrate
NPW	EPA 300.0	Nitrite
NPW	EPA 300.0	Sulfate
NPW	EPA 300.0	Nitrate - nitrite
NPW	EPA 310.2	Alkalinity as CaCO <sub>3</sub>
NPW	EPA 314.0	Perchlorate
NPW	EPA 335.4	Cyanide
NPW	EPA 350.1	Ammonia
NPW	EPA 351.1, .2 - 350.1	Organic nitrogen
NPW	EPA 351.2	Kjeldahl nitrogen - total
NPW	EPA 353.2	Nitrate - nitrite
NPW	EPA 410.4	Chemical oxygen demand
NPW	EPA 420.4	Phenols
NPW	EPA 507	Alachlor
NPW	EPA 507	Metribuzin
NPW	EPA 507	Ethoprop
NPW	EPA 507	Merphos
NPW	EPA 507	Mevinphos
NPW	EPA 602	Benzene
NPW	EPA 602	Ethylbenzene
NPW	EPA 602	Methyl tert-butyl ether
NPW	EPA 602	Tert-butyl alcohol

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	EPA 602	Toluene
NPW	EPA 602	Xylenes (total)
NPW	EPA 608	Chloroneb
NPW	EPA 608	Chlorothalonil
NPW	EPA 608	Chlordane (alpha)
NPW	EPA 608	Chlordane (gamma)
NPW	EPA 608	Hexachlorobenzene
NPW	EPA 608	PCB 1016
NPW	EPA 608	PCB 1221
NPW	EPA 608	PCB 1232
NPW	EPA 608	PCB 1242
NPW	EPA 608	PCB 1248
NPW	EPA 608	PCB 1254
NPW	EPA 608	PCB 1260
NPW	EPA 608	Aldrin
NPW	EPA 608	Alpha BHC
NPW	EPA 608	Beta BHC
NPW	EPA 608	Delta BHC
NPW	EPA 608	Lindane (gamma BHC)
NPW	EPA 608	Chlordane
NPW	EPA 608	DDD (4,4'-)
NPW	EPA 608	DDE (4,4'-)
NPW	EPA 608	DDT (4,4'-)
NPW	EPA 608	Dieldrin
NPW	EPA 608	Endosulfan I
NPW	EPA 608	Endosulfan II
NPW	EPA 608	Endosulfan sulfate
NPW	EPA 608	Endrin
NPW	EPA 608	Endrin aldehyde
NPW	EPA 608	Endrin ketone
NPW	EPA 608	Heptachlor
NPW	EPA 608	Heptachlor epoxide
NPW	EPA 608	Methoxychlor
NPW	EPA 608	Toxaphene
NPW	EPA 610	Acenaphthene
NPW	EPA 610	Acenaphthylene
NPW	EPA 610	Anthracene
NPW	EPA 610	Benzo(a)anthracene
NPW	EPA 610	Benzo(a)pyrene
NPW	EPA 610	Benzo(b)fluoranthene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	EPA 610	Benzo(ghi)perylene
NPW	EPA 610	Benzo(k)fluoranthene
NPW	EPA 610	Chrysene
NPW	EPA 610	Dibenzo(a,h)anthracene
NPW	EPA 610	Fluoranthene
NPW	EPA 610	Fluorene
NPW	EPA 610	Indeno(1,2,3-cd)pyrene
NPW	EPA 610	Naphthalene
NPW	EPA 610	Phenanthrene
NPW	EPA 610	Pyrene
NPW	EPA 615	Dicamba
NPW	EPA 615	DB (2,4-)
NPW	EPA 615	Dinoseb
NPW	EPA 615	Dalapon
NPW	EPA 615	Dichlorprop
NPW	EPA 615	D (2,4-)
NPW	EPA 615	T (2,4,5-)
NPW	EPA 615	TP (2,4,5-) (Silvex)
NPW	EPA 615	MCPA
NPW	EPA 615	MCPP
NPW	EPA 622	Coumaphos
NPW	EPA 622	Demeton (o-)
NPW	EPA 622	Demeton (s-)
NPW	EPA 622	Dimethoate
NPW	EPA 622	Parathion ethyl
NPW	EPA 622	Parathion methyl
NPW	EPA 622	Stirofos
NPW	EPA 622	Sulfotepp
NPW	EPA 622	TEPP
NPW	EPA 622	Tokuthion [Protothiofos]
NPW	EPA 622	Trichloronate
NPW	EPA 624	Amyl alcohol (n-)
NPW	EPA 624	Propionitrile
NPW	EPA 624	Trimethylbenzene (1,2,3-)
NPW	EPA 624	Allyl chloride
NPW	EPA 624	Bromoethane
NPW	EPA 624	Butanone (2-)
NPW	EPA 624	Butadiene (2-chloro-1,3-)
NPW	EPA 624	Carbon disulfide
NPW	EPA 624	Cyclohexanone



<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	EPA 624	Dichloro-2-butene (cis-1,4-)
NPW	EPA 624	Dichloro-2-butene (trans-1,4-)
NPW	EPA 624	Diethyl ether (Ethyl ether)
NPW	EPA 624	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
NPW	EPA 624	Vinyl acetate
NPW	EPA 624	Acetonitrile
NPW	EPA 624	Cyclohexane
NPW	EPA 624	Hexanone (2-)
NPW	EPA 624	Methylcyclohexane
NPW	EPA 624	Methyl iodide
NPW	EPA 624	Ethyl-tert-butyl Ether [ETBE]
NPW	EPA 624	Diisopropyl Ether [DIPE]
NPW	EPA 624	Dioxane (1,4-)
NPW	EPA 624	Butanol (1-)
NPW	EPA 624	Ethanol
NPW	EPA 624	Ethyl methacrylate
NPW	EPA 624	Iso-butyl alcohol
NPW	EPA 624	Methacrylonitrile
NPW	EPA 624	Methyl methacrylate
NPW	EPA 624	Octane (-n)
NPW	EPA 624	Pentachloroethane
NPW	EPA 624	tert-Amylmethyl ether [TAME]
NPW	EPA 624	Acrolein
NPW	EPA 624	Acrylonitrile
NPW	EPA 624	Bromobenzene
NPW	EPA 624	Bromochloromethane
NPW	EPA 624	Butyl benzene (n-)
NPW	EPA 624	Chlorotoluene (2-)
NPW	EPA 624	Chlorotoluene (4-)
NPW	EPA 624	Dibromo-3-chloropropane (1,2-)
NPW	EPA 624	Dibromoethane (1,2-) (EDB)
NPW	EPA 624	Dibromomethane
NPW	EPA 624	Dichlorodifluoromethane
NPW	EPA 624	Dichloroethene (cis-1,2-)
NPW	EPA 624	Dichloropropane (1,3-)
NPW	EPA 624	Dichloropropane (2,2-)
NPW	EPA 624	Dichloropropene (1,1-)
NPW	EPA 624	Hexane (n-)
NPW	EPA 624	Methyl isobutyl ketone (MIBK)
NPW	EPA 624	Tetrahydrofuran

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	EPA 624	Styrene
NPW	EPA 624	Tetrachloroethane (1,1,1,2-)
NPW	EPA 624	Xylene (m-)
NPW	EPA 624	Xylene (o-)
NPW	EPA 624	Xylene (p-)
NPW	EPA 624	Hexachlorobutadiene (1,3-)
NPW	EPA 624	Isopropylbenzene
NPW	EPA 624	Isopropyltoluene (4-)
NPW	EPA 624	Naphthalene
NPW	EPA 624	Propylbenzene (n-)
NPW	EPA 624	Sec-butylbenzene
NPW	EPA 624	Tert-butylbenzene
NPW	EPA 624	Trichlorobenzene (1,2,3-)
NPW	EPA 624	Trichlorobenzene (1,2,4-)
NPW	EPA 624	Trichloropropane (1,2,3-)
NPW	EPA 624	Trimethylbenzene (1,2,4-)
NPW	EPA 624	Trimethylbenzene (1,3,5-)
NPW	EPA 624	Acetone
NPW	EPA 624	Ethyl acetate
NPW	EPA 624	Methyl tert-butyl ether
NPW	EPA 624	Tert-butyl alcohol
NPW	EPA 624	Xylenes (total)
NPW	EPA 624	Benzene
NPW	EPA 624	Bromodichloromethane
NPW	EPA 624	Bromoform
NPW	EPA 624	Bromomethane
NPW	EPA 624	Carbon tetrachloride
NPW	EPA 624	Chlorobenzene
NPW	EPA 624	Chloroethane
NPW	EPA 624	Chloroethyl vinyl ether (2-)
NPW	EPA 624	Chloroform
NPW	EPA 624	Chloromethane
NPW	EPA 624	Dibromochloromethane
NPW	EPA 624	Dichlorobenzene (1,2-)
NPW	EPA 624	Dichlorobenzene (1,3-)
NPW	EPA 624	Dichlorobenzene (1,4-)
NPW	EPA 624	Dichloroethane (1,1-)
NPW	EPA 624	Dichloroethane (1,2-)
NPW	EPA 624	Dichloroethene (1,1-)
NPW	EPA 624	Dichloroethene (trans-1,2-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	EPA 624	Dichloropropane (1,2-)
NPW	EPA 624	Dichloropropene (cis-1,3-)
NPW	EPA 624	Dichloropropene (trans-1,3-)
NPW	EPA 624	Ethylbenzene
NPW	EPA 624	Methylene chloride (Dichloromethane)
NPW	EPA 624	Tetrachloroethane (1,1,2,2-)
NPW	EPA 624	Tetrachloroethene
NPW	EPA 624	Toluene
NPW	EPA 624	Trichloroethane (1,1,1-)
NPW	EPA 624	Trichloroethane (1,1,2-)
NPW	EPA 624	Trichloroethene
NPW	EPA 624	Trichlorofluoromethane
NPW	EPA 624	Vinyl chloride
NPW	EPA 625	Tetrachlorophenol (2,3,4,6-)
NPW	EPA 625	Hexachlorophene
NPW	EPA 625	Decane (n-)
NPW	EPA 625	Octadecane (n-)
NPW	EPA 625	Chloronaphthalene (1-)
NPW	EPA 625	Famphur
NPW	EPA 625	Hexachloropropene
NPW	EPA 625	Kepone
NPW	EPA 625	Napththylamine (1-)
NPW	EPA 625	Napththylamine (2-)
NPW	EPA 625	Pentachloroethane
NPW	EPA 625	Methylnaphthalene (2-)
NPW	EPA 625	Chloroaniline (4-)
NPW	EPA 625	Nitroaniline (2-)
NPW	EPA 625	Nitroaniline (3-)
NPW	EPA 625	Nitroaniline (4-)
NPW	EPA 625	Pentachlorobenzene
NPW	EPA 625	Tetrachlorobenzene (1,2,4,5-)
NPW	EPA 625	Methylphenol (4-)
NPW	EPA 625	Acetophenone
NPW	EPA 625	Aniline
NPW	EPA 625	Dichloroaniline (2,3-)
NPW	EPA 625	Diphenylhydrazine (1,2-)
NPW	EPA 625	Methylphenol (2-)
NPW	EPA 625	N-Nitroso-di-n-butylamine
NPW	EPA 625	N-Nitrosodiethylamine
NPW	EPA 625	N-Nitrosopyrrolidine

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	EPA 625	Hexachlorocyclopentadiene
NPW	EPA 625	N-Nitrosodimethylamine
NPW	EPA 625	N-Nitrosodiphenylamine
NPW	EPA 625	Dibenzofuran
NPW	EPA 625	Methylphenol (2-)
NPW	EPA 625	Methylphenol (4-)
NPW	EPA 625	Trichlorophenol (2,4,5-)
NPW	EPA 625	Benzoic acid
NPW	EPA 625	Benzidine
NPW	EPA 625	Carbazole
NPW	EPA 625	Pyridine
NPW	EPA 625	Acenaphthene
NPW	EPA 625	Acenaphthylene
NPW	EPA 625	Anthracene
NPW	EPA 625	Benzo(a)anthracene
NPW	EPA 625	Benzo(b)fluoranthene
NPW	EPA 625	Benzo(k)fluoranthene
NPW	EPA 625	Benzo(a)pyrene
NPW	EPA 625	Benzo(g,h,i)perylene
NPW	EPA 625	Butyl benzyl phthalate
NPW	EPA 625	Bis (2-chloroethyl) ether
NPW	EPA 625	Bis (2-chloroethoxy) methane
NPW	EPA 625	Bis (2-ethylhexyl) phthalate
NPW	EPA 625	Bis (2-chloroisopropyl) ether
NPW	EPA 625	Bromophenyl-phenyl ether (4-)
NPW	EPA 625	Chloronaphthalene (2-)
NPW	EPA 625	Chlorophenyl-phenyl ether (4-)
NPW	EPA 625	Chrysene
NPW	EPA 625	Dibenzo(a,h)anthracene
NPW	EPA 625	Di-n-butyl phthalate
NPW	EPA 625	Dichlorobenzidine (3,3'-)
NPW	EPA 625	Diethyl phthalate
NPW	EPA 625	Dimethyl phthalate
NPW	EPA 625	Dinitrotoluene (2,4-)
NPW	EPA 625	Dinitrotoluene (2,6-)
NPW	EPA 625	Di-n-octyl phthalate
NPW	EPA 625	Fluoranthene
NPW	EPA 625	Fluorene
NPW	EPA 625	Hexachlorobenzene
NPW	EPA 625	Hexachlorobutadiene (1,3-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	EPA 625	Hexachloroethane
NPW	EPA 625	Indeno(1,2,3-c,d)pyrene
NPW	EPA 625	Isophorone
NPW	EPA 625	Naphthalene
NPW	EPA 625	Nitrobenzene
NPW	EPA 625	N-Nitroso-di-n-propylamine
NPW	EPA 625	Phenanthrene
NPW	EPA 625	Pyrene
NPW	EPA 625	Trichlorobenzene (1,2,4-)
NPW	EPA 625	Methyl phenol (4-chloro-3-)
NPW	EPA 625	Chlorophenol (2-)
NPW	EPA 625	Dichlorophenol (2,4-)
NPW	EPA 625	Dimethylphenol (2,4-)
NPW	EPA 625	Dinitrophenol (2,4-)
NPW	EPA 625	Dinitrophenol (2-methyl-4,6-)
NPW	EPA 625	Nitrophenol (2-)
NPW	EPA 625	Nitrophenol (4-)
NPW	EPA 625	Pentachlorophenol
NPW	EPA 625	Phenol
NPW	EPA 625	Trichlorophenol (2,4,6-)
NPW	Other FL - PRO	Petroleum Organics
NPW	Other IA - OA-1	Petroleum Organics
NPW	Other IA - OA-2	Petroleum Organics
NPW	Other NJ-OQA-QAM-025	Petroleum Organics
NPW	Other NJ-OQA-QAM-025, Rev. 7	Petroleum Organics
NPW	Other NJ-OQA-QAM-025, Rev. 7	Petroleum Organics
NPW	Other NJ DEP EPH 10/08, Rev 3	Petroleum Organics
NPW	Other USDA-LOI (Loss on ignition)	Total organic carbon (TOC)
NPW	Other Walkley Black	Total organic carbon (TOC)
NPW	SM 2120 B-11	Color
NPW	SM 2130 B-11	Turbidity
NPW	SM 2310 B-11	Acidity as CaCO <sub>3</sub>
NPW	SM 2320 B-11	Alkalinity as CaCO <sub>3</sub>
NPW	SM 2340 B-11	Hardness - total as CaCO <sub>3</sub>
NPW	SM 2340 C-11	Hardness - total as CaCO <sub>3</sub>
NPW	SM 2510 B-11	Specific conductance
NPW	SM 2540 B-11	Residue - total

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SM 2540 C-11	Residue - filterable (TDS)
NPW	SM 2540 D-11	Residue - nonfilterable (TSS)
NPW	SM 2540 F-11	Residue - settleable
NPW	SM 2540 G SM 18th Ed.	Total, fixed, and volatile solids (SQAR)
NPW	SM 2550 B-00	Temperature
NPW	SM 3500-Cr B-11	Chromium (VI)
NPW	SM 3500-Cr C-11	Chromium (VI)
NPW	SM 3500-Fe B-11	Iron, Ferrous
NPW	SM 4110 B or C-11	Nitrate - nitrite
NPW	SM 4110 B or C-11	Chloride
NPW	SM 4110 B or C-11	Fluoride
NPW	SM 4110 B or C-11	Nitrate
NPW	SM 4110 B or C-11	Nitrite
NPW	SM 4110 B or C-11	Sulfate
NPW	SM 4500-Cl G-11	Chlorine
NPW	SM 4500-Cl G-11	Chlorine
NPW	SM 4500-CN B or C-11 plus E-11	Cyanide
NPW	SM 4500-CN B or C-11 and G-11	Cyanide - amenable to Cl <sub>2</sub>
NPW	SM 4500-H B-11	pH
NPW	SM 4500-N Org B or C-11 plus NH <sub>3</sub> B-11 plus NH <sub>3</sub> C-11	Kjeldahl nitrogen - total
NPW	SM 4500-NH <sub>3</sub> B plus G-11	Ammonia
NPW	SM 4500-NH <sub>3</sub> B, C, D, E, F, G, H-11	Organic nitrogen
NPW	SM 4500-NO <sub>3</sub> F-11	Nitrate - nitrite
NPW	SM 4500-O C-11	Oxygen (dissolved)
NPW	SM 4500-O G-11	Oxygen (dissolved)
NPW	SM 4500-P B5-11 plus E-11	Phosphorus (total)
NPW	SM 4500-P E-11	Orthophosphate
NPW	SM 4500-S B, C plus D-11	Sulfides
NPW	SM 4500-SO <sub>3</sub> B-11	Sulfite - SO <sub>3</sub>
NPW	SM 5210 B-11	Carbonaceous BOD (CBOD)
NPW	SM 5210 B-11	Biochemical oxygen demand
NPW	SM 5220 D-11	Chemical oxygen demand
NPW	SM 5310 B, C or D-11	Dissolved organic carbon (DOC)
NPW	SM 5310 B-11	Total organic carbon (TOC)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SM 5320 B-11	Total organic halides (TOX)
NPW	SM 5520 B-11	Oil & grease - total recov
NPW	SM 5520 B-11	Oil & grease - hem-LL
NPW	SM 5540 C-11	Surfactants
NPW	SM 6200 B-97	Propionitrile
NPW	SM 6200 B-97	Trimethylbenzene (1,2,3-)
NPW	SM 6200 B-97	Allyl chloride
NPW	SM 6200 B-97	Bromoethane
NPW	SM 6200 B-97	Butadiene (2-chloro-1,3-)
NPW	SM 6200 B-97	Cyclohexanone
NPW	SM 6200 B-97	Dichloro-2-butene (cis-1,4-)
NPW	SM 6200 B-97	Dichloro-2-butene (trans-1,4-)
NPW	SM 6200 B-97	Diethyl ether (Ethyl ether)
NPW	SM 6200 B-97	Isopropanol
NPW	SM 6200 B-97	Ethyl-tert-butyl Ether [ETBE]
NPW	SM 6200 B-97	Diisopropyl Ether [DIPE]
NPW	SM 6200 B-97	Dioxane (1,4-)
NPW	SM 6200 B-97	Ethanol
NPW	SM 6200 B-97	Ethyl methacrylate
NPW	SM 6200 B-97	Iso-butyl alcohol
NPW	SM 6200 B-97	Methacrylonitrile
NPW	SM 6200 B-97	Methyl methacrylate
NPW	SM 6200 B-97	Pentachloroethane
NPW	SM 6200 B-97	tert-Amylmethyl ether [TAME]
NPW	SM 6200 B-97	Acrolein
NPW	SM 6200 B-97	Acrylonitrile
NPW	SM 6200 B-97	Bromobenzene
NPW	SM 6200 B-97	Bromochloromethane
NPW	SM 6200 B-97	Butyl benzene (n-)
NPW	SM 6200 B-97	Chlorotoluene (2-)
NPW	SM 6200 B-97	Chlorotoluene (4-)
NPW	SM 6200 B-97	Dibromo-3-chloropropane (1,2-)
NPW	SM 6200 B-97	Dibromomethane
NPW	SM 6200 B-97	Dichlorodifluoromethane
NPW	SM 6200 B-97	Dichloropropane (1,3-)
NPW	SM 6200 B-97	Dichloropropane (2,2-)
NPW	SM 6200 B-97	Dichloropropene (1,1-)
NPW	SM 6200 B-97	Hexane (n-)
NPW	SM 6200 B-97	Methyl isobutyl ketone (MIBK)
NPW	SM 6200 B-97	Tetrahydrofuran

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SM 6200 B-97	Tetrachloroethane (1,1,1,2-)
NPW	SM 6200 B-97	Xylene (m-)
NPW	SM 6200 B-97	Xylene (p-)
NPW	SM 6200 B-97	Hexachlorobutadiene (1,3-)
NPW	SM 6200 B-97	Isopropylbenzene
NPW	SM 6200 B-97	Isopropyltoluene (4-)
NPW	SM 6200 B-97	Propylbenzene (n-)
NPW	SM 6200 B-97	Sec-butylbenzene
NPW	SM 6200 B-97	Tert-butylbenzene
NPW	SM 6200 B-97	Trichlorobenzene (1,2,3-)
NPW	SM 6200 B-97	Trichloropropane (1,2,3-)
NPW	SM 6200 B-97	Trimethylbenzene (1,2,4-)
NPW	SM 6200 B-97	Trimethylbenzene (1,3,5-)
NPW	SM 6200 B-97	Acetone
NPW	SM 6200 B-97	Ethyl acetate
NPW	SM 6200 B-97	Methyl tert-butyl ether
NPW	SM 6200 B-97	Tert-butyl alcohol
NPW	SM 6200 B-97	Benzene
NPW	SM 6200 B-97	Bromodichloromethane
NPW	SM 6200 B-97	Bromoform
NPW	SM 6200 B-97	Bromomethane
NPW	SM 6200 B-97	Carbon tetrachloride
NPW	SM 6200 B-97	Chlorobenzene
NPW	SM 6200 B-97	Chloroethane
NPW	SM 6200 B-97	Chloroform
NPW	SM 6200 B-97	Chloromethane
NPW	SM 6200 B-97	Dibromochloromethane
NPW	SM 6200 B-97	Dichlorobenzene (1,2-)
NPW	SM 6200 B-97	Dichlorobenzene (1,3-)
NPW	SM 6200 B-97	Dichlorobenzene (1,4-)
NPW	SM 6200 B-97	Dichloroethane (1,1-)
NPW	SM 6200 B-97	Dichloroethane (1,2-)
NPW	SM 6200 B-97	Dichloroethene (1,1-)
NPW	SM 6200 B-97	Dichloroethene (trans-1,2-)
NPW	SM 6200 B-97	Dichloropropane (1,2-)
NPW	SM 6200 B-97	Dichloropropene (cis-1,3-)
NPW	SM 6200 B-97	Dichloropropene (trans-1,3-)
NPW	SM 6200 B-97	Ethylbenzene
NPW	SM 6200 B-97	Methylene chloride (Dichloromethane)
NPW	SM 6200 B-97	Tetrachloroethane (1,1,2,2-)



<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SM 6200 B-97	Tetrachloroethene
NPW	SM 6200 B-97	Toluene
NPW	SM 6200 B-97	Trichloroethane (1,1,1-)
NPW	SM 6200 B-97	Trichloroethane (1,1,2-)
NPW	SM 6200 B-97	Trichloroethene
NPW	SM 6200 B-97	Trichlorofluoromethane
NPW	SM 6200 B-97	Vinyl chloride
NPW	SM 6200 B-97	Naphthalene
NPW	SM 6200 B-97	Trichlorobenzene (1,2,4-)
NPW	SM 6410 B-00	Tetrachlorophenol (2,3,4,6-)
NPW	SM 6410 B-00	Hexachlorophene
NPW	SM 6410 B-00	Decane (n-)
NPW	SM 6410 B-00	Octadecane (n-)
NPW	SM 6410 B-00	Biphenylamine (4-)
NPW	SM 6410 B-00	Chloronaphthalene (1-)
NPW	SM 6410 B-00	Famphur
NPW	SM 6410 B-00	Hexachloropropene
NPW	SM 6410 B-00	Kepone
NPW	SM 6410 B-00	Naphthylamine (1-)
NPW	SM 6410 B-00	Naphthylamine (2-)
NPW	SM 6410 B-00	Pentachloroethane
NPW	SM 6410 B-00	Napthoquinone (1,4-)
NPW	SM 6410 B-00	Methylphenol (4-)
NPW	SM 6410 B-00	Acetophenone
NPW	SM 6410 B-00	Alpha - terpineol
NPW	SM 6410 B-00	Aniline
NPW	SM 6410 B-00	Dichloroaniline (2,3-)
NPW	SM 6410 B-00	Methylphenol (2-)
NPW	SM 6410 B-00	Hexachlorocyclopentadiene
NPW	SM 6410 B-00	N-Nitrosodimethylamine
NPW	SM 6410 B-00	N-Nitrosodiphenylamine
NPW	SM 6410 B-00	Benzoic acid
NPW	SM 6410 B-00	Benzidine
NPW	SM 6410 B-00	Carbazole
NPW	SM 6410 B-00	Pyridine
NPW	SM 6410 B-00	Acenaphthene
NPW	SM 6410 B-00	Acenaphthylene
NPW	SM 6410 B-00	Anthracene
NPW	SM 6410 B-00	Benzo(a)anthracene
NPW	SM 6410 B-00	Benzo(b)fluoranthene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SM 6410 B-00	Benzo(k)fluoranthene
NPW	SM 6410 B-00	Benzo(a)pyrene
NPW	SM 6410 B-00	Benzo(ghi)perylene
NPW	SM 6410 B-00	Butyl benzyl phthalate
NPW	SM 6410 B-00	Bis (2-chloroethyl) ether
NPW	SM 6410 B-00	Bis (2-chloroethoxy) methane
NPW	SM 6410 B-00	Bis (2-ethylhexyl) phthalate
NPW	SM 6410 B-00	Bis (2-chloroisopropyl) ether
NPW	SM 6410 B-00	Bromophenyl-phenyl ether (4-)
NPW	SM 6410 B-00	Chloronaphthalene (2-)
NPW	SM 6410 B-00	Chlorophenyl-phenyl ether (4-)
NPW	SM 6410 B-00	Chrysene
NPW	SM 6410 B-00	Dibenzo(a,h)anthracene
NPW	SM 6410 B-00	Di-n-butyl phthalate
NPW	SM 6410 B-00	Dichlorobenzidine (3,3'-)
NPW	SM 6410 B-00	Diethyl phthalate
NPW	SM 6410 B-00	Dimethyl phthalate
NPW	SM 6410 B-00	Dinitrotoluene (2,4-)
NPW	SM 6410 B-00	Dinitrotoluene (2,6-)
NPW	SM 6410 B-00	Di-n-octyl phthalate
NPW	SM 6410 B-00	Fluoranthene
NPW	SM 6410 B-00	Fluorene
NPW	SM 6410 B-00	Hexachlorobenzene
NPW	SM 6410 B-00	Hexachlorobutadiene (1,3-)
NPW	SM 6410 B-00	Hexachloroethane
NPW	SM 6410 B-00	Indeno(1,2,3-cd)pyrene
NPW	SM 6410 B-00	Isophorone
NPW	SM 6410 B-00	Naphthalene
NPW	SM 6410 B-00	Nitrobenzene
NPW	SM 6410 B-00	N-Nitroso-di-n-propylamine
NPW	SM 6410 B-00	Phenanthrene
NPW	SM 6410 B-00	Pyrene
NPW	SM 6410 B-00	Trichlorobenzene (1,2,4-)
NPW	SM 6410 B-00	Methyl phenol (4-chloro-3-)
NPW	SM 6410 B-00	Chlorophenol (2-)
NPW	SM 6410 B-00	Dichlorophenol (2,4-)
NPW	SM 6410 B-00	Dimethylphenol (2,4-)
NPW	SM 6410 B-00	Dinitrophenol (2,4-)
NPW	SM 6410 B-00	Dinitrophenol (2-methyl-4,6-)
NPW	SM 6410 B-00	Nitrophenol (2-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SM 6410 B-00	Nitrophenol (4-)
NPW	SM 6410 B-00	Pentachlorophenol
NPW	SM 6410 B-00	Phenol
NPW	SM 6410 B-00	Trichlorophenol (2,4,6-)
NPW	SM 6440 B-00	Acenaphthene
NPW	SM 6440 B-00	Acenaphthylene
NPW	SM 6440 B-00	Anthracene
NPW	SM 6440 B-00	Benzo(a)anthracene
NPW	SM 6440 B-00	Benzo(a)pyrene
NPW	SM 6440 B-00	Benzo(b)fluoranthene
NPW	SM 6440 B-00	Benzo(ghi)perylene
NPW	SM 6440 B-00	Benzo(k)fluoranthene
NPW	SM 6440 B-00	Chrysene
NPW	SM 6440 B-00	Dibenzo(a,h)anthracene
NPW	SM 6440 B-00	Fluoranthene
NPW	SM 6440 B-00	Fluorene
NPW	SM 6440 B-00	Indeno(1,2,3-cd)pyrene
NPW	SM 6440 B-00	Naphthalene
NPW	SM 6440 B-00	Phenanthrene
NPW	SM 6440 B-00	Pyrene
NPW	SM 6630 B-00	Trifluralin
NPW	SM 6630 B-00	Aldrin
NPW	SM 6630 B-00	Alpha BHC
NPW	SM 6630 B-00	Lindane (gamma BHC)
NPW	SM 6630 B-00	Chlordane
NPW	SM 6630 B-00	DDD (4,4'-)
NPW	SM 6630 B-00	DDE (4,4'-)
NPW	SM 6630 B-00	DDT (4,4'-)
NPW	SM 6630 B-00	Dieldrin
NPW	SM 6630 B-00	Endosulfan I
NPW	SM 6630 B-00	Endosulfan II
NPW	SM 6630 B-00	Endrin
NPW	SM 6630 B-00	Heptachlor
NPW	SM 6630 B-00	Heptachlor epoxide
NPW	SM 6630 B-00	Methoxychlor
NPW	SM 6630 B-00	Toxaphene
NPW	SM 6630C-00	Etridiazole
NPW	SM 6630C-00	Aldrin
NPW	SM 6630C-00	Alpha BHC
NPW	SM 6630C-00	Beta BHC

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SM 6630C-00	Delta BHC
NPW	SM 6630C-00	Lindane (gamma BHC)
NPW	SM 6630C-00	Chlordane
NPW	SM 6630C-00	DDD (4,4'-)
NPW	SM 6630C-00	DDE (4,4'-)
NPW	SM 6630C-00	DDT (4,4'-)
NPW	SM 6630C-00	Dieldrin
NPW	SM 6630C-00	Endosulfan I
NPW	SM 6630C-00	Endosulfan II
NPW	SM 6630C-00	Endosulfan sulfate
NPW	SM 6630C-00	Endrin
NPW	SM 6630C-00	Heptachlor
NPW	SM 6630C-00	Heptachlor epoxide
NPW	SM 6630C-00	Methoxychlor
NPW	SM 6630C-00	Toxaphene
NPW	SM 6640 B-01	D (2,4-)
NPW	SM 6640 B-01	Dalapon
NPW	SM 6640 B-01	T (2,4,5-)
NPW	SM 6640 B-01	TP (2,4,5-) (Silvex)
NPW	SM 9215 B-00	Heterotrophic plate count
NPW	SM 9222 B-97	Total coliform
NPW	SM 9222 D-97	Fecal coliform
NPW	SM 9222D-97 (Class B only) plus EPA 625/R-92/013 App. F	Fecal coliform
NPW	SW-846 1010	Ignitability
NPW	SW-846 1010A	Ignitability
NPW	SW-846 1110	Corrosivity toward steel
NPW	SW-846 1110A	Corrosivity toward steel
NPW	SW-846 1310A	Metals - organics
NPW	SW-846 1310B	Metals - organics
NPW	SW-846 1311	Volatile organics
NPW	SW-846 1311	Semivolatile organics
NPW	SW-846 1311	Metals
NPW	SW-846 1312	Metals - organics
NPW	SW-846 1320	Metals - organics
NPW	SW-846 3005A	Metals, Total Rec and Dissolved
NPW	SW-846 3010A	Metals, Total
NPW	SW-846 3015	Metals
NPW	SW-846 3015A	Metals

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 3020A	Metals
NPW	SW-846 3510C	Semivolatile organics
NPW	SW-846 3511	Semivolatile organics
NPW	SW-846 3520C	Semivolatile organics
NPW	SW-846 5030B	Volatile organics
NPW	SW-846 6010B	Aluminum
NPW	SW-846 6010B	Antimony
NPW	SW-846 6010B	Arsenic
NPW	SW-846 6010B	Barium
NPW	SW-846 6010B	Beryllium
NPW	SW-846 6010B	Boron
NPW	SW-846 6010B	Cadmium
NPW	SW-846 6010B	Calcium
NPW	SW-846 6010B	Calcium-hardness
NPW	SW-846 6010B	Total hardness
NPW	SW-846 6010B	Chromium
NPW	SW-846 6010B	Cobalt
NPW	SW-846 6010B	Copper
NPW	SW-846 6010B	Iron
NPW	SW-846 6010B	Lead
NPW	SW-846 6010B	Lithium
NPW	SW-846 6010B	Magnesium
NPW	SW-846 6010B	Manganese
NPW	SW-846 6010B	Molybdenum
NPW	SW-846 6010B	Nickel
NPW	SW-846 6010B	Potassium
NPW	SW-846 6010B	Selenium
NPW	SW-846 6010B	Silica
NPW	SW-846 6010B	Silver
NPW	SW-846 6010B	Sulfur
NPW	SW-846 6010B	Sodium
NPW	SW-846 6010B	Strontium
NPW	SW-846 6010B	Thallium
NPW	SW-846 6010B	Tin
NPW	SW-846 6010B	Titanium
NPW	SW-846 6010B	Vanadium
NPW	SW-846 6010B	Zinc
NPW	SW-846 6010C	Antimony
NPW	SW-846 6010C	Arsenic
NPW	SW-846 6010C	Barium

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 6010C	Beryllium
NPW	SW-846 6010C	Boron
NPW	SW-846 6010C	Cadmium
NPW	SW-846 6010C	Calcium
NPW	SW-846 6010C	Calcium-hardness
NPW	SW-846 6010C	Total hardness
NPW	SW-846 6010C	Chromium
NPW	SW-846 6010C	Cobalt
NPW	SW-846 6010C	Copper
NPW	SW-846 6010C	Iron
NPW	SW-846 6010C	Lead
NPW	SW-846 6010C	Lithium
NPW	SW-846 6010C	Magnesium
NPW	SW-846 6010C	Manganese
NPW	SW-846 6010C	Molybdenum
NPW	SW-846 6010C	Nickel
NPW	SW-846 6010C	Potassium
NPW	SW-846 6010C	Selenium
NPW	SW-846 6010C	Silica
NPW	SW-846 6010C	Silver
NPW	SW-846 6010C	Sulfur
NPW	SW-846 6010C	Sodium
NPW	SW-846 6010C	Strontium
NPW	SW-846 6010C	Thallium
NPW	SW-846 6010C	Tin
NPW	SW-846 6010C	Titanium
NPW	SW-846 6010C	Vanadium
NPW	SW-846 6010C	Zinc
NPW	SW-846 6020	Aluminum
NPW	SW-846 6020	Antimony
NPW	SW-846 6020	Arsenic
NPW	SW-846 6020	Barium
NPW	SW-846 6020	Beryllium
NPW	SW-846 6020	Boron
NPW	SW-846 6020	Cadmium
NPW	SW-846 6020	Calcium
NPW	SW-846 6020	Chromium
NPW	SW-846 6020	Cobalt
NPW	SW-846 6020	Copper
NPW	SW-846 6020	Iron

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 6020	Lead
NPW	SW-846 6020	Magnesium
NPW	SW-846 6020	Manganese
NPW	SW-846 6020	Molybdenum
NPW	SW-846 6020	Nickel
NPW	SW-846 6020	Potassium
NPW	SW-846 6020	Selenium
NPW	SW-846 6020	Silver
NPW	SW-846 6020	Sodium
NPW	SW-846 6020	Strontium
NPW	SW-846 6020	Thallium
NPW	SW-846 6020	Thorium
NPW	SW-846 6020	Tin
NPW	SW-846 6020	Titanium
NPW	SW-846 6020	Uranium
NPW	SW-846 6020	Vanadium
NPW	SW-846 6020	Zinc
NPW	SW-846 6020A	Aluminum
NPW	SW-846 6020A	Antimony
NPW	SW-846 6020A	Arsenic
NPW	SW-846 6020A	Barium
NPW	SW-846 6020A	Beryllium
NPW	SW-846 6020A	Boron
NPW	SW-846 6020A	Cadmium
NPW	SW-846 6020A	Calcium
NPW	SW-846 6020A	Chromium
NPW	SW-846 6020A	Cobalt
NPW	SW-846 6020A	Copper
NPW	SW-846 6020A	Iron
NPW	SW-846 6020A	Lead
NPW	SW-846 6020A	Magnesium
NPW	SW-846 6020A	Manganese
NPW	SW-846 6020A	Molybdenum
NPW	SW-846 6020A	Nickel
NPW	SW-846 6020A	Potassium
NPW	SW-846 6020A	Selenium
NPW	SW-846 6020A	Silver
NPW	SW-846 6020A	Sodium
NPW	SW-846 6020A	Strontium
NPW	SW-846 6020A	Thallium

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 6020A	Thorium
NPW	SW-846 6020A	Tin
NPW	SW-846 6020A	Titanium
NPW	SW-846 6020A	Uranium
NPW	SW-846 6020A	Vanadium
NPW	SW-846 6020A	Zinc
NPW	SW-846 7196A	Chromium (VI)
NPW	SW-846 7199	Chromium (VI)
NPW	SW-846 7470A	Mercury - liquid waste
NPW	SW-846 8011	Dibromoethane (1,2-) (EDB)
NPW	SW-846 8011	Dibromo-3-chloropropane (1,2-)
NPW	SW-846 8015B	Ethylene glycol
NPW	SW-846 8015B	Propylene glycol
NPW	SW-846 8015B	Gasoline range organic
NPW	SW-846 8015B	Diesel range organic
NPW	SW-846 8015C	Ethylene glycol
NPW	SW-846 8015C	Propylene glycol
NPW	SW-846 8015D	Ethylene glycol
NPW	SW-846 8015D	Propylene glycol
NPW	SW-846 8015D	Gasoline range organic
NPW	SW-846 8015D	Diesel range organic
NPW	SW-846 8021B	Xylenes (total)
NPW	SW-846 8021B	Methyl tert-butyl ether
NPW	SW-846 8021B	Benzene
NPW	SW-846 8021B	Ethylbenzene
NPW	SW-846 8021B	Toluene
NPW	SW-846 8021B	Xylene (o-)
NPW	SW-846 8021B	Xylene (m-)
NPW	SW-846 8021B	Xylene (p-)
NPW	SW-846 8081A	Alachlor
NPW	SW-846 8081A	Chlordane (alpha)
NPW	SW-846 8081A	Chlordane (gamma)
NPW	SW-846 8081A	Chloroneb
NPW	SW-846 8081A	Chlorothalonil
NPW	SW-846 8081A	Etridiazole
NPW	SW-846 8081A	Hexachlorobenzene
NPW	SW-846 8081A	Hexachlorocyclopentadiene
NPW	SW-846 8081A	Permethrin
NPW	SW-846 8081A	Propachlor
NPW	SW-846 8081A	Trifluralin



<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8081A	Aldrin
NPW	SW-846 8081A	Alpha BHC
NPW	SW-846 8081A	Beta BHC
NPW	SW-846 8081A	Delta BHC
NPW	SW-846 8081A	Lindane (gamma BHC)
NPW	SW-846 8081A	Chlordane (technical)
NPW	SW-846 8081A	DDD (4,4'-)
NPW	SW-846 8081A	DDE (4,4'-)
NPW	SW-846 8081A	DDT (4,4'-)
NPW	SW-846 8081A	Dieldrin
NPW	SW-846 8081A	Endosulfan I
NPW	SW-846 8081A	Endosulfan II
NPW	SW-846 8081A	Endosulfan sulfate
NPW	SW-846 8081A	Endrin
NPW	SW-846 8081A	Endrin aldehyde
NPW	SW-846 8081A	Endrin ketone
NPW	SW-846 8081A	Heptachlor
NPW	SW-846 8081A	Heptachlor epoxide
NPW	SW-846 8081A	Methoxychlor
NPW	SW-846 8081A	Toxaphene
NPW	SW-846 8081B	Alachlor
NPW	SW-846 8081B	Chlordane (alpha)
NPW	SW-846 8081B	Chlordane (gamma)
NPW	SW-846 8081B	Chloroneb
NPW	SW-846 8081B	Chlorothalonil
NPW	SW-846 8081B	Etridiazole
NPW	SW-846 8081B	Hexachlorobenzene
NPW	SW-846 8081B	Hexachlorocyclopentadiene
NPW	SW-846 8081B	Permethrin
NPW	SW-846 8081B	Propachlor
NPW	SW-846 8081B	Trifluralin
NPW	SW-846 8081B	Aldrin
NPW	SW-846 8081B	Alpha BHC
NPW	SW-846 8081B	Beta BHC
NPW	SW-846 8081B	Delta BHC
NPW	SW-846 8081B	Lindane (gamma BHC)
NPW	SW-846 8081B	Chlordane (technical)
NPW	SW-846 8081B	DDD (4,4'-)
NPW	SW-846 8081B	DDE (4,4'-)
NPW	SW-846 8081B	DDT (4,4'-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8081B	Dieldrin
NPW	SW-846 8081B	Endosulfan I
NPW	SW-846 8081B	Endosulfan II
NPW	SW-846 8081B	Endosulfan sulfate
NPW	SW-846 8081B	Endrin
NPW	SW-846 8081B	Endrin aldehyde
NPW	SW-846 8081B	Endrin ketone
NPW	SW-846 8081B	Heptachlor
NPW	SW-846 8081B	Heptachlor epoxide
NPW	SW-846 8081B	Methoxychlor
NPW	SW-846 8081B	Toxaphene
NPW	SW-846 8082	PCB 1016
NPW	SW-846 8082	PCB 1221
NPW	SW-846 8082	PCB 1232
NPW	SW-846 8082	PCB 1242
NPW	SW-846 8082	PCB 1248
NPW	SW-846 8082	PCB 1254
NPW	SW-846 8082	PCB 1260
NPW	SW-846 8082A	PCB 1016
NPW	SW-846 8082A	PCB 1221
NPW	SW-846 8082A	PCB 1232
NPW	SW-846 8082A	PCB 1242
NPW	SW-846 8082A	PCB 1248
NPW	SW-846 8082A	PCB 1254
NPW	SW-846 8082A	PCB 1260
NPW	SW-846 8141A	Azinphos methyl
NPW	SW-846 8141A	Chlorpyrifos
NPW	SW-846 8141A	Demeton (o-)
NPW	SW-846 8141A	Demeton (s-)
NPW	SW-846 8141A	Disulfoton
NPW	SW-846 8141A	Bolstar
NPW	SW-846 8141A	Coumaphos
NPW	SW-846 8141A	Dichlorvos
NPW	SW-846 8141A	Dimethoate
NPW	SW-846 8141A	EPN
NPW	SW-846 8141A	Ethoprop
NPW	SW-846 8141A	Fensulfothion
NPW	SW-846 8141A	Fenthion
NPW	SW-846 8141A	Merphos
NPW	SW-846 8141A	Mevinphos

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8141A	Naled
NPW	SW-846 8141A	Parathion
NPW	SW-846 8141A	Parathion methyl
NPW	SW-846 8141A	Phorate
NPW	SW-846 8141A	Ronnel
NPW	SW-846 8141A	Stirofos
NPW	SW-846 8141A	Sulfotepp
NPW	SW-846 8141A	TEPP
NPW	SW-846 8141A	Tokuthion [Protothiofos]
NPW	SW-846 8141A	Trichloronate
NPW	SW-846 8141A	Diazinon
NPW	SW-846 8141A	Malathion
NPW	SW-846 8141B	Azinphos methyl
NPW	SW-846 8141B	Chlorpyrifos
NPW	SW-846 8141B	Demeton (o-)
NPW	SW-846 8141B	Demeton (s-)
NPW	SW-846 8141B	Disulfoton
NPW	SW-846 8141B	Bolstar
NPW	SW-846 8141B	Coumaphos
NPW	SW-846 8141B	Dichlorvos
NPW	SW-846 8141B	Dimethoate
NPW	SW-846 8141B	EPN
NPW	SW-846 8141B	Ethoprop
NPW	SW-846 8141B	Fensulfothion
NPW	SW-846 8141B	Fenthion
NPW	SW-846 8141B	Merphos
NPW	SW-846 8141B	Mevinphos
NPW	SW-846 8141B	Naled
NPW	SW-846 8141B	Parathion
NPW	SW-846 8141B	Parathion methyl
NPW	SW-846 8141B	Phorate
NPW	SW-846 8141B	Ronnel
NPW	SW-846 8141B	Stirofos
NPW	SW-846 8141B	Sulfotepp
NPW	SW-846 8141B	TEPP
NPW	SW-846 8141B	Tokuthion [Protothiofos]
NPW	SW-846 8141B	Trichloronate
NPW	SW-846 8141B	Diazinon
NPW	SW-846 8141B	Malathion
NPW	SW-846 8151A	Dicamba

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8151A	DB (2,4-)
NPW	SW-846 8151A	Dinoseb
NPW	SW-846 8151A	Dalapon
NPW	SW-846 8151A	Dichlorprop
NPW	SW-846 8151A	D (2,4-)
NPW	SW-846 8151A	T (2,4,5-)
NPW	SW-846 8151A	TP (2,4,5-) (Silvex)
NPW	SW-846 8151A	MCPA
NPW	SW-846 8151A	MCPP
NPW	SW-846 8260B	Methyl alcohol (Methanol)
NPW	SW-846 8260B	Ethyl alcohol
NPW	SW-846 8260B	Hexane (n-)
NPW	SW-846 8260B	Trimethylpentane (2,2,4-)
NPW	SW-846 8260B	Methylnaphthalene (1-)
NPW	SW-846 8260B	Methylnaphthalene (2-)
NPW	SW-846 8260B	Butanol (3,3-Dimethyl-1-)
NPW	SW-846 8260B	Trimethylpentane (2,2,4-)
NPW	SW-846 8260B	Trimethylbenzene (1,2,3-)
NPW	SW-846 8260B	Cyclohexane
NPW	SW-846 8260B	Butanol (1-)
NPW	SW-846 8260B	Nitropropane (2-)
NPW	SW-846 8260B	Butyl formate (t-)
NPW	SW-846 8260B	Methyl acetate
NPW	SW-846 8260B	Pentanol (2-Methyl-2-)
NPW	SW-846 8260B	Amyl alcohol (t-)
NPW	SW-846 8260B	Methylcyclohexane
NPW	SW-846 8260B	Octane (-n)
NPW	SW-846 8260B	tert-Amylmethyl ether [TAME]
NPW	SW-846 8260B	Bromoethane
NPW	SW-846 8260B	Cyclohexanone
NPW	SW-846 8260B	Diisopropyl Ether [DIPE]
NPW	SW-846 8260B	Tetrahydrofuran
NPW	SW-846 8260B	Ethyl-tert-butyl Ether [ETBE]
NPW	SW-846 8260B	Safrole
NPW	SW-846 8260B	Xylene (m-)
NPW	SW-846 8260B	Xylene (o-)
NPW	SW-846 8260B	Xylene (p-)
NPW	SW-846 8260B	Dichloro-2-butene (cis-1,4-)
NPW	SW-846 8260B	Diethyl ether (Ethyl ether)
NPW	SW-846 8260B	Dichloro-2-butene (trans-1,4-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8260B	Ethanol
NPW	SW-846 8260B	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
NPW	SW-846 8260B	Vinyl acetate
NPW	SW-846 8260B	Pentachloroethane
NPW	SW-846 8260B	Tert-butyl alcohol
NPW	SW-846 8260B	Dioxane (1,4-)
NPW	SW-846 8260B	Bromobenzene
NPW	SW-846 8260B	Butyl benzene (n-)
NPW	SW-846 8260B	Sec-butylbenzene
NPW	SW-846 8260B	Tert-butylbenzene
NPW	SW-846 8260B	Chlorotoluene (2-)
NPW	SW-846 8260B	Chlorotoluene (4-)
NPW	SW-846 8260B	Isopropylbenzene
NPW	SW-846 8260B	Propylbenzene (n-)
NPW	SW-846 8260B	Isopropyltoluene (4-)
NPW	SW-846 8260B	Trichlorobenzene (1,2,3-)
NPW	SW-846 8260B	Trimethylbenzene (1,2,4-)
NPW	SW-846 8260B	Trimethylbenzene (1,3,5-)
NPW	SW-846 8260B	Allyl chloride
NPW	SW-846 8260B	Bromochloromethane
NPW	SW-846 8260B	Butadiene (2-chloro-1,3-)
NPW	SW-846 8260B	Dibromoethane (1,2-) (EDB)
NPW	SW-846 8260B	Dibromomethane
NPW	SW-846 8260B	Dibromo-3-chloropropane (1,2-)
NPW	SW-846 8260B	Dichloropropane (1,3-)
NPW	SW-846 8260B	Dichloropropane (2,2-)
NPW	SW-846 8260B	Dichloropropene (1,1-)
NPW	SW-846 8260B	Trichloropropane (1,2,3-)
NPW	SW-846 8260B	Ethyl acetate
NPW	SW-846 8260B	Ethyl methacrylate
NPW	SW-846 8260B	Methacrylonitrile
NPW	SW-846 8260B	Methyl acrylate
NPW	SW-846 8260B	Methyl methacrylate
NPW	SW-846 8260B	Methyl iodide
NPW	SW-846 8260B	Iso-butyl alcohol
NPW	SW-846 8260B	Isopropanol
NPW	SW-846 8260B	N-Nitroso-di-n-butylamine
NPW	SW-846 8260B	Propionitrile
NPW	SW-846 8260B	Acetonitrile
NPW	SW-846 8260B	Benzene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8260B	Chlorobenzene
NPW	SW-846 8260B	Dichlorobenzene (1,2-)
NPW	SW-846 8260B	Dichlorobenzene (1,3-)
NPW	SW-846 8260B	Dichlorobenzene (1,4-)
NPW	SW-846 8260B	Ethylbenzene
NPW	SW-846 8260B	Toluene
NPW	SW-846 8260B	Xylenes (total)
NPW	SW-846 8260B	Bromodichloromethane
NPW	SW-846 8260B	Bromoform
NPW	SW-846 8260B	Bromomethane
NPW	SW-846 8260B	Carbon tetrachloride
NPW	SW-846 8260B	Chloroethane
NPW	SW-846 8260B	Chloroethyl vinyl ether (2-)
NPW	SW-846 8260B	Chloroform
NPW	SW-846 8260B	Chloromethane
NPW	SW-846 8260B	Dichloropropene (trans-1,3-)
NPW	SW-846 8260B	Dibromochloromethane
NPW	SW-846 8260B	Dichlorodifluoromethane
NPW	SW-846 8260B	Dichloroethane (1,1-)
NPW	SW-846 8260B	Dichloroethane (1,2-)
NPW	SW-846 8260B	Dichloroethene (1,1-)
NPW	SW-846 8260B	Dichloroethene (trans-1,2-)
NPW	SW-846 8260B	Dichloroethene (cis-1,2-)
NPW	SW-846 8260B	Dichloropropane (1,2-)
NPW	SW-846 8260B	Dichloropropene (cis-1,3-)
NPW	SW-846 8260B	Methylene chloride (Dichloromethane)
NPW	SW-846 8260B	Tetrachloroethane (1,1,2,2-)
NPW	SW-846 8260B	Tetrachloroethene
NPW	SW-846 8260B	Trichloroethane (1,1,1-)
NPW	SW-846 8260B	Trichloroethane (1,1,2-)
NPW	SW-846 8260B	Trichloroethene
NPW	SW-846 8260B	Trichlorofluoromethane
NPW	SW-846 8260B	Vinyl chloride
NPW	SW-846 8260B	Acetone
NPW	SW-846 8260B	Carbon disulfide
NPW	SW-846 8260B	Butanone (2-)
NPW	SW-846 8260B	Hexanone (2-)
NPW	SW-846 8260B	Pentanone (4-methyl-2-) (MIBK)
NPW	SW-846 8260B	Methyl tert-butyl ether
NPW	SW-846 8260B	Acrolein

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8260B	Acrylonitrile
NPW	SW-846 8260B	Hexachlorobutadiene (1,3-)
NPW	SW-846 8260B	Hexachloroethane
NPW	SW-846 8260B	Naphthalene
NPW	SW-846 8260B	Styrene
NPW	SW-846 8260B	Tetrachloroethane (1,1,1,2-)
NPW	SW-846 8260B	Trichlorobenzene (1,2,4-)
NPW	SW-846 8260C	Methyl alcohol (Methanol)
NPW	SW-846 8260C	Ethyl alcohol
NPW	SW-846 8260C	Trimethylpentane (2,2,4-)
NPW	SW-846 8260C	Methylnaphthalene (1-)
NPW	SW-846 8260C	Methylnaphthalene (2-)
NPW	SW-846 8260C	Butanol (3,3-Dimethyl-1-)
NPW	SW-846 8260C	Trimethylbenzene (1,2,3-)
NPW	SW-846 8260C	Cyclohexane
NPW	SW-846 8260C	Butanol (1-)
NPW	SW-846 8260C	Nitropropane (2-)
NPW	SW-846 8260C	Butyl formate (t-)
NPW	SW-846 8260C	Methyl acetate
NPW	SW-846 8260C	Pentanol (2-Methyl-2-)
NPW	SW-846 8260C	Amyl alcohol (t-)
NPW	SW-846 8260C	Methylcyclohexane
NPW	SW-846 8260C	Octane (-n)
NPW	SW-846 8260C	tert-Amylmethyl ether [TAME]
NPW	SW-846 8260C	Bromoethane
NPW	SW-846 8260C	Cyclohexanone
NPW	SW-846 8260C	Diisopropyl Ether [DIPE]
NPW	SW-846 8260C	Tetrahydrofuran
NPW	SW-846 8260C	Ethyl-tert-butyl Ether [ETBE]
NPW	SW-846 8260C	Xylene (m-)
NPW	SW-846 8260C	Xylene (o-)
NPW	SW-846 8260C	Xylene (p-)
NPW	SW-846 8260C	Dichloro-2-butene (cis-1,4-)
NPW	SW-846 8260C	Diethyl ether (Ethyl ether)
NPW	SW-846 8260C	Dichloro-2-butene (trans-1,4-)
NPW	SW-846 8260C	Ethanol
NPW	SW-846 8260C	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
NPW	SW-846 8260C	Vinyl acetate
NPW	SW-846 8260C	Pentachloroethane
NPW	SW-846 8260C	Tert-butyl alcohol

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8260C	Dioxane (1,4-)
NPW	SW-846 8260C	Bromobenzene
NPW	SW-846 8260C	Butyl benzene (n-)
NPW	SW-846 8260C	Sec-butylbenzene
NPW	SW-846 8260C	Tert-butylbenzene
NPW	SW-846 8260C	Chlorotoluene (2-)
NPW	SW-846 8260C	Chlorotoluene (4-)
NPW	SW-846 8260C	Isopropylbenzene
NPW	SW-846 8260C	Propylbenzene (n-)
NPW	SW-846 8260C	Isopropyltoluene (4-)
NPW	SW-846 8260C	Trichlorobenzene (1,2,3-)
NPW	SW-846 8260C	Trimethylbenzene (1,2,4-)
NPW	SW-846 8260C	Trimethylbenzene (1,3,5-)
NPW	SW-846 8260C	Allyl chloride
NPW	SW-846 8260C	Bromochloromethane
NPW	SW-846 8260C	Butadiene (2-chloro-1,3-)
NPW	SW-846 8260C	Dibromoethane (1,2-) (EDB)
NPW	SW-846 8260C	Dibromomethane
NPW	SW-846 8260C	Dibromo-3-chloropropane (1,2-)
NPW	SW-846 8260C	Dichloropropane (1,3-)
NPW	SW-846 8260C	Dichloropropane (2,2-)
NPW	SW-846 8260C	Dichloropropene (1,1-)
NPW	SW-846 8260C	Trichloropropane (1,2,3-)
NPW	SW-846 8260C	Ethyl acetate
NPW	SW-846 8260C	Ethyl methacrylate
NPW	SW-846 8260C	Methacrylonitrile
NPW	SW-846 8260C	Methyl acrylate
NPW	SW-846 8260C	Methyl methacrylate
NPW	SW-846 8260C	Methyl iodide
NPW	SW-846 8260C	Iso-butyl alcohol
NPW	SW-846 8260C	Isopropanol
NPW	SW-846 8260C	N-Nitroso-di-n-butylamine
NPW	SW-846 8260C	Propionitrile
NPW	SW-846 8260C	Acetonitrile
NPW	SW-846 8260C	Benzene
NPW	SW-846 8260C	Chlorobenzene
NPW	SW-846 8260C	Dichlorobenzene (1,2-)
NPW	SW-846 8260C	Dichlorobenzene (1,3-)
NPW	SW-846 8260C	Dichlorobenzene (1,4-)
NPW	SW-846 8260C	Ethylbenzene



<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8260C	Toluene
NPW	SW-846 8260C	Xylenes (total)
NPW	SW-846 8260C	Bromodichloromethane
NPW	SW-846 8260C	Bromoform
NPW	SW-846 8260C	Bromomethane
NPW	SW-846 8260C	Carbon tetrachloride
NPW	SW-846 8260C	Chloroethane
NPW	SW-846 8260C	Chloroethyl vinyl ether (2-)
NPW	SW-846 8260C	Chloroform
NPW	SW-846 8260C	Chloromethane
NPW	SW-846 8260C	Dichloropropene (trans-1,3-)
NPW	SW-846 8260C	Dibromochloromethane
NPW	SW-846 8260C	Dichlorodifluoromethane
NPW	SW-846 8260C	Dichloroethane (1,1-)
NPW	SW-846 8260C	Dichloroethane (1,2-)
NPW	SW-846 8260C	Dichloroethene (1,1-)
NPW	SW-846 8260C	Dichloroethene (trans-1,2-)
NPW	SW-846 8260C	Dichloroethene (cis-1,2-)
NPW	SW-846 8260C	Dichloropropane (1,2-)
NPW	SW-846 8260C	Dichloropropene (cis-1,3-)
NPW	SW-846 8260C	Methylene chloride (Dichloromethane)
NPW	SW-846 8260C	Tetrachloroethane (1,1,2,2-)
NPW	SW-846 8260C	Tetrachloroethene
NPW	SW-846 8260C	Trichloroethane (1,1,1-)
NPW	SW-846 8260C	Trichloroethane (1,1,2-)
NPW	SW-846 8260C	Trichloroethene
NPW	SW-846 8260C	Trichlorofluoromethane
NPW	SW-846 8260C	Vinyl chloride
NPW	SW-846 8260C	Acetone
NPW	SW-846 8260C	Carbon disulfide
NPW	SW-846 8260C	Butanone (2-)
NPW	SW-846 8260C	Hexanone (2-)
NPW	SW-846 8260C	Pentanone (4-methyl-2-) (MIBK)
NPW	SW-846 8260C	Methyl tert-butyl ether
NPW	SW-846 8260C	Acrolein
NPW	SW-846 8260C	Acrylonitrile
NPW	SW-846 8260C	Hexachlorobutadiene (1,3-)
NPW	SW-846 8260C	Hexachloroethane
NPW	SW-846 8260C	Naphthalene
NPW	SW-846 8260C	Styrene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8260C	Tetrachloroethane (1,1,1,2-)
NPW	SW-846 8260C	Trichlorobenzene (1,2,4-)
NPW	SW-846 8270C	Biphenyl (1,1'-)
NPW	SW-846 8270C	Benzaldehyde
NPW	SW-846 8270C	Caprolactam
NPW	SW-846 8270C	Atrazine
NPW	SW-846 8270C	Phenanthrene
NPW	SW-846 8270C	Pyrene
NPW	SW-846 8270C	Acenaphthene
NPW	SW-846 8270C	Acenaphthylene
NPW	SW-846 8270C	Anthracene
NPW	SW-846 8270C	Benzo(ghi)perylene
NPW	SW-846 8270C	Chrysene
NPW	SW-846 8270C	Methylnaphthalene (1-)
NPW	SW-846 8270C	Methylnaphthalene (2-)
NPW	SW-846 8270C	Naphthalene
NPW	SW-846 8270C	Fluoranthene
NPW	SW-846 8270C	Fluorene
NPW	SW-846 8270C	Methylnaphthalene (1-)
NPW	SW-846 8270C	Nitrodiphenylamine (2-)
NPW	SW-846 8270C	Nitrodiphenylamine (2-)
NPW	SW-846 8270C	Hexachlorophene
NPW	SW-846 8270C	Diphenylhydrazine (1,2-)
NPW	SW-846 8270C	Decane (n-)
NPW	SW-846 8270C	Octadecane (n-)
NPW	SW-846 8270C	Benzo(a)anthracene
NPW	SW-846 8270C	Benzo(a)pyrene
NPW	SW-846 8270C	Benzo(b)fluoranthene
NPW	SW-846 8270C	Benzo(k)fluoranthene
NPW	SW-846 8270C	Dibenzo(a,h)anthracene
NPW	SW-846 8270C	Indeno(1,2,3-cd)pyrene
NPW	SW-846 8270C	Benzal chloride
NPW	SW-846 8270C	Benzo(j)fluoranthene
NPW	SW-846 8270C	Benzotrichloride
NPW	SW-846 8270C	Benzyl chloride
NPW	SW-846 8270C	Chlorobenzilate
NPW	SW-846 8270C	Dibenz(a,h)acridine
NPW	SW-846 8270C	Dibenzo(a,h)pyrene
NPW	SW-846 8270C	Dibenzo(a,i)pyrene
NPW	SW-846 8270C	Dibenzo(c,g)carbazole (7H-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8270C	Pentachloroethane
NPW	SW-846 8270C	Tetrachlorobenzene (1,2,3,4-)
NPW	SW-846 8270C	Tetrachlorobenzene (1,2,3,5-)
NPW	SW-846 8270C	Benzyl alcohol
NPW	SW-846 8270C	Acetophenone
NPW	SW-846 8270C	Acetylaminofluorene (2-)
NPW	SW-846 8270C	Aminobiphenyl (4-)
NPW	SW-846 8270C	Aramite
NPW	SW-846 8270C	Chloronaphthalene (1-)
NPW	SW-846 8270C	Diallate (cis)
NPW	SW-846 8270C	Diallate (trans)
NPW	SW-846 8270C	Dibenzo(a,e)pyrene
NPW	SW-846 8270C	Dibenz(a,j)acridine
NPW	SW-846 8270C	Dichlorophenol (2,6-)
NPW	SW-846 8270C	Dimethoate
NPW	SW-846 8270C	Dimethylaminoazobenzene
NPW	SW-846 8270C	Dimethylbenz(a)anthracene (7,12-)
NPW	SW-846 8270C	Dimethyl benzidine (3,3-)
NPW	SW-846 8270C	Dinitrobenzene (1,3-)
NPW	SW-846 8270C	Dinoseb
NPW	SW-846 8270C	Disulfoton
NPW	SW-846 8270C	Famphur
NPW	SW-846 8270C	Hexachloropropene
NPW	SW-846 8270C	Isodrin
NPW	SW-846 8270C	Isosafrole (cis-)
NPW	SW-846 8270C	Isosafrole (trans-)
NPW	SW-846 8270C	Kepone
NPW	SW-846 8270C	Methanesulfonate (Ethyl-)
NPW	SW-846 8270C	Methanesulfonate (Methyl-)
NPW	SW-846 8270C	Methapyrilene
NPW	SW-846 8270C	Methylcholanthrene (3-)
NPW	SW-846 8270C	Napthoquinone (1,4-)
NPW	SW-846 8270C	Napththylamine (1-)
NPW	SW-846 8270C	Napththylamine (2-)
NPW	SW-846 8270C	N-Nitroso-di-n-butylamine
NPW	SW-846 8270C	N-Nitrosomorpholine
NPW	SW-846 8270C	N-Nitrosopiperidine
NPW	SW-846 8270C	Parathion
NPW	SW-846 8270C	Parathion methyl
NPW	SW-846 8270C	Pentachlorobenzene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8270C	Pentachloronitrobenzene
NPW	SW-846 8270C	Phenacetin
NPW	SW-846 8270C	Phenylenediamine (1,4-)
NPW	SW-846 8270C	Phenylethylamine (alpha, alpha-Dimethyl)
NPW	SW-846 8270C	Phorate
NPW	SW-846 8270C	Phosphorothioate (O,O,O-triethyl)
NPW	SW-846 8270C	Phosphorothioate (O,O-diethyl-O-2-pyrazinyl) [Thionazin]
NPW	SW-846 8270C	Picoline (2-)
NPW	SW-846 8270C	Pronamide
NPW	SW-846 8270C	Quinoline -1-Oxide (4-Nitro)
NPW	SW-846 8270C	Safrole
NPW	SW-846 8270C	Sulfotepp
NPW	SW-846 8270C	Tetrachlorobenzene (1,2,4,5-)
NPW	SW-846 8270C	Tetrachlorophenol (2,3,4,6-)
NPW	SW-846 8270C	Toluidine (2-) (2-Methylaniline)
NPW	SW-846 8270C	Toluidine (5-nitro-2-)
NPW	SW-846 8270C	Trinitrobenzene (1,3,5-)
NPW	SW-846 8270C	N-Nitrosodiethylamine
NPW	SW-846 8270C	N-Nitrosopyrrolidine
NPW	SW-846 8270C	Diphenylamine
NPW	SW-846 8270C	Carbazole
NPW	SW-846 8270C	Dichlorobenzene (1,2-)
NPW	SW-846 8270C	Dichlorobenzene (1,3-)
NPW	SW-846 8270C	N-Nitrosodimethylamine
NPW	SW-846 8270C	N-Nitroso-di-n-propylamine
NPW	SW-846 8270C	N-Nitrosomethylethylamine
NPW	SW-846 8270C	Benzidine
NPW	SW-846 8270C	Aniline
NPW	SW-846 8270C	Hexachloropropene
NPW	SW-846 8270C	Dibenzofuran
NPW	SW-846 8270C	Benzoic acid
NPW	SW-846 8270C	N-Nitrosodiphenylamine
NPW	SW-846 8270C	Dichlorobenzidine (3,3'-)
NPW	SW-846 8270C	Chloroaniline (4-)
NPW	SW-846 8270C	Nitroaniline (2-)
NPW	SW-846 8270C	Nitroaniline (3-)
NPW	SW-846 8270C	Nitroaniline (4-)
NPW	SW-846 8270C	Chloronaphthalene (2-)
NPW	SW-846 8270C	Hexachlorobenzene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8270C	Hexachlorobutadiene (1,3-)
NPW	SW-846 8270C	Hexachlorocyclopentadiene
NPW	SW-846 8270C	Hexachloroethane
NPW	SW-846 8270C	Trichlorobenzene (1,2,4-)
NPW	SW-846 8270C	Bis (2-chloroethoxy) methane
NPW	SW-846 8270C	Bis (2-chloroethyl) ether
NPW	SW-846 8270C	Bis (2-chloroisopropyl) ether
NPW	SW-846 8270C	Chlorophenyl-phenyl ether (4-)
NPW	SW-846 8270C	Bromophenyl-phenyl ether (4-)
NPW	SW-846 8270C	Dinitrotoluene (2,4-)
NPW	SW-846 8270C	Dinitrotoluene (2,6-)
NPW	SW-846 8270C	Isophorone
NPW	SW-846 8270C	Nitrobenzene
NPW	SW-846 8270C	Butyl benzyl phthalate
NPW	SW-846 8270C	Bis (2-ethylhexyl) phthalate
NPW	SW-846 8270C	Diethyl phthalate
NPW	SW-846 8270C	Dimethyl phthalate
NPW	SW-846 8270C	Di-n-butyl phthalate
NPW	SW-846 8270C	Di-n-octyl phthalate
NPW	SW-846 8270C	Acenaphthene
NPW	SW-846 8270C	Anthracene
NPW	SW-846 8270C	Acenaphthylene
NPW	SW-846 8270C	Benzo(a)anthracene
NPW	SW-846 8270C	Benzo(a)pyrene
NPW	SW-846 8270C	Benzo(b)fluoranthene
NPW	SW-846 8270C	Benzo(ghi)perylene
NPW	SW-846 8270C	Benzo(k)fluoranthene
NPW	SW-846 8270C	Chrysene
NPW	SW-846 8270C	Dibenzo(a,h)anthracene
NPW	SW-846 8270C	Fluoranthene
NPW	SW-846 8270C	Fluorene
NPW	SW-846 8270C	Indeno(1,2,3-cd)pyrene
NPW	SW-846 8270C	Methylnaphthalene (2-)
NPW	SW-846 8270C	Naphthalene
NPW	SW-846 8270C	Phenanthrene
NPW	SW-846 8270C	Pyrene
NPW	SW-846 8270C	Methyl phenol (4-chloro-3-)
NPW	SW-846 8270C	Chlorophenol (2-)
NPW	SW-846 8270C	Dichlorophenol (2,4-)
NPW	SW-846 8270C	Dimethylphenol (2,4-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8270C	Dinitrophenol (2,4-)
NPW	SW-846 8270C	Dinitrophenol (2-methyl-4,6-)
NPW	SW-846 8270C	Methylphenol (2-)
NPW	SW-846 8270C	Methylphenol (4-)
NPW	SW-846 8270C	Nitrophenol (2-)
NPW	SW-846 8270C	Nitrophenol (4-)
NPW	SW-846 8270C	Pentachlorophenol
NPW	SW-846 8270C	Phenol
NPW	SW-846 8270C	Trichlorophenol (2,4,5-)
NPW	SW-846 8270C	Trichlorophenol (2,4,6-)
NPW	SW-846 8270C	Dichlorobenzene (1,4-)
NPW	SW-846 8270C	Pyridine
NPW	SW-846 8270D	Biphenyl (1,1'-)
NPW	SW-846 8270D	Benzaldehyde
NPW	SW-846 8270D	Caprolactam
NPW	SW-846 8270D	Atrazine
NPW	SW-846 8270D	Phenanthrene
NPW	SW-846 8270D	Pyrene
NPW	SW-846 8270D	Acenaphthene
NPW	SW-846 8270D	Acenaphthylene
NPW	SW-846 8270D	Anthracene
NPW	SW-846 8270D	Benzo(ghi)perylene
NPW	SW-846 8270D	Chrysene
NPW	SW-846 8270D	Methylnaphthalene (1-)
NPW	SW-846 8270D	Methylnaphthalene (2-)
NPW	SW-846 8270D	Naphthalene
NPW	SW-846 8270D	Fluoranthene
NPW	SW-846 8270D	Fluorene
NPW	SW-846 8270D	Methylnaphthalene (1-)
NPW	SW-846 8270D	Nitrodiphenylamine (2-)
NPW	SW-846 8270D	Hexachlorophene
NPW	SW-846 8270D	Diphenylhydrazine (1,2-)
NPW	SW-846 8270D	Decane (n-)
NPW	SW-846 8270D	Octadecane (n-)
NPW	SW-846 8270D	Benzo(a)anthracene
NPW	SW-846 8270D	Benzo(a)pyrene
NPW	SW-846 8270D	Benzo(b)fluoranthene
NPW	SW-846 8270D	Benzo(k)fluoranthene
NPW	SW-846 8270D	Dibenzo(a,h)anthracene
NPW	SW-846 8270D	Indeno(1,2,3-cd)pyrene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8270D	Benzal chloride
NPW	SW-846 8270D	Benzo(j)fluoranthene
NPW	SW-846 8270D	Benzotrichloride
NPW	SW-846 8270D	Benzyl chloride
NPW	SW-846 8270D	Chlorobenzilate
NPW	SW-846 8270D	Dibenz(a,h)acridine
NPW	SW-846 8270D	Dibenzo(a,h)pyrene
NPW	SW-846 8270D	Dibenzo(a,i)pyrene
NPW	SW-846 8270D	Dibenzo(c,g)carbazole (7H-)
NPW	SW-846 8270D	Pentachloroethane
NPW	SW-846 8270D	Tetrachlorobenzene (1,2,3,4-)
NPW	SW-846 8270D	Tetrachlorobenzene (1,2,3,5-)
NPW	SW-846 8270D	Benzyl alcohol
NPW	SW-846 8270D	Acetophenone
NPW	SW-846 8270D	Acetylaminofluorene (2-)
NPW	SW-846 8270D	Aminobiphenyl (4-)
NPW	SW-846 8270D	Aramite
NPW	SW-846 8270D	Chloronaphthalene (1-)
NPW	SW-846 8270D	Diallate (cis)
NPW	SW-846 8270D	Diallate (trans)
NPW	SW-846 8270D	Dibenzo(a,e)pyrene
NPW	SW-846 8270D	Dibenz(a,j)acridine
NPW	SW-846 8270D	Dichlorophenol (2,6-)
NPW	SW-846 8270D	Dimethoate
NPW	SW-846 8270D	Dimethylaminoazobenzene
NPW	SW-846 8270D	Dimethylbenz(a)anthracene (7,12-)
NPW	SW-846 8270D	Dimethyl benzidine (3,3-)
NPW	SW-846 8270D	Dinitrobenzene (1,3-)
NPW	SW-846 8270D	Dinoseb
NPW	SW-846 8270D	Disulfoton
NPW	SW-846 8270D	Famphur
NPW	SW-846 8270D	Isodrin
NPW	SW-846 8270D	Isosafrole (cis-)
NPW	SW-846 8270D	Isosafrole (trans-)
NPW	SW-846 8270D	Kepone
NPW	SW-846 8270D	Methanesulfonate (Ethyl-)
NPW	SW-846 8270D	Methanesulfonate (Methyl-)
NPW	SW-846 8270D	Methapyrilene
NPW	SW-846 8270D	Methylcholanthrene (3-)
NPW	SW-846 8270D	Napthoquinone (1,4-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8270D	Napththylamine (1-)
NPW	SW-846 8270D	Napththylamine (2-)
NPW	SW-846 8270D	N-Nitroso-di-n-butylamine
NPW	SW-846 8270D	N-Nitrosomorpholine
NPW	SW-846 8270D	N-Nitrosopiperidine
NPW	SW-846 8270D	Parathion
NPW	SW-846 8270D	Parathion methyl
NPW	SW-846 8270D	Pentachlorobenzene
NPW	SW-846 8270D	Pentachloronitrobenzene
NPW	SW-846 8270D	Phenacetin
NPW	SW-846 8270D	Phenylenediamine (1,4-)
NPW	SW-846 8270D	Phenylethylamine (alpha, alpha-Dimethyl)
NPW	SW-846 8270D	Phorate
NPW	SW-846 8270D	Phosphorothioate (O,O,O-triethyl)
NPW	SW-846 8270D	Phosphorothioate (O,O-diethyl-O-2-pyrazinyl) [Thionazin]
NPW	SW-846 8270D	Picoline (2-)
NPW	SW-846 8270D	Pronamide
NPW	SW-846 8270D	Quinoline -1-Oxide (4-Nitro)
NPW	SW-846 8270D	Safrole
NPW	SW-846 8270D	Sulfotepp
NPW	SW-846 8270D	Tetrachlorobenzene (1,2,4,5-)
NPW	SW-846 8270D	Tetrachlorophenol (2,3,4,6-)
NPW	SW-846 8270D	Toluidine (2-) (2-Methylaniline)
NPW	SW-846 8270D	Toluidine (5-nitro-2-)
NPW	SW-846 8270D	Trinitrobenzene (1,3,5-)
NPW	SW-846 8270D	N-Nitrosodiethylamine
NPW	SW-846 8270D	N-Nitrosopyrrolidine
NPW	SW-846 8270D	Diphenylamine
NPW	SW-846 8270D	Carbazole
NPW	SW-846 8270D	Dichlorobenzene (1,2-)
NPW	SW-846 8270D	Dichlorobenzene (1,3-)
NPW	SW-846 8270D	N-Nitrosodimethylamine
NPW	SW-846 8270D	N-Nitroso-di-n-propylamine
NPW	SW-846 8270D	N-Nitrosomethylethylamine
NPW	SW-846 8270D	Benzidine
NPW	SW-846 8270D	Aniline
NPW	SW-846 8270D	Hexachloropropene
NPW	SW-846 8270D	Dibenzofuran
NPW	SW-846 8270D	Benzoic acid



<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8270D	N-Nitrosodiphenylamine
NPW	SW-846 8270D	Dichlorobenzidine (3,3'-)
NPW	SW-846 8270D	Chloroaniline (4-)
NPW	SW-846 8270D	Nitroaniline (2-)
NPW	SW-846 8270D	Nitroaniline (3-)
NPW	SW-846 8270D	Nitroaniline (4-)
NPW	SW-846 8270D	Chloronaphthalene (2-)
NPW	SW-846 8270D	Hexachlorobenzene
NPW	SW-846 8270D	Hexachlorobutadiene (1,3-)
NPW	SW-846 8270D	Hexachlorocyclopentadiene
NPW	SW-846 8270D	Hexachloroethane
NPW	SW-846 8270D	Trichlorobenzene (1,2,4-)
NPW	SW-846 8270D	Bis (2-chloroethoxy) methane
NPW	SW-846 8270D	Bis (2-chloroethyl) ether
NPW	SW-846 8270D	Bis (2-chloroisopropyl) ether
NPW	SW-846 8270D	Chlorophenyl-phenyl ether (4-)
NPW	SW-846 8270D	Bromophenyl-phenyl ether (4-)
NPW	SW-846 8270D	Dinitrotoluene (2,4-)
NPW	SW-846 8270D	Dinitrotoluene (2,6-)
NPW	SW-846 8270D	Isophorone
NPW	SW-846 8270D	Nitrobenzene
NPW	SW-846 8270D	Butyl benzyl phthalate
NPW	SW-846 8270D	Bis (2-ethylhexyl) phthalate
NPW	SW-846 8270D	Diethyl phthalate
NPW	SW-846 8270D	Dimethyl phthalate
NPW	SW-846 8270D	Di-n-butyl phthalate
NPW	SW-846 8270D	Di-n-octyl phthalate
NPW	SW-846 8270D	Acenaphthene
NPW	SW-846 8270D	Anthracene
NPW	SW-846 8270D	Acenaphthylene
NPW	SW-846 8270D	Benzo(a)anthracene
NPW	SW-846 8270D	Benzo(a)pyrene
NPW	SW-846 8270D	Benzo(b)fluoranthene
NPW	SW-846 8270D	Benzo(ghi)perylene
NPW	SW-846 8270D	Benzo(k)fluoranthene
NPW	SW-846 8270D	Chrysene
NPW	SW-846 8270D	Dibenzo(a,h)anthracene
NPW	SW-846 8270D	Fluoranthene
NPW	SW-846 8270D	Fluorene
NPW	SW-846 8270D	Indeno(1,2,3-cd)pyrene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8270D	Methylnaphthalene (2-)
NPW	SW-846 8270D	Naphthalene
NPW	SW-846 8270D	Phenanthrene
NPW	SW-846 8270D	Pyrene
NPW	SW-846 8270D	Methyl phenol (4-chloro-3-)
NPW	SW-846 8270D	Chlorophenol (2-)
NPW	SW-846 8270D	Dichlorophenol (2,4-)
NPW	SW-846 8270D	Dimethylphenol (2,4-)
NPW	SW-846 8270D	Dinitrophenol (2,4-)
NPW	SW-846 8270D	Dinitrophenol (2-methyl-4,6-)
NPW	SW-846 8270D	Methylphenol (2-)
NPW	SW-846 8270D	Methylphenol (4-)
NPW	SW-846 8270D	Nitrophenol (2-)
NPW	SW-846 8270D	Nitrophenol (4-)
NPW	SW-846 8270D	Pentachlorophenol
NPW	SW-846 8270D	Phenol
NPW	SW-846 8270D	Trichlorophenol (2,4,5-)
NPW	SW-846 8270D	Trichlorophenol (2,4,6-)
NPW	SW-846 8270D	Dichlorobenzene (1,4-)
NPW	SW-846 8270D	Pyridine
NPW	SW-846 8310	Acenaphthene
NPW	SW-846 8310	Acenaphthylene
NPW	SW-846 8310	Anthracene
NPW	SW-846 8310	Benzo(a)anthracene
NPW	SW-846 8310	Benzo(a)pyrene
NPW	SW-846 8310	Benzo(b)fluoranthene
NPW	SW-846 8310	Benzo(ghi)perylene
NPW	SW-846 8310	Benzo(k)fluoranthene
NPW	SW-846 8310	Chrysene
NPW	SW-846 8310	Dibenzo(a,h)anthracene
NPW	SW-846 8310	Fluoranthene
NPW	SW-846 8310	Fluorene
NPW	SW-846 8310	Indeno(1,2,3-cd)pyrene
NPW	SW-846 8310	Naphthalene
NPW	SW-846 8310	Phenanthrene
NPW	SW-846 8310	Pyrene
NPW	SW-846 8330	Nitroglycerine
NPW	SW-846 8330	Guanidine nitrate
NPW	SW-846 8330	PETN
NPW	SW-846 8330	HMX

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 8330	RDX
NPW	SW-846 8330	Trinitrobenzene (1,3,5-)
NPW	SW-846 8330	Dinitrobenzene (1,3-)
NPW	SW-846 8330	Tetryl
NPW	SW-846 8330	Nitrobenzene
NPW	SW-846 8330	Trinitrotoluene (2,4,6-)
NPW	SW-846 8330	Dinitrotoluene (4-amino-2,6-)
NPW	SW-846 8330	Dinitrotoluene (2-amino-4,6-)
NPW	SW-846 8330	Dinitrotoluene (2,4-)
NPW	SW-846 8330	Dinitrotoluene (2,6-)
NPW	SW-846 8330	Nitrotoluene (2-)
NPW	SW-846 8330	Nitrotoluene (3-)
NPW	SW-846 8330	Nitrotoluene (4-)
NPW	SW-846 8330A	Nitroglycerine
NPW	SW-846 8330A	PETN
NPW	SW-846 8330A	HMX
NPW	SW-846 8330A	RDX
NPW	SW-846 8330A	Trinitrobenzene (1,3,5-)
NPW	SW-846 8330A	Dinitrobenzene (1,3-)
NPW	SW-846 8330A	Tetryl
NPW	SW-846 8330A	Nitrobenzene
NPW	SW-846 8330A	Trinitrotoluene (2,4,6-)
NPW	SW-846 8330A	Dinitrotoluene (4-amino-2,6-)
NPW	SW-846 8330A	Dinitrotoluene (2-amino-4,6-)
NPW	SW-846 8330A	Dinitrotoluene (2,4-)
NPW	SW-846 8330A	Dinitrotoluene (2,6-)
NPW	SW-846 8330A	Nitrotoluene (2-)
NPW	SW-846 8330A	Nitrotoluene (3-)
NPW	SW-846 8330A	Nitrotoluene (4-)
NPW	SW-846 9010C	Cyanide - amenable to Cl <sub>2</sub>
NPW	SW-846 9010C	Cyanide
NPW	SW-846 9012B	Cyanide
NPW	SW-846 9020B	Total organic halides (TOX)
NPW	SW-846 9030B	Sulfides, acid sol. & insol.
NPW	SW-846 9034	Sulfides, acid sol. & insol.
NPW	SW-846 9040B	Corrosivity - pH waste, >20% water
NPW	SW-846 9040B	pH
NPW	SW-846 9040C	Corrosivity - pH waste, >20% water
NPW	SW-846 9040C	pH
NPW	SW-846 9040C	pH - waste, >20% water

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	SW-846 9050A	Specific conductance
NPW	SW-846 9056	Bromide
NPW	SW-846 9056	Nitrite
NPW	SW-846 9056	Sulfate
NPW	SW-846 9056	Nitrate
NPW	SW-846 9056	Chloride
NPW	SW-846 9056	Fluoride
NPW	SW-846 9056A	Bromide
NPW	SW-846 9056A	Nitrite
NPW	SW-846 9056A	Sulfate
NPW	SW-846 9056A	Nitrate
NPW	SW-846 9056A	Chloride
NPW	SW-846 9056A	Fluoride
NPW	SW-846 9060	Total organic carbon (TOC)
NPW	SW-846 9060A	Total organic carbon (TOC)
NPW	SW-846 9066	Phenols
NPW	User Defined 5030C	Volatile organics
NPW	User Defined 8260C	Hexane (n-)
NPW	User Defined 9010B	Cyanide - amenable to Cl <sub>2</sub>
NPW	User Defined 9010B	Cyanide
NPW	User Defined 9012A	Cyanide
NPW	User Defined ASTM D93	Ignitability
NPW	User Defined CA LUFT - diesel	Petroleum Organics
NPW	User Defined CA LUFT - diesel	Petroleum Organics
NPW	User Defined EPA 1657	Parathion ethyl
NPW	User Defined EPA 1657	Azinphos methyl
NPW	User Defined EPA 1657	Demeton (o-)
NPW	User Defined EPA 1657	Demeton (s-)
NPW	User Defined EPA 1657	Diazinon
NPW	User Defined EPA 1657	Disulfoton
NPW	User Defined EPA 1657	Malathion
NPW	User Defined EPA 1657	Parathion methyl
NPW	User Defined EPA 353.2 Modified	Nitrocellulose
NPW	User Defined EPA 624	Dichlorodifluoromethane
NPW	User Defined LUFT	Xylene (m-)
NPW	User Defined LUFT	Xylene (o-)
NPW	User Defined LUFT	Xylene (p-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	User Defined LUFT	Benzene
NPW	User Defined LUFT	Ethylbenzene
NPW	User Defined LUFT	Toluene
NPW	User Defined LUFT	Xylenes (total)
NPW	User Defined LUFT	Methyl tert-butyl ether
NPW	User Defined MA- DEP-EPH, TN-EPH, WI DRO, NW TPH D <sub>x</sub>	Diesel range organic
NPW	User Defined MA- DEP-VPH, WI GRO, NW TPH G <sub>x</sub>	Gasoline range organic
NPW	User Defined NWTPH- D <sub>x</sub> , NWTPH-G <sub>x</sub> , NWTPHID	Petroleum Organics
NPW	User Defined SM 6200 B-97	Butanone (2-)
NPW	User Defined SM 6200 B-97	Carbon disulfide
NPW	User Defined SM 6200 B-97	Isopropanol
NPW	User Defined SM 6200 B-97	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
NPW	User Defined SM 6200 B-97	Vinyl acetate
NPW	User Defined SM 6200 B-97	Acetonitrile
NPW	User Defined SM 6200 B-97	Hexanone (2-)
NPW	User Defined SM 6200 B-97	Methyl iodide
NPW	User Defined SM 6200 B-97	Dibromoethane (1,2-) (EDB)
NPW	User Defined SM 6200 B-97	Dichlorodifluoromethane
NPW	User Defined SM 6200 B-97	Dichloroethene (cis-1,2-)
NPW	User Defined SM 6200 B-97	Hexane (n-)
NPW	User Defined SM 6200 B-97	Methyl isobutyl ketone (MIBK)
NPW	User Defined SM 6200 B-97	Tetrahydrofuran
NPW	User Defined SM 6200 B-97	Styrene
NPW	User Defined SM 6200 B-97	Xylene (o-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	User Defined SM 6200 B-97	Acetone
NPW	User Defined SM 6200 B-97	Ethyl acetate
NPW	User Defined SM 6200 B-97	Methyl tert-butyl ether
NPW	User Defined SM 6200 B-97	Tert-butyl alcohol
NPW	User Defined SM 6200 B-97	Xylenes (total)
NPW	User Defined SM 6200 B-97	Benzene
NPW	User Defined SM 6200 B-97	Bromodichloromethane
NPW	User Defined SM 6200 B-97	Bromoform
NPW	User Defined SM 6200 B-97	Bromomethane
NPW	User Defined SM 6200 B-97	Carbon tetrachloride
NPW	User Defined SM 6200 B-97	Chlorobenzene
NPW	User Defined SM 6200 B-97	Chloroethane
NPW	User Defined SM 6200 B-97	Chloroethyl vinyl ether (2-)
NPW	User Defined SM 6200 B-97	Chloroform
NPW	User Defined SM 6200 B-97	Chloromethane
NPW	User Defined SM 6200 B-97	Dibromochloromethane
NPW	User Defined SM 6200 B-97	Dichlorobenzene (1,2-)
NPW	User Defined SM 6200 B-97	Dichlorobenzene (1,3-)
NPW	User Defined SM 6200 B-97	Dichlorobenzene (1,4-)
NPW	User Defined SM 6200 B-97	Dichloroethane (1,1-)
NPW	User Defined SM 6200 B-97	Dichloroethane (1,2-)
NPW	User Defined SM 6200 B-97	Dichloroethene (1,1-)
NPW	User Defined SM 6200 B-97	Dichloroethene (trans-1,2-)
NPW	User Defined SM 6200	Dichloropropane (1,2-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	User Defined SM 6200 B-97	Dichloropropene (cis-1,3-)
NPW	User Defined SM 6200 B-97	Dichloropropene (trans-1,3-)
NPW	User Defined SM 6200 B-97	Ethylbenzene
NPW	User Defined SM 6200 B-97	Methylene chloride (Dichloromethane)
NPW	User Defined SM 6200 B-97	Tetrachloroethane (1,1,2,2-)
NPW	User Defined SM 6200 B-97	Tetrachloroethene
NPW	User Defined SM 6200 B-97	Toluene
NPW	User Defined SM 6200 B-97	Trichloroethane (1,1,1-)
NPW	User Defined SM 6200 B-97	Trichloroethane (1,1,2-)
NPW	User Defined SM 6200 B-97	Trichloroethene
NPW	User Defined SM 6200 B-97	Trichlorofluoromethane
NPW	User Defined SM 6200 B-97	Vinyl chloride
NPW	User Defined SM 6200C-97	Benzene
NPW	User Defined SM 6200C-97	Ethylbenzene
NPW	User Defined SM 6200C-97	Methyl tert-butyl ether
NPW	User Defined SM 6200C-97	Tert-butyl alcohol
NPW	User Defined SM 6200C-97	Toluene
NPW	User Defined SM 6200C-97	Xylenes (total)
NPW	User Defined SM 6630C-00	Chlordane (alpha)
NPW	User Defined SM 6630C-00	Chlordane (gamma)
NPW	User Defined SM 6630C-00	Hexachlorobenzene
NPW	User Defined SM 6630C-00	Endrin aldehyde
NPW	User Defined SM 6630C-00	Endrin ketone

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
NPW	User Defined SM 6640B-01	Dinoseb
NPW	User Defined SM 6640B-01	Dicamba
NPW	User Defined SW846 8260B & 8260C	Gasoline range organic
NPW	User Defined SW-846 8330	Nitroguanidine
NPW	User Defined TX 1005, TX 1006, CT ETPH, NW TPH ID	Petroleum Organics
SCM	EPA 314.0-mod	Perchlorate
SCM	ASTM D240	Heat of combustion (BTU)
SCM	ASTM D5468 and D482	% ash
SCM	ASTM F1647-02A	Total organic carbon (TOC)
SCM	EPA 300.0	Guanidine nitrate
SCM	Other FL - PRO	Petroleum Organics
SCM	Other IA - OA-1	Petroleum Organics
SCM	Other IA - OA-2	Petroleum Organics
SCM	Other NJ DEP EPH 10/08, Rev. 3	Extractable Petroleum Hydrocarbons
SCM	Other NJ-OQA-QAM- 025, Rev. 7	Petroleum Organics
SCM	Other USDA-LOI (Loss on ignition)	Total organic carbon (TOC)
SCM	Other Walkley Black	Total organic carbon (TOC)
SCM	SM 2540 G SM 18th Ed.	Total, fixed, and volatile solids (SQAR)
SCM	SM 9222D-97 (Class B only) plus EPA 625/R- 92/013 App. F	Fecal coliform
SCM	SW-846 1010	Ignitability
SCM	SW-846 1010A	Ignitability
SCM	SW-846 1030	Ignitability of solids
SCM	SW-846 1110	Corrosivity toward steel
SCM	SW-846 1110A	Corrosivity toward steel
SCM	SW-846 1310A	Metals - organics
SCM	SW-846 1310B	Metals - organics
SCM	SW-846 1311	Volatile organics
SCM	SW-846 1311	Semivolatile organics
SCM	SW-846 1311	Metals
SCM	SW-846 1312	Metals - organics
SCM	SW-846 1320	Metals - organics



<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 3031	Metals
SCM	SW-846 3040A	Metals
SCM	SW-846 3050B	Metals
SCM	SW-846 3051	Metals
SCM	SW-846 3051A	Metals
SCM	SW-846 3052	Metals
SCM	SW-846 3060A	Metals
SCM	SW-846 3540C	Semivolatile organics
SCM	SW-846 3546	Semivolatile organics
SCM	SW-846 3550B	Semivolatile organics
SCM	SW-846 3550C	Semivolatile organics
SCM	SW-846 3580A	Organics
SCM	SW-846 3585	Organics
SCM	SW-846 3610B	Semivolatile organics
SCM	SW-846 3611B	Semivolatile organics
SCM	SW-846 3620B	Semivolatile organics
SCM	SW-846 3620C	Semivolatile organics
SCM	SW-846 3630C	Semivolatile organics
SCM	SW-846 3660B	Semivolatile organics
SCM	SW-846 3665A	Semivolatile organics
SCM	SW-846 5035A-H	Volatile organics - high conc.
SCM	SW-846 5035A-L	Volatile organics - low conc.
SCM	SW-846 5035H	Volatile organics - high conc.
SCM	SW-846 5035L	Volatile organics - low conc.
SCM	SW-846 6010B	Aluminum
SCM	SW-846 6010B	Antimony
SCM	SW-846 6010B	Arsenic
SCM	SW-846 6010B	Barium
SCM	SW-846 6010B	Beryllium
SCM	SW-846 6010B	Boron
SCM	SW-846 6010B	Cadmium
SCM	SW-846 6010B	Calcium
SCM	SW-846 6010B	Calcium-hardness
SCM	SW-846 6010B	Total hardness
SCM	SW-846 6010B	Chromium
SCM	SW-846 6010B	Cobalt
SCM	SW-846 6010B	Copper
SCM	SW-846 6010B	Iron
SCM	SW-846 6010B	Lead
SCM	SW-846 6010B	Lithium

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 6010B	Magnesium
SCM	SW-846 6010B	Manganese
SCM	SW-846 6010B	Molybdenum
SCM	SW-846 6010B	Nickel
SCM	SW-846 6010B	Potassium
SCM	SW-846 6010B	Selenium
SCM	SW-846 6010B	Silica
SCM	SW-846 6010B	Silver
SCM	SW-846 6010B	Sulfur
SCM	SW-846 6010B	Sodium
SCM	SW-846 6010B	Strontium
SCM	SW-846 6010B	Thallium
SCM	SW-846 6010B	Tin
SCM	SW-846 6010B	Titanium
SCM	SW-846 6010B	Vanadium
SCM	SW-846 6010B	Zinc
SCM	SW-846 6010C	Aluminum
SCM	SW-846 6010C	Antimony
SCM	SW-846 6010C	Arsenic
SCM	SW-846 6010C	Barium
SCM	SW-846 6010C	Beryllium
SCM	SW-846 6010C	Boron
SCM	SW-846 6010C	Cadmium
SCM	SW-846 6010C	Calcium
SCM	SW-846 6010C	Calcium-hardness
SCM	SW-846 6010C	Total hardness
SCM	SW-846 6010C	Chromium
SCM	SW-846 6010C	Cobalt
SCM	SW-846 6010C	Copper
SCM	SW-846 6010C	Iron
SCM	SW-846 6010C	Lead
SCM	SW-846 6010C	Lithium
SCM	SW-846 6010C	Magnesium
SCM	SW-846 6010C	Manganese
SCM	SW-846 6010C	Molybdenum
SCM	SW-846 6010C	Nickel
SCM	SW-846 6010C	Potassium
SCM	SW-846 6010C	Selenium
SCM	SW-846 6010C	Silica
SCM	SW-846 6010C	Silver

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 6010C	Sulfur
SCM	SW-846 6010C	Sodium
SCM	SW-846 6010C	Strontium
SCM	SW-846 6010C	Thallium
SCM	SW-846 6010C	Tin
SCM	SW-846 6010C	Titanium
SCM	SW-846 6010C	Vanadium
SCM	SW-846 6010C	Zinc
SCM	SW-846 6020	Aluminum
SCM	SW-846 6020	Antimony
SCM	SW-846 6020	Arsenic
SCM	SW-846 6020	Barium
SCM	SW-846 6020	Beryllium
SCM	SW-846 6020	Boron
SCM	SW-846 6020	Cadmium
SCM	SW-846 6020	Calcium
SCM	SW-846 6020	Chromium
SCM	SW-846 6020	Cobalt
SCM	SW-846 6020	Copper
SCM	SW-846 6020	Iron
SCM	SW-846 6020	Lead
SCM	SW-846 6020	Magnesium
SCM	SW-846 6020	Manganese
SCM	SW-846 6020	Molybdenum
SCM	SW-846 6020	Nickel
SCM	SW-846 6020	Potassium
SCM	SW-846 6020	Selenium
SCM	SW-846 6020	Silver
SCM	SW-846 6020	Sodium
SCM	SW-846 6020	Strontium
SCM	SW-846 6020	Thallium
SCM	SW-846 6020	Thorium
SCM	SW-846 6020	Tin
SCM	SW-846 6020	Titanium
SCM	SW-846 6020	Uranium
SCM	SW-846 6020	Vanadium
SCM	SW-846 6020	Zinc
SCM	SW-846 6020A	Aluminum
SCM	SW-846 6020A	Antimony
SCM	SW-846 6020A	Arsenic

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 6020A	Barium
SCM	SW-846 6020A	Beryllium
SCM	SW-846 6020A	Boron
SCM	SW-846 6020A	Cadmium
SCM	SW-846 6020A	Calcium
SCM	SW-846 6020A	Chromium
SCM	SW-846 6020A	Cobalt
SCM	SW-846 6020A	Copper
SCM	SW-846 6020A	Iron
SCM	SW-846 6020A	Lead
SCM	SW-846 6020A	Magnesium
SCM	SW-846 6020A	Manganese
SCM	SW-846 6020A	Molybdenum
SCM	SW-846 6020A	Nickel
SCM	SW-846 6020A	Potassium
SCM	SW-846 6020A	Selenium
SCM	SW-846 6020A	Silver
SCM	SW-846 6020A	Sodium
SCM	SW-846 6020A	Strontium
SCM	SW-846 6020A	Thallium
SCM	SW-846 6020A	Thorium
SCM	SW-846 6020A	Tin
SCM	SW-846 6020A	Titanium
SCM	SW-846 6020A	Uranium
SCM	SW-846 6020A	Vanadium
SCM	SW-846 6020A	Zinc
SCM	SW-846 7.3.3.2	Reactivity
SCM	SW-846 7.3.4.2	Reactivity
SCM	SW-846 7196A	Chromium (VI)
SCM	SW-846 7199	Chromium (VI)
SCM	SW-846 7471A	Mercury - solid waste
SCM	SW-846 7471B	Mercury - solid waste
SCM	SW-846 8011	Dibromoethane (1,2-) (EDB)
SCM	SW-846 8011	Dibromo-3-chloropropane (1,2-)
SCM	SW-846 8015B	Ethylene glycol
SCM	SW-846 8015B	Propylene glycol
SCM	SW-846 8015B	Gasoline range organic
SCM	SW-846 8015B	Diesel range organic
SCM	SW-846 8015C	Ethylene glycol
SCM	SW-846 8015C	Propylene glycol

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8015D	Ethylene glycol
SCM	SW-846 8015D	Propylene glycol
SCM	SW-846 8015D	Gasoline range organic
SCM	SW-846 8015D	Diesel range organic
SCM	SW-846 8021B	Xylenes (total)
SCM	SW-846 8021B	Methyl tert-butyl ether
SCM	SW-846 8021B	Benzene
SCM	SW-846 8021B	Ethylbenzene
SCM	SW-846 8021B	Toluene
SCM	SW-846 8021B	Xylene (o-)
SCM	SW-846 8021B	Xylene (m-)
SCM	SW-846 8021B	Xylene (p-)
SCM	SW-846 8081A	Alachlor
SCM	SW-846 8081A	Chlordane (alpha)
SCM	SW-846 8081A	Chlordane (gamma)
SCM	SW-846 8081A	Chloroneb
SCM	SW-846 8081A	Chlorothalonil
SCM	SW-846 8081A	Etridiazole
SCM	SW-846 8081A	Hexachlorobenzene
SCM	SW-846 8081A	Hexachlorocyclopentadiene
SCM	SW-846 8081A	Permethrin
SCM	SW-846 8081A	Propachlor
SCM	SW-846 8081A	Trifluralin
SCM	SW-846 8081A	Aldrin
SCM	SW-846 8081A	Alpha BHC
SCM	SW-846 8081A	Beta BHC
SCM	SW-846 8081A	Delta BHC
SCM	SW-846 8081A	Lindane (gamma BHC)
SCM	SW-846 8081A	Chlordane (technical)
SCM	SW-846 8081A	DDD (4,4'-)
SCM	SW-846 8081A	DDE (4,4'-)
SCM	SW-846 8081A	DDT (4,4'-)
SCM	SW-846 8081A	Dieldrin
SCM	SW-846 8081A	Endosulfan I
SCM	SW-846 8081A	Endosulfan II
SCM	SW-846 8081A	Endosulfan sulfate
SCM	SW-846 8081A	Endrin
SCM	SW-846 8081A	Endrin aldehyde
SCM	SW-846 8081A	Endrin ketone
SCM	SW-846 8081A	Heptachlor

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8081A	Heptachlor epoxide
SCM	SW-846 8081A	Methoxychlor
SCM	SW-846 8081A	Toxaphene
SCM	SW-846 8081B	Alachlor
SCM	SW-846 8081B	Chlordane (alpha)
SCM	SW-846 8081B	Chlordane (gamma)
SCM	SW-846 8081B	Chloroneb
SCM	SW-846 8081B	Chlorothalonil
SCM	SW-846 8081B	Etridiazole
SCM	SW-846 8081B	Hexachlorobenzene
SCM	SW-846 8081B	Hexachlorocyclopentadiene
SCM	SW-846 8081B	Permethrin
SCM	SW-846 8081B	Propachlor
SCM	SW-846 8081B	Trifluralin
SCM	SW-846 8081B	Aldrin
SCM	SW-846 8081B	Alpha BHC
SCM	SW-846 8081B	Beta BHC
SCM	SW-846 8081B	Delta BHC
SCM	SW-846 8081B	Lindane (gamma BHC)
SCM	SW-846 8081B	Chlordane (technical)
SCM	SW-846 8081B	DDD (4,4'-)
SCM	SW-846 8081B	DDE (4,4'-)
SCM	SW-846 8081B	DDT (4,4'-)
SCM	SW-846 8081B	Dieldrin
SCM	SW-846 8081B	Endosulfan I
SCM	SW-846 8081B	Endosulfan II
SCM	SW-846 8081B	Endosulfan sulfate
SCM	SW-846 8081B	Endrin
SCM	SW-846 8081B	Endrin aldehyde
SCM	SW-846 8081B	Endrin ketone
SCM	SW-846 8081B	Heptachlor
SCM	SW-846 8081B	Heptachlor epoxide
SCM	SW-846 8081B	Methoxychlor
SCM	SW-846 8081B	Toxaphene
SCM	SW-846 8082	PCB 1016
SCM	SW-846 8082	PCB 1221
SCM	SW-846 8082	PCB 1232
SCM	SW-846 8082	PCB 1242
SCM	SW-846 8082	PCB 1248
SCM	SW-846 8082	PCB 1254

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8082	PCB 1260
SCM	SW-846 8082A	PCB 1016
SCM	SW-846 8082A	PCB 1221
SCM	SW-846 8082A	PCB 1232
SCM	SW-846 8082A	PCB 1242
SCM	SW-846 8082A	PCB 1248
SCM	SW-846 8082A	PCB 1254
SCM	SW-846 8082A	PCB 1260
SCM	SW-846 8141A	Azinphos methyl
SCM	SW-846 8141A	Chlorpyrifos
SCM	SW-846 8141A	Demeton (o-)
SCM	SW-846 8141A	Demeton (s-)
SCM	SW-846 8141A	Disulfoton
SCM	SW-846 8141A	Bolstar
SCM	SW-846 8141A	Coumaphos
SCM	SW-846 8141A	Dichlorvos
SCM	SW-846 8141A	Dimethoate
SCM	SW-846 8141A	EPN
SCM	SW-846 8141A	Ethoprop
SCM	SW-846 8141A	Fensulfothion
SCM	SW-846 8141A	Fenthion
SCM	SW-846 8141A	Merphos
SCM	SW-846 8141A	Mevinphos
SCM	SW-846 8141A	Naled
SCM	SW-846 8141A	Parathion
SCM	SW-846 8141A	Parathion methyl
SCM	SW-846 8141A	Phorate
SCM	SW-846 8141A	Ronnel
SCM	SW-846 8141A	Stirofos
SCM	SW-846 8141A	Sulfotepp
SCM	SW-846 8141A	TEPP
SCM	SW-846 8141A	Tokuthion [Protothiofos]
SCM	SW-846 8141A	Trichloronate
SCM	SW-846 8141A	Diazinon
SCM	SW-846 8141A	Malathion
SCM	SW-846 8141B	Azinphos methyl
SCM	SW-846 8141B	Chlorpyrifos
SCM	SW-846 8141B	Demeton (o-)
SCM	SW-846 8141B	Demeton (s-)
SCM	SW-846 8141B	Disulfoton

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8141B	Bolstar
SCM	SW-846 8141B	Coumaphos
SCM	SW-846 8141B	Dichlorvos
SCM	SW-846 8141B	Dimethoate
SCM	SW-846 8141B	EPN
SCM	SW-846 8141B	Ethoprop
SCM	SW-846 8141B	Fensulfothion
SCM	SW-846 8141B	Fenthion
SCM	SW-846 8141B	Merphos
SCM	SW-846 8141B	Mevinphos
SCM	SW-846 8141B	Naled
SCM	SW-846 8141B	Parathion
SCM	SW-846 8141B	Parathion methyl
SCM	SW-846 8141B	Phorate
SCM	SW-846 8141B	Ronnel
SCM	SW-846 8141B	Stirofos
SCM	SW-846 8141B	Sulfotepp
SCM	SW-846 8141B	TEPP
SCM	SW-846 8141B	Tokuthion [Protothiofos]
SCM	SW-846 8141B	Trichloronate
SCM	SW-846 8141B	Diazinon
SCM	SW-846 8141B	Malathion
SCM	SW-846 8151A	Dicamba
SCM	SW-846 8151A	DB (2,4-)
SCM	SW-846 8151A	Dinoseb
SCM	SW-846 8151A	Dalapon
SCM	SW-846 8151A	Dichlorprop
SCM	SW-846 8151A	D (2,4-)
SCM	SW-846 8151A	T (2,4,5-)
SCM	SW-846 8151A	TP (2,4,5-) (Silvex)
SCM	SW-846 8151A	MCPA
SCM	SW-846 8151A	MCPP
SCM	SW-846 8015M	Methyl alcohol (Methanol)
SCM	SW-846 8260B	Ethyl alcohol
SCM	SW-846 8260B	Hexane (n-)
SCM	SW-846 8260B	Methylnaphthalene (1-)
SCM	SW-846 8260B	Methylnaphthalene (2-)
SCM	SW-846 8260B	Butanol (3,3-Dimethyl-1-)
SCM	SW-846 8260B	Trimethylpentane (2,2,4-)
SCM	SW-846 8260B	Trimethylbenzene (1,2,3-)



<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8260B	Cyclohexane
SCM	SW-846 8260B	Butanol (1-)
SCM	SW-846 8260B	Nitropropane (2-)
SCM	SW-846 8260B	Butyl formate (t-)
SCM	SW-846 8260B	Methyl acetate
SCM	SW-846 8260B	Amyl alcohol (t-)
SCM	SW-846 8260B	Methylcyclohexane
SCM	SW-846 8260B	Octane (-n)
SCM	SW-846 8260B	tert-Amyl Methyl Ether [TAME]
SCM	SW-846 8260B	Bromoethane
SCM	SW-846 8260B	Cyclohexanone
SCM	SW-846 8260B	Diisopropyl Ether [DIPE]
SCM	SW-846 8260B	Tetrahydrofuran
SCM	SW-846 8260B	Ethyl-tert-butyl Ether [ETBE]
SCM	SW-846 8260B	Xylene (m-)
SCM	SW-846 8260B	Xylene (o-)
SCM	SW-846 8260B	Xylene (p-)
SCM	SW-846 8260B	Dichloro-2-butene (cis-1,4-)
SCM	SW-846 8260B	Diethyl ether (Ethyl ether)
SCM	SW-846 8260B	Dichloro-2-butene (trans-1,4-)
SCM	SW-846 8260B	Ethanol
SCM	SW-846 8260B	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
SCM	SW-846 8260B	Vinyl acetate
SCM	SW-846 8260B	Pentachloroethane
SCM	SW-846 8260B	Tert-butyl alcohol
SCM	SW-846 8260B	Dioxane (1,4-)
SCM	SW-846 8260B	Bromobenzene
SCM	SW-846 8260B	Butyl benzene (n-)
SCM	SW-846 8260B	Sec-butylbenzene
SCM	SW-846 8260B	Tert-butylbenzene
SCM	SW-846 8260B	Chlorotoluene (2-)
SCM	SW-846 8260B	Chlorotoluene (4-)
SCM	SW-846 8260B	Isopropylbenzene
SCM	SW-846 8260B	Propylbenzene (n-)
SCM	SW-846 8260B	Isopropyltoluene (4-)
SCM	SW-846 8260B	Trichlorobenzene (1,2,3-)
SCM	SW-846 8260B	Trimethylbenzene (1,2,4-)
SCM	SW-846 8260B	Trimethylbenzene (1,3,5-)
SCM	SW-846 8260B	Allyl chloride
SCM	SW-846 8260B	Bromochloromethane

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8260B	Butadiene (2-chloro-1,3-)
SCM	SW-846 8260B	Dibromoethane (1,2-) (EDB)
SCM	SW-846 8260B	Dibromomethane
SCM	SW-846 8260B	Dibromo-3-chloropropane (1,2-)
SCM	SW-846 8260B	Dichloropropane (1,3-)
SCM	SW-846 8260B	Dichloropropane (2,2-)
SCM	SW-846 8260B	Dichloropropene (1,1-)
SCM	SW-846 8260B	Trichloropropane (1,2,3-)
SCM	SW-846 8260B	Ethyl acetate
SCM	SW-846 8260B	Ethyl methacrylate
SCM	SW-846 8260B	Methacrylonitrile
SCM	SW-846 8260B	Methyl acrylate
SCM	SW-846 8260B	Methyl methacrylate
SCM	SW-846 8260B	Iso-butyl alcohol
SCM	SW-846 8260B	Isopropanol
SCM	SW-846 8260B	N-Nitroso-di-n-butylamine
SCM	SW-846 8260B	Propionitrile
SCM	SW-846 8260B	Acetonitrile
SCM	SW-846 8260B	Benzene
SCM	SW-846 8260B	Chlorobenzene
SCM	SW-846 8260B	Dichlorobenzene (1,2-)
SCM	SW-846 8260B	Dichlorobenzene (1,3-)
SCM	SW-846 8260B	Dichlorobenzene (1,4-)
SCM	SW-846 8260B	Ethylbenzene
SCM	SW-846 8260B	Toluene
SCM	SW-846 8260B	Xylenes (total)
SCM	SW-846 8260B	Bromodichloromethane
SCM	SW-846 8260B	Bromoform
SCM	SW-846 8260B	Bromomethane
SCM	SW-846 8260B	Carbon tetrachloride
SCM	SW-846 8260B	Chloroethane
SCM	SW-846 8260B	Chloroethyl vinyl ether (2-)
SCM	SW-846 8260B	Chloroform
SCM	SW-846 8260B	Chloromethane
SCM	SW-846 8260B	Dichloropropene (trans-1,3-)
SCM	SW-846 8260B	Dibromochloromethane
SCM	SW-846 8260B	Dichlorodifluoromethane
SCM	SW-846 8260B	Dichloroethane (1,1-)
SCM	SW-846 8260B	Dichloroethane (1,2-)
SCM	SW-846 8260B	Dichloroethene (1,1-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8260B	Dichloroethene (trans-1,2-)
SCM	SW-846 8260B	Dichloroethene (cis-1,2-)
SCM	SW-846 8260B	Dichloropropane (1,2-)
SCM	SW-846 8260B	Dichloropropene (cis-1,3-)
SCM	SW-846 8260B	Methylene chloride (Dichloromethane)
SCM	SW-846 8260B	Tetrachloroethane (1,1,2,2-)
SCM	SW-846 8260B	Tetrachloroethene
SCM	SW-846 8260B	Trichloroethane (1,1,1-)
SCM	SW-846 8260B	Trichloroethane (1,1,2-)
SCM	SW-846 8260B	Trichloroethene
SCM	SW-846 8260B	Trichlorofluoromethane
SCM	SW-846 8260B	Vinyl chloride
SCM	SW-846 8260B	Acetone
SCM	SW-846 8260B	Carbon disulfide
SCM	SW-846 8260B	Butanone (2-)
SCM	SW-846 8260B	Hexanone (2-)
SCM	SW-846 8260B	Pentanone (4-methyl-2-) (MIBK)
SCM	SW-846 8260B	Methyl tert-butyl ether
SCM	SW-846 8260B	Acrolein
SCM	SW-846 8260B	Acrylonitrile
SCM	SW-846 8260B	Hexachlorobutadiene (1,3-)
SCM	SW-846 8260B	Hexachloroethane
SCM	SW-846 8260B	Naphthalene
SCM	SW-846 8260B	Styrene
SCM	SW-846 8260B	Tetrachloroethane (1,1,1,2-)
SCM	SW-846 8260B	Trichlorobenzene (1,2,4-)
SCM	SW-846 8260C	Ethyl alcohol
SCM	SW-846 8260C	Methylnaphthalene (1-)
SCM	SW-846 8260C	Methylnaphthalene (2-)
SCM	SW-846 8260C	Butanol (3,3-Dimethyl-1-)
SCM	SW-846 8260C	Trimethylbenzene (1,2,3-)
SCM	SW-846 8260C	Cyclohexane
SCM	SW-846 8260C	Butanol (1-)
SCM	SW-846 8260C	Nitropropane (2-)
SCM	SW-846 8260C	Butyl formate (t-)
SCM	SW-846 8260C	Methyl acetate
SCM	SW-846 8260C	Pentanol (2-Methyl-2-)
SCM	SW-846 8260C	Amyl alcohol (t-)
SCM	SW-846 8260C	Methylcyclohexane
SCM	SW-846 8260C	Octane (-n)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8260C	tert-Amylmethyl ether [TAME]
SCM	SW-846 8260C	Bromoethane
SCM	SW-846 8260C	Cyclohexanone
SCM	SW-846 8260C	Diisopropyl Ether [DIPE]
SCM	SW-846 8260C	Tetrahydrofuran
SCM	SW-846 8260C	Ethyl-tert-butyl Ether [ETBE]
SCM	SW-846 8260C	Xylene (m-)
SCM	SW-846 8260C	Xylene (o-)
SCM	SW-846 8260C	Xylene (p-)
SCM	SW-846 8260C	Dichloro-2-butene (cis-1,4-)
SCM	SW-846 8260C	Diethyl ether (Ethyl ether)
SCM	SW-846 8260C	Dichloro-2-butene (trans-1,4-)
SCM	SW-846 8260C	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
SCM	SW-846 8260C	Vinyl acetate
SCM	SW-846 8260C	Pentachloroethane
SCM	SW-846 8260C	Tert-butyl alcohol
SCM	SW-846 8260C	Dioxane (1,4-)
SCM	SW-846 8260C	Bromobenzene
SCM	SW-846 8260C	Butyl benzene (n-)
SCM	SW-846 8260C	Sec-butylbenzene
SCM	SW-846 8260C	Tert-butylbenzene
SCM	SW-846 8260C	Chlorotoluene (2-)
SCM	SW-846 8260C	Chlorotoluene (4-)
SCM	SW-846 8260C	Isopropylbenzene
SCM	SW-846 8260C	Propylbenzene (n-)
SCM	SW-846 8260C	Isopropyltoluene (4-)
SCM	SW-846 8260C	Trichlorobenzene (1,2,3-)
SCM	SW-846 8260C	Trimethylbenzene (1,2,4-)
SCM	SW-846 8260C	Trimethylbenzene (1,3,5-)
SCM	SW-846 8260C	Allyl chloride
SCM	SW-846 8260C	Bromochloromethane
SCM	SW-846 8260C	Butadiene (2-chloro-1,3-)
SCM	SW-846 8260C	Dibromoethane (1,2-) (EDB)
SCM	SW-846 8260C	Dibromomethane
SCM	SW-846 8260C	Dibromo-3-chloropropane (1,2-)
SCM	SW-846 8260C	Dichloropropane (1,3-)
SCM	SW-846 8260C	Dichloropropane (2,2-)
SCM	SW-846 8260C	Dichloropropene (1,1-)
SCM	SW-846 8260C	Trichloropropane (1,2,3-)
SCM	SW-846 8260C	Ethyl acetate

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8260C	Ethyl methacrylate
SCM	SW-846 8260C	Methacrylonitrile
SCM	SW-846 8260C	Methyl acrylate
SCM	SW-846 8260C	Methyl methacrylate
SCM	SW-846 8260C	Iso-butyl alcohol
SCM	SW-846 8260C	Isopropanol
SCM	SW-846 8260C	N-Nitroso-di-n-butylamine
SCM	SW-846 8260C	Propionitrile
SCM	SW-846 8260C	Acetonitrile
SCM	SW-846 8260C	Benzene
SCM	SW-846 8260C	Chlorobenzene
SCM	SW-846 8260C	Dichlorobenzene (1,2-)
SCM	SW-846 8260C	Dichlorobenzene (1,3-)
SCM	SW-846 8260C	Dichlorobenzene (1,4-)
SCM	SW-846 8260C	Ethylbenzene
SCM	SW-846 8260C	Toluene
SCM	SW-846 8260C	Xylenes (total)
SCM	SW-846 8260C	Bromodichloromethane
SCM	SW-846 8260C	Bromoform
SCM	SW-846 8260C	Bromomethane
SCM	SW-846 8260C	Carbon tetrachloride
SCM	SW-846 8260C	Chloroethane
SCM	SW-846 8260C	Chloroethyl vinyl ether (2-)
SCM	SW-846 8260C	Chloroform
SCM	SW-846 8260C	Chloromethane
SCM	SW-846 8260C	Dichloropropene (trans-1,3-)
SCM	SW-846 8260C	Dibromochloromethane
SCM	SW-846 8260C	Dichlorodifluoromethane
SCM	SW-846 8260C	Dichloroethane (1,1-)
SCM	SW-846 8260C	Dichloroethane (1,2-)
SCM	SW-846 8260C	Dichloroethene (1,1-)
SCM	SW-846 8260C	Dichloroethene (trans-1,2-)
SCM	SW-846 8260C	Dichloroethene (cis-1,2-)
SCM	SW-846 8260C	Dichloropropane (1,2-)
SCM	SW-846 8260C	Dichloropropene (cis-1,3-)
SCM	SW-846 8260C	Methylene chloride (Dichloromethane)
SCM	SW-846 8260C	Tetrachloroethane (1,1,2,2-)
SCM	SW-846 8260C	Tetrachloroethene
SCM	SW-846 8260C	Trichloroethane (1,1,1-)
SCM	SW-846 8260C	Trichloroethane (1,1,2-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8260C	Trichloroethene
SCM	SW-846 8260C	Trichlorofluoromethane
SCM	SW-846 8260C	Vinyl chloride
SCM	SW-846 8260C	Acetone
SCM	SW-846 8260C	Carbon disulfide
SCM	SW-846 8260C	Butanone (2-)
SCM	SW-846 8260C	Hexanone (2-)
SCM	SW-846 8260C	Pentanone (4-methyl-2-) (MIBK)
SCM	SW-846 8260C	Methyl tert-butyl ether
SCM	SW-846 8260C	Acrolein
SCM	SW-846 8260C	Acrylonitrile
SCM	SW-846 8260C	Hexachlorobutadiene (1,3-)
SCM	SW-846 8260C	Hexachloroethane
SCM	SW-846 8260C	Naphthalene
SCM	SW-846 8260C	Styrene
SCM	SW-846 8260C	Tetrachloroethane (1,1,1,2-)
SCM	SW-846 8260C	Trichlorobenzene (1,2,4-)
SCM	SW-846 8270C	Biphenyl (1,1'-)
SCM	SW-846 8270C	Caprolactam
SCM	SW-846 8270C	Atrazine
SCM	SW-846 8270C	Phenanthrene
SCM	SW-846 8270C	Pyrene
SCM	SW-846 8270C	Acenaphthene
SCM	SW-846 8270C	Acenaphthylene
SCM	SW-846 8270C	Anthracene
SCM	SW-846 8270C	Benzo(g,h,i)perylene
SCM	SW-846 8270C	Chrysene
SCM	SW-846 8270C	Methylnaphthalene (1-)
SCM	SW-846 8270C	Methylnaphthalene (2-)
SCM	SW-846 8270C	Naphthalene
SCM	SW-846 8270C	Fluoranthene
SCM	SW-846 8270C	Fluorene
SCM	SW-846 8270C	Methylnaphthalene (1-)
SCM	SW-846 8270C	Nitrodiphenylamine (2-)
SCM	SW-846 8270C	Nitrodiphenylamine (2-)
SCM	SW-846 8270C	Hexachlorophene
SCM	SW-846 8270C	Diphenylhydrazine (1,2-)
SCM	SW-846 8270C	Decane (n-)
SCM	SW-846 8270C	Octadecane (n-)
SCM	SW-846 8270C	Benzo(a)anthracene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8270C	Benzo(a)pyrene
SCM	SW-846 8270C	Benzo(b)fluoranthene
SCM	SW-846 8270C	Benzo(k)fluoranthene
SCM	SW-846 8270C	Dibenzo(a,h)anthracene
SCM	SW-846 8270C	Indeno(1,2,3-c,d)pyrene
SCM	SW-846 8270C	Benzal chloride
SCM	SW-846 8270C	Benzo(j)fluoranthene
SCM	SW-846 8270C	Benzotrichloride
SCM	SW-846 8270C	Benzyl chloride
SCM	SW-846 8270C	Chlorobenzilate
SCM	SW-846 8270C	Dibenz(a,h)acridine
SCM	SW-846 8270C	Dibenzo(a,h)pyrene
SCM	SW-846 8270C	Dibenzo(a,i)pyrene
SCM	SW-846 8270C	Dibenzo(c,g)carbazole (7H-)
SCM	SW-846 8270C	Pentachloroethane
SCM	SW-846 8270C	Tetrachlorobenzene (1,2,3,4-)
SCM	SW-846 8270C	Tetrachlorobenzene (1,2,3,5-)
SCM	SW-846 8270C	Benzyl alcohol
SCM	SW-846 8270C	Acetophenone
SCM	SW-846 8270C	Acetylaminofluorene (2-)
SCM	SW-846 8270C	Aminobiphenyl (4-)
SCM	SW-846 8270C	Aramite
SCM	SW-846 8270C	Chloronaphthalene (1-)
SCM	SW-846 8270C	Diallate (cis)
SCM	SW-846 8270C	Diallate (trans)
SCM	SW-846 8270C	Dibenzo(a,e)pyrene
SCM	SW-846 8270C	Dibenz(a,j)acridine
SCM	SW-846 8270C	Dichlorophenol (2,6-)
SCM	SW-846 8270C	Dimethoate
SCM	SW-846 8270C	Dimethylaminoazobenzene
SCM	SW-846 8270C	Dimethylbenz(a)anthracene (7,12-)
SCM	SW-846 8270C	Dimethyl benzidine (3,3-)
SCM	SW-846 8270C	Dinitrobenzene (1,3-)
SCM	SW-846 8270C	Dinoseb
SCM	SW-846 8270C	Disulfoton
SCM	SW-846 8270C	Famphur
SCM	SW-846 8270C	Hexachloropropene
SCM	SW-846 8270C	Isodrin
SCM	SW-846 8270C	Isosafrole (cis-)
SCM	SW-846 8270C	Isosafrole (trans-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8270C	Kepone
SCM	SW-846 8270C	Methanesulfonate (Ethyl-)
SCM	SW-846 8270C	Methanesulfonate (Methyl-)
SCM	SW-846 8270C	Methapyrilene
SCM	SW-846 8270C	Methylcholanthrene (3-)
SCM	SW-846 8270C	Napthoquinone (1,4-)
SCM	SW-846 8270C	Napththylamine (1-)
SCM	SW-846 8270C	Napththylamine (2-)
SCM	SW-846 8270C	N-Nitroso-di-n-butylamine
SCM	SW-846 8270C	N-Nitrosomorpholine
SCM	SW-846 8270C	N-Nitrosopiperidine
SCM	SW-846 8270C	Parathion
SCM	SW-846 8270C	Parathion methyl
SCM	SW-846 8270C	Pentachlorobenzene
SCM	SW-846 8270C	Pentachloronitrobenzene
SCM	SW-846 8270C	Phenacetin
SCM	SW-846 8270C	Phenylenediamine (1,4-)
SCM	SW-846 8270C	Phenylethylamine (alpha, alpha-Dimethyl)
SCM	SW-846 8270C	Phorate
SCM	SW-846 8270C	Phosphorothioate (O,O,O-triethyl)
SCM	SW-846 8270C	Phosphorothioate (O,O-diethyl-O-2-pyrazinyl) [Thionazin]
SCM	SW-846 8270C	Picoline (2-)
SCM	SW-846 8270C	Pronamide
SCM	SW-846 8270C	Quinoline -1-Oxide (4-Nitro)
SCM	SW-846 8270C	Safrole
SCM	SW-846 8270C	Sulfotepp
SCM	SW-846 8270C	Tetrachlorobenzene (1,2,4,5-)
SCM	SW-846 8270C	Tetrachlorophenol (2,3,4,6-)
SCM	SW-846 8270C	Toluidine (2-) (2-Methylaniline)
SCM	SW-846 8270C	Toluidine (5-nitro-2-)
SCM	SW-846 8270C	Trinitrobenzene (1,3,5-)
SCM	SW-846 8270C	N-Nitrosodiethylamine
SCM	SW-846 8270C	N-Nitrosopyrrolidine
SCM	SW-846 8270C	Diphenylamine
SCM	SW-846 8270C	Carbazole
SCM	SW-846 8270C	Dichlorobenzene (1,2-)
SCM	SW-846 8270C	Dichlorobenzene (1,3-)
SCM	SW-846 8270C	N-Nitrosodimethylamine
SCM	SW-846 8270C	N-Nitroso-di-n-propylamine



<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8270C	N-Nitrosomethylethylamine
SCM	SW-846 8270C	Benzidine
SCM	SW-846 8270C	Aniline
SCM	SW-846 8270C	Hexachloropropene
SCM	SW-846 8270C	Dibenzofuran
SCM	SW-846 8270C	Benzoic acid
SCM	SW-846 8270C	N-Nitrosodiphenylamine
SCM	SW-846 8270C	Dichlorobenzidine (3,3'-)
SCM	SW-846 8270C	Chloroaniline (4-)
SCM	SW-846 8270C	Nitroaniline (2-)
SCM	SW-846 8270C	Nitroaniline (3-)
SCM	SW-846 8270C	Nitroaniline (4-)
SCM	SW-846 8270C	Chloronaphthalene (2-)
SCM	SW-846 8270C	Hexachlorobenzene
SCM	SW-846 8270C	Hexachlorobutadiene (1,3-)
SCM	SW-846 8270C	Hexachlorocyclopentadiene
SCM	SW-846 8270C	Hexachloroethane
SCM	SW-846 8270C	Trichlorobenzene (1,2,4-)
SCM	SW-846 8270C	Bis (2-chloroethoxy) methane
SCM	SW-846 8270C	Bis (2-chloroethyl) ether
SCM	SW-846 8270C	Bis (2-chloroisopropyl) ether
SCM	SW-846 8270C	Chlorophenyl-phenyl ether (4-)
SCM	SW-846 8270C	Bromophenyl-phenyl ether (4-)
SCM	SW-846 8270C	Dinitrotoluene (2,4-)
SCM	SW-846 8270C	Dinitrotoluene (2,6-)
SCM	SW-846 8270C	Isophorone
SCM	SW-846 8270C	Nitrobenzene
SCM	SW-846 8270C	Butyl benzyl phthalate
SCM	SW-846 8270C	Bis (2-ethylhexyl) phthalate
SCM	SW-846 8270C	Diethyl phthalate
SCM	SW-846 8270C	Dimethyl phthalate
SCM	SW-846 8270C	Di-n-butyl phthalate
SCM	SW-846 8270C	Di-n-octyl phthalate
SCM	SW-846 8270C	Acenaphthene
SCM	SW-846 8270C	Anthracene
SCM	SW-846 8270C	Acenaphthylene
SCM	SW-846 8270C	Benzo(a)anthracene
SCM	SW-846 8270C	Benzo(a)pyrene
SCM	SW-846 8270C	Benzo(b)fluoranthene
SCM	SW-846 8270C	Benzo(g,h,i)perylene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8270C	Benzo(k)fluoranthene
SCM	SW-846 8270C	Chrysene
SCM	SW-846 8270C	Dibenzo(a,h)anthracene
SCM	SW-846 8270C	Fluoranthene
SCM	SW-846 8270C	Fluorene
SCM	SW-846 8270C	Indeno(1,2,3-c,d)pyrene
SCM	SW-846 8270C	Methylnaphthalene (2-)
SCM	SW-846 8270C	Naphthalene
SCM	SW-846 8270C	Phenanthrene
SCM	SW-846 8270C	Pyrene
SCM	SW-846 8270C	Methyl phenol (4-chloro-3-)
SCM	SW-846 8270C	Chlorophenol (2-)
SCM	SW-846 8270C	Dichlorophenol (2,4-)
SCM	SW-846 8270C	Dimethylphenol (2,4-)
SCM	SW-846 8270C	Dinitrophenol (2,4-)
SCM	SW-846 8270C	Dinitrophenol (2-methyl-4,6-)
SCM	SW-846 8270C	Methylphenol (2-)
SCM	SW-846 8270C	Methylphenol (4-)
SCM	SW-846 8270C	Nitrophenol (2-)
SCM	SW-846 8270C	Nitrophenol (4-)
SCM	SW-846 8270C	Pentachlorophenol
SCM	SW-846 8270C	Phenol
SCM	SW-846 8270C	Trichlorophenol (2,4,5-)
SCM	SW-846 8270C	Trichlorophenol (2,4,6-)
SCM	SW-846 8270C	Dichlorobenzene (1,4-)
SCM	SW-846 8270C	Pyridine
SCM	SW-846 8270D	Biphenyl (1,1'-)
SCM	SW-846 8270D	Benzaldehyde
SCM	SW-846 8270D	Caprolactam
SCM	SW-846 8270D	Atrazine
SCM	SW-846 8270D	Phenanthrene
SCM	SW-846 8270D	Pyrene
SCM	SW-846 8270D	Acenaphthene
SCM	SW-846 8270D	Acenaphthylene
SCM	SW-846 8270D	Anthracene
SCM	SW-846 8270D	Benzo(g,h,i)perylene
SCM	SW-846 8270D	Chrysene
SCM	SW-846 8270D	Methylnaphthalene (1-)
SCM	SW-846 8270D	Methylnaphthalene (2-)
SCM	SW-846 8270D	Naphthalene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8270D	Fluoranthene
SCM	SW-846 8270D	Fluorene
SCM	SW-846 8270D	Methylnaphthalene (1-)
SCM	SW-846 8270D	Nitrodiphenylamine (2-)
SCM	SW-846 8270D	Hexachlorophene
SCM	SW-846 8270D	Diphenylhydrazine (1,2-)
SCM	SW-846 8270D	Decane (n-)
SCM	SW-846 8270D	Octadecane (n-)
SCM	SW-846 8270D	Benzo(a)anthracene
SCM	SW-846 8270D	Benzo(a)pyrene
SCM	SW-846 8270D	Benzo(b)fluoranthene
SCM	SW-846 8270D	Benzo(k)fluoranthene
SCM	SW-846 8270D	Dibenzo(a,h)anthracene
SCM	SW-846 8270D	Indeno(1,2,3-c,d)pyrene
SCM	SW-846 8270D	Benzal chloride
SCM	SW-846 8270D	Benzo(j)fluoranthene
SCM	SW-846 8270D	Benzotrichloride
SCM	SW-846 8270D	Benzyl chloride
SCM	SW-846 8270D	Chlorobenzilate
SCM	SW-846 8270D	Dibenz(a,h)acridine
SCM	SW-846 8270D	Dibenzo(a,h)pyrene
SCM	SW-846 8270D	Dibenzo(a,i)pyrene
SCM	SW-846 8270D	Dibenzo(c,g)carbazole (7H-)
SCM	SW-846 8270D	Pentachloroethane
SCM	SW-846 8270D	Tetrachlorobenzene (1,2,3,4-)
SCM	SW-846 8270D	Tetrachlorobenzene (1,2,3,5-)
SCM	SW-846 8270D	Benzyl alcohol
SCM	SW-846 8270D	Acetophenone
SCM	SW-846 8270D	Acetylaminofluorene (2-)
SCM	SW-846 8270D	Aminobiphenyl (4-)
SCM	SW-846 8270D	Aramite
SCM	SW-846 8270D	Chloronaphthalene (1-)
SCM	SW-846 8270D	Diallate (cis)
SCM	SW-846 8270D	Diallate (trans)
SCM	SW-846 8270D	Dibenzo(a,e)pyrene
SCM	SW-846 8270D	Dibenz(a,j)acridine
SCM	SW-846 8270D	Dichlorophenol (2,6-)
SCM	SW-846 8270D	Dimethoate
SCM	SW-846 8270D	Dimethylaminoazobenzene
SCM	SW-846 8270D	Dimethylbenz(a)anthracene (7,12-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8270D	Dimethyl benzidine (3,3-)
SCM	SW-846 8270D	Dinitrobenzene (1,3-)
SCM	SW-846 8270D	Dinoseb
SCM	SW-846 8270D	Disulfoton
SCM	SW-846 8270D	Famphur
SCM	SW-846 8270D	Isodrin
SCM	SW-846 8270D	Isosafrole (cis-)
SCM	SW-846 8270D	Isosafrole (trans-)
SCM	SW-846 8270D	Kepone
SCM	SW-846 8270D	Methanesulfonate (Ethyl-)
SCM	SW-846 8270D	Methanesulfonate (Methyl-)
SCM	SW-846 8270D	Methapyrilene
SCM	SW-846 8270D	Methylcholanthrene (3-)
SCM	SW-846 8270D	Napthoquinone (1,4-)
SCM	SW-846 8270D	Napththylamine (1-)
SCM	SW-846 8270D	Napththylamine (2-)
SCM	SW-846 8270D	N-Nitroso-di-n-butylamine
SCM	SW-846 8270D	N-Nitrosomorpholine
SCM	SW-846 8270D	N-Nitrosopiperidine
SCM	SW-846 8270D	Parathion
SCM	SW-846 8270D	Parathion methyl
SCM	SW-846 8270D	Pentachlorobenzene
SCM	SW-846 8270D	Pentachloronitrobenzene
SCM	SW-846 8270D	Phenacetin
SCM	SW-846 8270D	Phenylenediamine (1,4-)
SCM	SW-846 8270D	Phenylethylamine (alpha, alpha-Dimethyl)
SCM	SW-846 8270D	Phorate
SCM	SW-846 8270D	Phosphorothioate (O,O,O-triethyl)
SCM	SW-846 8270D	Phosphorothioate (O,O-diethyl-O-2-pyrazinyl) [Thionazin]
SCM	SW-846 8270D	Picoline (2-)
SCM	SW-846 8270D	Pronamide
SCM	SW-846 8270D	Quinoline -1-Oxide (4-Nitro)
SCM	SW-846 8270D	Safrole
SCM	SW-846 8270D	Sulfotepp
SCM	SW-846 8270D	Tetrachlorobenzene (1,2,4,5-)
SCM	SW-846 8270D	Tetrachlorophenol (2,3,4,6-)
SCM	SW-846 8270D	Toluidine (2-) (2-Methylaniline)
SCM	SW-846 8270D	Toluidine (5-nitro-2-)
SCM	SW-846 8270D	Trinitrobenzene (1,3,5-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8270D	N-Nitrosodiethylamine
SCM	SW-846 8270D	N-Nitrosopyrrolidine
SCM	SW-846 8270D	Diphenylamine
SCM	SW-846 8270D	Carbazole
SCM	SW-846 8270D	Dichlorobenzene (1,2-)
SCM	SW-846 8270D	Dichlorobenzene (1,3-)
SCM	SW-846 8270D	N-Nitrosodimethylamine
SCM	SW-846 8270D	N-Nitroso-di-n-propylamine
SCM	SW-846 8270D	N-Nitrosomethylethylamine
SCM	SW-846 8270D	Benzidine
SCM	SW-846 8270D	Aniline
SCM	SW-846 8270D	Hexachloropropene
SCM	SW-846 8270D	Dibenzofuran
SCM	SW-846 8270D	Benzoic acid
SCM	SW-846 8270D	N-Nitrosodiphenylamine
SCM	SW-846 8270D	Dichlorobenzidine (3,3'-)
SCM	SW-846 8270D	Chloroaniline (4-)
SCM	SW-846 8270D	Nitroaniline (2-)
SCM	SW-846 8270D	Nitroaniline (3-)
SCM	SW-846 8270D	Nitroaniline (4-)
SCM	SW-846 8270D	Chloronaphthalene (2-)
SCM	SW-846 8270D	Hexachlorobenzene
SCM	SW-846 8270D	Hexachlorobutadiene (1,3-)
SCM	SW-846 8270D	Hexachlorocyclopentadiene
SCM	SW-846 8270D	Hexachloroethane
SCM	SW-846 8270D	Trichlorobenzene (1,2,4-)
SCM	SW-846 8270D	Bis (2-chloroethoxy) methane
SCM	SW-846 8270D	Bis (2-chloroethyl) ether
SCM	SW-846 8270D	Bis (2-chloroisopropyl) ether
SCM	SW-846 8270D	Chlorophenyl-phenyl ether (4-)
SCM	SW-846 8270D	Bromophenyl-phenyl ether (4-)
SCM	SW-846 8270D	Dinitrotoluene (2,4-)
SCM	SW-846 8270D	Dinitrotoluene (2,6-)
SCM	SW-846 8270D	Isophorone
SCM	SW-846 8270D	Nitrobenzene
SCM	SW-846 8270D	Butyl benzyl phthalate
SCM	SW-846 8270D	Bis (2-ethylhexyl) phthalate
SCM	SW-846 8270D	Diethyl phthalate
SCM	SW-846 8270D	Dimethyl phthalate
SCM	SW-846 8270D	Di-n-butyl phthalate

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8270D	Di-n-octyl phthalate
SCM	SW-846 8270D	Acenaphthene
SCM	SW-846 8270D	Anthracene
SCM	SW-846 8270D	Acenaphthylene
SCM	SW-846 8270D	Benzo(a)anthracene
SCM	SW-846 8270D	Benzo(a)pyrene
SCM	SW-846 8270D	Benzo(b)fluoranthene
SCM	SW-846 8270D	Benzo(g,h,i)perylene
SCM	SW-846 8270D	Benzo(k)fluoranthene
SCM	SW-846 8270D	Chrysene
SCM	SW-846 8270D	Dibenzo(a,h)anthracene
SCM	SW-846 8270D	Fluoranthene
SCM	SW-846 8270D	Fluorene
SCM	SW-846 8270D	Indeno(1,2,3-c,d)pyrene
SCM	SW-846 8270D	Methylnaphthalene (2-)
SCM	SW-846 8270D	Naphthalene
SCM	SW-846 8270D	Phenanthrene
SCM	SW-846 8270D	Pyrene
SCM	SW-846 8270D	Methyl phenol (4-chloro-3-)
SCM	SW-846 8270D	Chlorophenol (2-)
SCM	SW-846 8270D	Dichlorophenol (2,4-)
SCM	SW-846 8270D	Dimethylphenol (2,4-)
SCM	SW-846 8270D	Dinitrophenol (2,4-)
SCM	SW-846 8270D	Dinitrophenol (2-methyl-4,6-)
SCM	SW-846 8270D	Methylphenol (2-)
SCM	SW-846 8270D	Methylphenol (4-)
SCM	SW-846 8270D	Nitrophenol (2-)
SCM	SW-846 8270D	Nitrophenol (4-)
SCM	SW-846 8270D	Pentachlorophenol
SCM	SW-846 8270D	Phenol
SCM	SW-846 8270D	Trichlorophenol (2,4,5-)
SCM	SW-846 8270D	Trichlorophenol (2,4,6-)
SCM	SW-846 8270D	Dichlorobenzene (1,4-)
SCM	SW-846 8270D	Pyridine
SCM	SW-846 8310	Acenaphthene
SCM	SW-846 8310	Acenaphthylene
SCM	SW-846 8310	Anthracene
SCM	SW-846 8310	Benzo(a)anthracene
SCM	SW-846 8310	Benzo(a)pyrene
SCM	SW-846 8310	Benzo(b)fluoranthene

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8310	Benzo(g,h,i)perylene
SCM	SW-846 8310	Benzo(k)fluoranthene
SCM	SW-846 8310	Chrysene
SCM	SW-846 8310	Dibenzo(a,h)anthracene
SCM	SW-846 8310	Fluoranthene
SCM	SW-846 8310	Fluorene
SCM	SW-846 8310	Indeno(1,2,3-c,d)pyrene
SCM	SW-846 8310	Naphthalene
SCM	SW-846 8310	Phenanthrene
SCM	SW-846 8310	Pyrene
SCM	SW-846 8330	Nitroglycerine
SCM	SW-846 8330	Guanidine nitrate
SCM	SW-846 8330	PETN
SCM	SW-846 8330	HMX
SCM	SW-846 8330	RDX
SCM	SW-846 8330	Trinitrobenzene (1,3,5-)
SCM	SW-846 8330	Dinitrobenzene (1,3-)
SCM	SW-846 8330	Tetryl
SCM	SW-846 8330	Nitrobenzene
SCM	SW-846 8330	Trinitrotoluene (2,4,6-)
SCM	SW-846 8330	Dinitrotoluene (4-amino-2,6-)
SCM	SW-846 8330	Dinitrotoluene (2-amino-4,6-)
SCM	SW-846 8330	Dinitrotoluene (2,4-)
SCM	SW-846 8330	Dinitrotoluene (2,6-)
SCM	SW-846 8330	Nitrotoluene (2-)
SCM	SW-846 8330	Nitrotoluene (3-)
SCM	SW-846 8330	Nitrotoluene (4-)
SCM	SW-846 8330A	Nitroglycerine
SCM	SW-846 8330A	PETN
SCM	SW-846 8330A	HMX
SCM	SW-846 8330A	RDX
SCM	SW-846 8330A	Trinitrobenzene (1,3,5-)
SCM	SW-846 8330A	Dinitrobenzene (1,3-)
SCM	SW-846 8330A	Tetryl
SCM	SW-846 8330A	Nitrobenzene
SCM	SW-846 8330A	Trinitrotoluene (2,4,6-)
SCM	SW-846 8330A	Dinitrotoluene (4-amino-2,6-)
SCM	SW-846 8330A	Dinitrotoluene (2-amino-4,6-)
SCM	SW-846 8330A	Dinitrotoluene (2,4-)
SCM	SW-846 8330A	Dinitrotoluene (2,6-)

<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	SW-846 8330A	Nitrotoluene (2-)
SCM	SW-846 8330A	Nitrotoluene (3-)
SCM	SW-846 8330A	Nitrotoluene (4-)
SCM	SW-846 8440	Total rec. petroleum hydrocarbons
SCM	SW-846 9010C	Cyanide - amenable to Cl2
SCM	SW-846 9010C	Cyanide
SCM	SW-846 9012B	Cyanide
SCM	SW-846 9013	Cyanide
SCM	SW-846 9023	Extractable organic halides (EOX)
SCM	SW-846 9030B	Sulfides, acid sol. & insol.
SCM	SW-846 9034	Sulfides, acid sol. & insol.
SCM	SW-846 9040B	Corrosivity - pH waste, >20% water
SCM	SW-846 9040C	Corrosivity - pH waste, >20% water
SCM	SW-846 9045C	pH - soil and waste
SCM	SW-846 9045D	pH - soil and waste
SCM	SW-846 9056	Bromide
SCM	SW-846 9056	Nitrite
SCM	SW-846 9056	Sulfate
SCM	SW-846 9056	Nitrate
SCM	SW-846 9056	Chloride
SCM	SW-846 9056	Fluoride
SCM	SW-846 9056	Orthophosphate
SCM	SW-846 9056A	Bromide
SCM	SW-846 9056A	Nitrite
SCM	SW-846 9056A	Sulfate
SCM	SW-846 9056A	Nitrate
SCM	SW-846 9056A	Chloride
SCM	SW-846 9056A	Fluoride
SCM	SW-846 9056A	Orthophosphate
SCM	SW-846 9060	Total organic carbon (TOC)
SCM	SW-846 9060A	Total organic carbon (TOC)
SCM	SW-846 9071B	Oil & grease - sludge-hem-npm
SCM	SW-846 9071B	Oil & grease - sludge-hem
SCM	SW-846 9095	Free liquid
SCM	SW-846 9095B	Free liquid
SCM	User Defined 8260C	Hexane (n-)
SCM	User Defined 9010B	Cyanide - amenable to Cl2
SCM	User Defined 9010B	Cyanide
SCM	User Defined 9012A	Cyanide
SCM	User Defined 9013A	Cyanide



<u>Matrix</u>	<u>Method</u>	<u>Parameter</u>
SCM	User Defined 9095A	Free liquid
SCM	User Defined ASTM D93	Ignitability
SCM	User Defined CA LUFT - diesel	Petroleum Organics
SCM	User Defined CA LUFT - diesel	Petroleum Organics
SCM	User Defined LUFT	Xylene (m-)
SCM	User Defined LUFT	Xylene (o-)
SCM	User Defined LUFT	Xylene (p-)
SCM	User Defined LUFT	Benzene
SCM	User Defined LUFT	Ethylbenzene
SCM	User Defined LUFT	Toluene
SCM	User Defined LUFT	Xylenes (total)
SCM	User Defined LUFT	Methyl tert-butyl ether
SCM	User Defined MA-DEP-EPH, TN-EPH, WI DRO, NW TPH Dx	Diesel range organic
SCM	User Defined MA-DEP-VPH, WI GRO, NW TPH Gx	Gasoline range organic
SCM	User Defined NWTPH-Dx, NWTPH-Gx, NWTPHID	Petroleum Organics
SCM	User Defined SW846 8260B & 8260C	Gasoline range organic
SCM	User Defined SW-846 8330	Nitroguanidine
SCM	User Defined TX 1005, TX 1006, CT ETPH, NW TPH ID	Petroleum Organics

### 3.4 A BBREVIATIONS/ACRONYMS

The quality department is responsible for setting up and maintaining a list of abbreviations used in the quality manual.

<b>ABBREVIATION</b>	<b>DESCRIPTION</b>
<i>A2LA</i>	<i>AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION</i>
<i>AIHA</i>	<i>AMERICAN INDUSTRIAL HYGIENE ASSOCIATION</i>
<i>BLANK</i>	<i>See FIELD, TRIP, METHOD, EQUIPMENT, INSTRUMENT, REAGENT</i>
<i>CAL</i>	<i>CALIBRATION</i>
<i>CCB</i>	<i>CONTINUING CALIBRATION BLANK</i>
<i>CCV</i>	<i>CONTINUING CALIBRATION VERIFICATION</i>
<i>CDOC</i>	<i>CONTINUING DEMONSTRATION OF CAPABILITY</i>
<i>COC</i>	<i>CHAIN OF CUSTODY</i>
<i>CA</i>	<i>CORRECTIVE ACTION</i>
<i>CRM</i>	<i>CERTIFIED REFERENCE MATERIAL</i>
<i>DQO</i>	<i>DATA QUALITY OBJECTIVES</i>
<i>DUP</i>	<i>DUPLICATE</i>
<i>EB</i>	<i>EQUIPMENT BLANK</i>
<i>FB</i>	<i>FIELD BLANK</i>
<i>GC</i>	<i>GAS CHROMATOGRAPHY</i>
<i>GCMS</i>	<i>GAS CHROMATOGRAPHY MASS SPECTROMETRY</i>
<i>HPLC</i>	<i>HIGH PRESSURE LIQUID CHROMATOGRAPHY</i>
<i>IB</i>	<i>INSTRUMENT BLANK</i>
<i>IC</i>	<i>ION CHROMATOGRAPHY</i>
<i>ICP</i>	<i>INDUCTIVELY COUPLED PLASMA</i>
<i>ICPMS</i>	<i>INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY</i>
<i>ICS</i>	<i>INTERFERENCE CHECK SAMPLE</i>
<i>ICV – See SSCV</i>	<i>INITIAL CALIBRATION VERIFICATION</i>
<i>IDOC</i>	<i>INITIAL DEMONSTRATION OF CAPABILITY (SEE ALSO CDOC)</i>
<i>IDL</i>	<i>INSTRUMENT DETECTION LIMIT</i>
<i>ISTD</i>	<i>INTERNAL STANDARD</i>
<i>LCS</i>	<i>LABORATORY CONTROL SAMPLE (Typically 2<sup>ND</sup> Source)</i>
<i>LCSD</i>	<i>LABORATORY CONTROL SAMPLE DUPLICATE</i>
<i>LOD</i>	<i>LIMIT OF DETECTION</i>
<i>LOQ</i>	<i>LIMIT OF QUANTITATION</i>
<i>LDR</i>	<i>LINEAR DYNAMIC RANGE</i>
<i>MAT</i>	<i>MATRIX</i>
<i>MS</i>	<i>MATRIX SPIKE</i>
<i>MSD</i>	<i>MATRIX SPIKE DUPLICATE</i>
<i>MDL</i>	<i>METHOD DETECTION LIMIT</i>
<i>MB</i>	<i>METHOD BLANK</i>

<b>ABBREVIATION</b>	<b>DESCRIPTION</b>
<i>NC</i>	<i>NEGATIVE CONTROL</i>
<i>% Rec</i>	<i>PERCENT RECOVERY</i>
<i>PC</i>	<i>POSITIVE CONTROL</i>
<i>PDL</i>	<i>PRACTICAL DETECTION LIMIT</i>
<i>PQL</i>	<i>PRACTICAL QUANTITATION LIMIT also See Reporting Limit (RL)</i>
<i>PT</i>	<i>PROFICIENCY TEST SAMPLE</i>
<i>QUAL</i>	<i>QUALIFIER</i>
<i>QA</i>	<i>QUALITY ASSURANCE</i>
<i>QAM</i>	<i>QUALITY ASSURANCE MANUAL</i>
<i>QAO</i>	<i>QUALITY ASSURANCE OFFICER</i>
<i>QC</i>	<i>QUALITY CONTROL</i>
<i>RF</i>	<i>RESPONSE FACTOR</i>
<i>RB</i>	<i>REAGENT BLANK</i>
<i>RL</i>	<i>REPORTING LIMIT</i>
<i>RLV</i>	<i>REPORTING LIMIT VERIFICATION</i>
<i>RPD</i>	<i>RELATIVE PERCENT DIFFERENCE</i>
<i>RSD</i>	<i>RELATIVE STANDARD DEVIATION</i>
<i>SSCV</i>	<i>SECONDARY SOURCE CALIBRATION VERIFICATION</i>
<i>SOP</i>	<i>STANDARD OPERATING PROCEDURE</i>
<i>SRM</i>	<i>STANDARD REFERENCE MATERIAL</i>
<i>SURR</i>	<i>SURROGATE</i>
<i>SVOC</i>	<i>SEMI-VOLATILE ORGANIC COMPOUND</i>
<i>TNI</i>	<i>THE NELAC INSTITUTE</i>
<i>UV</i>	<i>ULTRAVIOLET</i>
<i>VOC</i>	<i>VOLATILE ORGANIC COMPOUND</i>

## **4.0**    *MANAGEMENT REQUIREMENTS*

### **4.1**    **O**    **RGANIZATION**

#### 4.1.1    Legal identity

The laboratory is authorized under Title 62 of the Tennessee Code Annotated and is identified as Environmental Science Corporation (d.b.a. ESC Lab Sciences) located at 12065 Lebanon Road, Mount Juliet, TN 37122

#### 4.1.2    Organization

The laboratory is a public entity and is structured to provide environmental support services in compliance with numerous federal, state, and local regulations as well as to meet the analytical needs of the client.

#### 4.1.3    Facilities Under Management System

The scope of the ESC management system is comprehensive and covers all technical and supporting work conducted at all facilities including the primary Lebanon Road location as well as customer support and shipping operations across the US.

#### 4.1.4    Independence

ESC Lab Sciences is an independent analytical facility and therefore remains uninfluenced by external factors, such as financial or political considerations.

#### 4.1.5    Management Responsibilities and Policies

The assignment of responsibilities, authorities, and interrelationships of the personnel who manage, perform, or verify work affecting analytical quality is documented in the job descriptions maintained by the Human Resources department. Management bears specific responsibility for maintenance of the Quality System. This includes defining roles and responsibilities of personnel, approving documents, providing required training, providing a procedure for confidential reporting of data and ensuring data integrity, along with periodically reviewing data, procedures, and documentation. Management ensures that audit findings and corrective actions are completed within required time frames. Alternates are appointed by management during the absence of the Laboratory Director, Compliance Director, or the Quality Manager. The organizational structure indicated in this section is designed to minimize the potential for conflicts or undue stresses that might influence the technical judgment of analytical personnel. Additionally, it provides adequate management for consistent supervision of laboratory practices, personnel and procedures.

Operations Management is responsible for defining the minimal level of education, qualifications, experience, and skills necessary for all analytical positions in the laboratory and assuring that technical staff has demonstrated capabilities in their tasks. Training is kept up-to-date by periodic review of training records and through employee performance reviews. A brief description of the operations management positions is given below.

#### 4.1.5.1 **Chief Executive Officer**

Peter Schulert, Bachelor of Science in Chemistry, is the laboratory's Chief Executive Officer (CEO). He joined ESC in 1987 after the completion of his service with the United States Naval Submarine Service. In his five years of nuclear submarine experience in the Navy, Mr. Schulert qualified as an officer. This qualification included supervision of nuclear reactors and power plant operations. His vision for automation and client services has been a key component of ESC's rise to the top ranks of the industry. Under his leadership, ESC has become a large single location laboratory, with a comprehensive national certification program and industry leading data management tools. In his absence, all responsibilities are delegated to the ESC President.

#### 4.1.5.2 **President**

John Mitchell, Bachelor of Science in Chemistry, is the laboratory's President. He joined ESC in 2014 after gaining over 25 years of experience in commercial laboratory operations and management. He has served as a National Program Director for Oil and Gas Programs for several years, assisting exploration and production industrial clients with the establishment and management of risk-based analytical programs to ensure compliance with regulatory requirements and to develop additional strategies to reduce long term environmental impact liability. He directed emergency response actions, leading the laboratory response for multiple large scale mobilizations across the country. Mr. Mitchell is responsible for developing and executing ESC's strategic plan. In his absence, all operational responsibilities are delegated to the Chief Executive Officer.

#### 4.1.5.3 **Laboratory Director**

Eric Johnson, B.S. in Chemistry, is the Laboratory Director and is responsible for the supervision of each laboratory division and the overall compliance of the laboratory to this Quality Manual. Mr. Johnson provides ESC with necessary experience for all aspects of sample handling from sample shipping and receiving through sample disposal. He has been involved in many aspects of environmental analyses since 1991. He coordinates all production areas and is responsible for operational scheduling, process specifications, and implementation of quality standards. He focuses his background and experience on the improvement of existing systems in order to maximize efficiency and improve quality. He reports directly to the

President. In his absence, all operations responsibilities are delegated to the Technical Director and then to individual department managers.

#### 4.1.5.4 **Technical Director**

Ken Buckley, B.S. with emphasis in physics/chemistry/biology, is the Technical Director and works closely with the Laboratory Director to insure the lab is meeting method and certification requirements as well as playing an integral role in method development and improvement. He has extensive experience performing analysis in support of the Safe Drinking Water Act, Clean Water Act, Resource Conservation and Recovery Act and numerous state and specialty programs. His experience includes bench experience in organic and inorganic chemistry analysis. He has held several supervisory and management positions which have prepared him for the role of technical oversight of laboratory operations. Part of his day to day activities involves working closely with the Project Management group to better serve client inquiries. In his absence, all responsibilities are delegated to the Laboratory Director.

#### 4.1.5.5 **Compliance Director**

Jim Brownfield, B.S. in Chemistry, is the Compliance Director. His primary responsibility is to ensure regulatory compliance of the laboratory. He is also responsible for managing the implementation, monitoring, and development of the laboratory's Quality Assurance Systems; maintaining the laboratory's Quality Assurance Manual; and ensuring all laboratory personnel are strictly adhering to the laboratory's ethics policy. He also performs other Quality Assurance activities including method validation, technical writing, and participation in internal and external assessments. In addition, he oversees the Regulatory Affairs Department. He has more than 15 years of experience in various supervisory and managerial roles in the environmental laboratory industry. Over the years he has gained an extensive and detailed understanding of regulatory and accreditation requirements of federal and various state accreditation agencies. He has successfully designed, developed, implemented, and maintained Quality Assurance Systems in multiple laboratories. In his absence, all responsibilities are delegated to the Quality Assurance Manager.

#### 4.1.5.6 **Quality Assurance Manager**

Steve Miller, B.S. in Microbiology, is the laboratory Quality Assurance Manager and is responsible for managing the implementation, monitoring, and development of the laboratory's Quality Assurance Systems. In this role, he also oversees safety, waste management, internal and external audits, and new method implementation. He has been involved in many aspects of the environmental industry since 1990. He has an in-depth knowledge of GC and GCMS methods and instrumentation having hands-on experience with MS, PID/FID, FID, and ECD detectors. He has years of experience validating all types of environmental data. He has served as technical support for many environmental site investigation and/or remediation projects,

primary author of several project-specific Quality Assurance Project Plans, support for RCRA-permitted activities at major oil refineries (including permit modification), and primary author of the first hazardous waste delisting petition approved by U.S. EPA Region 8. In his absence, all responsibilities are delegated to the Compliance Director.

#### **4.1.5.7 Director of Information Technology**

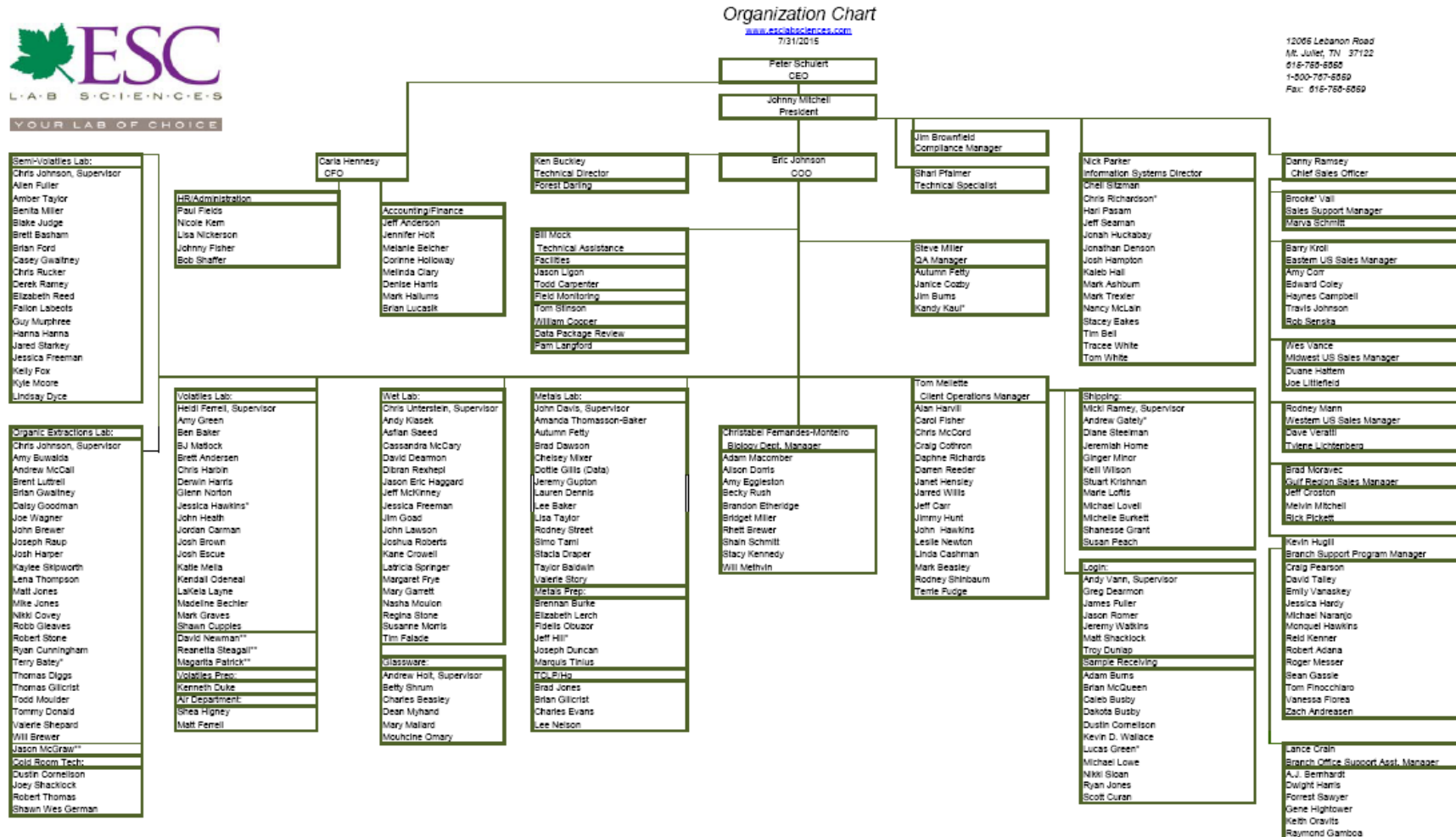
Nick Parker, B.S. in Plant and Soil Science, is the laboratory's Director of Information Technology. He has more than 15 years of laboratory experience in Organic analytical methods/instrumentation in the production laboratory environment and an expertise within information technologies and process automation. Mr. Parker is responsible for ESC's data management, information security, and software development while leading a team of developers, specialists, and Database Administrators. His unique understanding of laboratory operations and environmental methodology contributes to ESC's well managed software development and deployment within all laboratory and quality departments.

#### **4.1.6 Management System Effectiveness**

Senior management ensures that appropriate communication processes are established within the laboratory for implementation of the management system and that communication takes place regarding the effectiveness of the management system.

Figure 4.1 is the organizational chart which lists individuals and relevant departmental structure.

**Figure 4.1 Organizational Chart (Subject to change)**



12065 Lebanon Road  
 Mt. Juliet, TN 37122  
 615-760-6050  
 1-800-767-6550  
 Fax: 615-760-6050

Full Time: 261  
 Part Time: 7  
 Contract: 4  
 Summer help: 5  
 Total: 277



## **4.2 Management System**

### 4.2.1 Management Documentation

Management system documentation consists of different levels:

- Documented statements of the quality policy (issued under the authority of the Chief Executive Officer) and the quality objectives of this manual
- Documented procedures required by all applicable standards that detail the implementation of requirements and operation guidelines.
- Instructions: details of quality or inspection information and specific instructions for performance of individual tasks.
- Documents needed by the organization to ensure the effective planning, operation and management of its processes
- Records required by all applicable standards per the records procedure.

When the term “documented procedure” appears within this quality manual, the procedure is established, documented, implemented and maintained.

The laboratory maintains its documents in various formats including paper and various electronic formats.

### 4.2.2 Quality Management Policy

The management of ESC is committed to maintaining a quality assurance/quality control program that allows data generated by ESC, or any subcontractors under ESC's supervision, to meet both required and stated accuracy goals. The most important aspect of the program is to ensure that all activities whether involving sampling, analytical, or engineering activities, are congruent with EPA laboratory practices and regulatory guidelines. Issues relating to the quality program are reviewed during weekly operations meetings with upper management and in semi-annual management reviews. ESC personnel who have direct responsibility for overseeing the quality assurance program report to ESC's President.

ESC has a diverse accreditation/certification program, which requires continuous monitoring of changes and modifications within a variety of state and international organizations. The certification program represents greater than 48 separate state and national accreditations. ISO 17025 is maintained as the minimum foundation to meet each program requirement. This requires an extreme dedication to the overall quality system and analytical testing.

#### 4.2.3 Management System Implementation and Improvement

ESC management is committed to the development, implementation, and continual improvement of the laboratory's management system as well as compliance with all statutory and regulatory requirements. These commitments, along with the importance of meeting client requirements, are continually communicated to all levels of personnel within the laboratory.

#### 4.2.4 Commitment to Client and Regulatory Requirements

Data integrity is the result of the processes that work together to assure the production of data of known and documented quality.

The ESC Policy Manual requires a strict adherence to ethics and confidentiality. This policy covers all aspects of the laboratory function from client contact to sample analysis and analytical reporting, invoicing, and archive. Each staff member must maintain a professional attitude towards all colleagues, regulators, auditors, and laboratory clients while continuously striving to improve technical knowledge and professional competence.

ESC supports individual authority and provides the necessary resources for each staff member to carry out their duties. Each staff member is responsible for the identification of departures, from the quality system and/or established analytical procedures, within their area of concern, and for the initiation of actions to prevent or minimize such departures. In addition, ESC strives to ensure that its management and personnel are free from any undue internal and external commercial, financial, and other pressures and influences that may adversely affect the quality of their work.

All ESC personnel, including contract and temporary, are required to sign an "Attestation of Ethics and Confidentiality" at the time of employment and during annual refresher training. This document clearly identifies inappropriate and questionable behavior. Violations of this document result in serious consequences, including prosecution and termination, if necessary. The ESC Policy Manual addresses this subject in detail. See SOP# 010102, *Ethics, Data Integrity, and Confidentiality*.

##### 4.2.4.1 Quality Manual (QAM)

ESC has established and maintains a quality manual that:

- Defines the structure of the management system.
- Makes reference to the quality policy, the supporting procedures (also technical) and instructions.
- Defines the roles and responsibilities of technical and quality staff

The management system documentation is communicated to each laboratory staff member. All employees sign a document, kept in their personnel file, which states that they have read and understood the *Quality Assurance Manual*, including the quality policy. Refresher training is performed as needed to ensure that any changes to the *Quality Assurance Manual* are conveyed to all affected employees.

#### 4.2.4.2 Commitment to the QAM and Related Procedures

This *Quality Assurance Manual* outlines the procedures that have been developed to implement laboratory policies and to fulfill the laboratory's commitment to the client. These procedures are further defined and integrated into ESC's standard operating procedures. The policies are stated such that this manual serves as a QA handbook of responsibilities for all laboratory personnel. The manual is reviewed and approved under the authority of the highest level of laboratory management. Where the *Quality Assurance Manual* documents laboratory requirements, a separate SOP or policy is not required. This document is also used as a supplement for project planning, client reference, and personnel training.

#### 4.2.5 Procedure List

A list of the procedures, the instructions and the quality records, which are included in the management system, is maintained by the Quality Assurance/Regulatory Affairs Departments.

#### 4.2.6 Management Roles and Responsibilities

##### 4.2.6.1 Programs

The management of ESC is the main support of the quality program. Each manager is aware of the requirements of each external auditing agency and is responsible to ensure that their respective departments meet the requirements of each agency. ESC maintains full compliance and agreement with the following organizations/regulations: A2LA, ISO 17025, AIHA, EPA, GALP/GLP, TNI, and individual states who carry primacy concerning certification and regulation.

##### 4.2.6.2 ESC Policy Manual

ESC has policies and procedures, in the ESC Policy Manual, to insure that there is no employee involvement in any activities that would diminish confidence in their competence, impartiality, judgment or operational integrity.

All staff members employed by ESC are issued a Company Policy Manual that covers a wide array of topics and defines the expectations and policies of ESC. The Manual addresses both corporate and professional conduct, including confidentiality, professional ethics, and discipline. No deviations from the company policy are permitted without the approval of the CEO.

#### 4.2.7 Management of System Changes

Top management ensures that the integrity of the management system is maintained when changes to the management system are planned and implemented.

#### 4.2.8 Policy for Use and Control of Electronic Signatures

Electronic signatures must be controlled by the individual as electronic files. Electronic signature files must be stored in a secure password protected environment, and are not sent to or used by other individuals. Electronic signatures carry the same weight as handwritten signatures with regards to document approval.

### 4.3 D DOCUMENT MANAGEMENT

This section describes procedures for document management, which includes controlling, distributing, reviewing, and accepting modifications. The purpose of document management is to ensure that adequate instruction is readily available for laboratory employees and to preclude the use of invalid and/or obsolete documents.

The laboratory manages three types of documents: 1) controlled, 2) approved, and 3) obsolete.

*A CONTROLLED DOCUMENT is one that is uniquely identified, issued, tracked, and kept current as part of the quality system. Controlled documents may be internal documents or external documents.*

*APPROVED means reviewed, and either signed and dated, or acknowledged in writing or secure electronic means by the issuing authority(ies).*

*OBSOLETE DOCUMENTS are documents that have been superseded by more recent versions.*

#### 4.3.1 Required Documents

Documents required by the management system, as well as analytical records, are managed per the SOP #010103, *Document Control and Distribution*.

#### 4.3.2 Document Control

The documentation management procedure is established to define the means needed to:

- Approve documents for adequacy prior to issue
- Review, update and re-approve existing documents as necessary
- Ensure that changes and the current revision status of documents are identified
- Ensure that relevant versions of applicable documents are available at points of use
- Ensure that documents remain legible and readily identifiable
- Ensure that documents of external origin are identified and their distribution managed using the documentation master list
- Prevent the unintended use of obsolete documents and to apply suitable identification to them if they are retained for any purpose.

##### 4.3.2.1 Document Review and Approval

Documents are reviewed and approved for use by the individual department management and Compliance Director, or designee, prior to issue.

Documents are reviewed on a regular schedule, as deemed necessary to ensure their contents are suitable, that regulatory requirements are met, and that documents comply with the current quality systems requirements and accurately describe current operations.

Approved copies of documents are available at all locations where operations are essential to the effective functions of the laboratory.

##### 4.3.2.2 Document Distribution

Controlled internal documents are uniquely identified with:

- 1) date of issue
- 2) revision identification
- 3) page number
- 4) total number of pages or a mark to indicate the end of the document
- 5) the signatures of the issuing authority (i.e. management).

A master list of controlled internal documents is maintained that includes distribution, location, and revision dates. A master list of controlled external documents is also maintained that includes title, version or copyright date, and location. The controlled document list is maintained by the Quality Assurance Department and is continually updated. All invalid or obsolete documents are removed from circulation and clearly marked to prevent use. Obsolete documents

retained for legal use or historical knowledge preservation are appropriately marked and/or isolated and retained.

#### 4.3.3 Changes to Controlled Documents

##### 4.3.3.1 Review and Approval of Changes

Document changes are re-approved by the original approving authority.

##### 4.3.3.2 Identification of New or Altered Text

Where practicable, the altered text or new text in the draft is identified during the revision or review process to provide for easy identification of the modifications. Minor changes that occur in the interim of each major revision of the procedure are indicated in the ESC SOP/Minor Revision Form that is attached to the SOP. Historical changes are described in the SOP Attachment I, Revision History.

##### 4.3.3.3 Procedure for Document Revision

Document revision is controlled under SOP# 010103, *Document Control and Distribution*. Suggested revisions to electronic documents are presented to management for review and approval. Changes to electronic documents can only be made by the QA Department, or designee. The document management process allows for “minor revisions” or amendments to documents where changes are not sufficient to cause a full procedure change. Minor revisions may take the form of handwritten notes on an approved SOP Minor Revision form. Document changes are approved with signature by management. The modified document is then copied and distributed, and obsolete documents are removed. Minor revisions to documents are incorporated into the next full revision as soon as practicable.

##### 4.3.3.4 Changes in Electronic Documents

The QA Manual, SOPs, Safety Plan, and other controlled documents are maintained electronically on a protected directory. Access rights are restricted to Regulatory Affairs and Quality Assurance personnel and the IT Director. Electronic copies of current and previous versions of all controlled documents are maintained on the computer network system. They are stored with the same security settings as the current version; however previous versions of documents are access controlled to prevent employee use of outdated material. The documents are archived to tape storage with regular back up of the entire network system.

##### 4.3.3.5 Standard Operating Procedures

Standard Operating Procedures (SOPs) are written procedures that describe in detail how to accurately and consistently reproduce laboratory processes or

provide additional direction for laboratory personnel. Copies of all SOPs are accessible to all personnel. SOPs consist of three types:

- Technical SOPs, pertaining to a laboratory process which have specifically required details
- Administrative SOPs which document the more general organizational procedures.
- Quality SOPs that provide background and process for quality policy.

SOPs do not have to be formal documents with pre-defined section headings and contents. They can be less formal descriptions of procedures described in the *Quality Assurance Manual* or other documents.

#### 4.3.3.5.1 Format

Each SOP indicates the effective date, the revision number, and the signature(s) of the Regulatory Affairs Department and Department Supervisor. Department Supervisor approval is also required on technical procedures. Detailed information can be found in SOP# 010100, *Writing, Revising, and Maintaining Standard Operating Procedures*

All Standard Operating Procedures, QA Manuals, and Safety Plans are written in a format that incorporates the document name, date revised, pages included, and section.

Deviations from SOPs and Quality documents are not allowed without the permission of the Compliance Director, or designee. In the event that a deviation is requested, the circumstance is considered and the procedure is evaluated for necessary change and allowance.

#### **Determinative Method SOPs**

The laboratory has SOPs for all analytical methods within its scope, which is listed in Table 3.1. Where equipment manuals or published methods accurately reflect laboratory procedures in detail, a separate SOP is not required. Any deviation from a method is documented in the method modifications section of the respective SOP, including both a description of the change made and a technical justification. The deviation is reported to the client. Evidence of bias that is detected in an analytical result is reported to the client along with a defined qualifier that explains the bias. Each determinative method SOP includes or references (as applicable) the following:

- Scope and Application;
- Method Summary and Definitions;

- Health and Safety;
- Sample Preservation, Containers, Handling and Storage;
- Interferences;
- Equipment and Supplies;
- Reagents and Standards;
- Procedure;
- Data Analysis and Calculations;
- Quality Control and Method Performance;
- Data Validation and Corrective Action;
- Pollution Prevention and Waste Management;
- Method Modifications/Clarifications;
- References;
- Procedure Revision/Review History;

#### **4.4 R REVIEW OF REQUESTS, TENDERS, AND CONTRACTS**

##### 4.4.1 Procedure for Contract Review

When ESC enters into a contract to provide laboratory services, it follows SOP# 020303, *Contract Review*. Upon receipt of a request or invitation to tender a bid/proposal, the clients' requirements are examined by the contract review personnel to establish that the necessary details are adequately outlined and that the laboratory is able and willing to meet them.

##### 4.4.2 Records of Reviews

Records of reviews of requests, tenders and contracts (including significant changes) are maintained. Records are also maintained of pertinent discussions with the client relating to the client's requirements and the results of the work during the period of execution of the contract.

##### 4.4.3 Subcontracted Work

Clients' requirements for custom analyses and for work subcontracted to other laboratories are reviewed by the appropriate technical staff for logistics and feasibility.

##### 4.4.4 Deviations from the Contract

The client and the affected personnel are informed of any deviation from the contract.



#### 4.4.5 Contract Amendments

If a contract requires amendment after work has commenced, the same contract review process is repeated and any amendments are communicated to all affected parties.

### 4.5 S UBCONTRACTING

A subcontract laboratory is defined as a laboratory external to ESC, or at a different location than the address indicated on the front cover of this manual, that performs analyses for this laboratory.

#### 4.5.1 Subcontractor Competence

ESC only performs analytical techniques that are within its documented capability, when this is not possible, the laboratory follows SOP# 030209, *Subcontracting*. Subcontracting occurs in the special circumstances where technical, safety, or efficiency issues dictate need. When subcontracting analytical services, the laboratory assures work requiring specific accreditation is placed with an accredited laboratory or one that meets applicable statutory and regulatory requirements of the project/client.

#### 4.5.2 Client Notification

ESC notifies the client of the intent to subcontract the work in writing. The laboratory typically gains the approval of the client to subcontract their work prior to implementation, preferably in writing.

#### 4.5.3 ESC Responsibility

ESC assumes responsibility for the qualifications of the subcontractor (except when the client or an authority specifies a subcontractor) and the client is advised.

All reports, which contain data from subcontracted laboratories, include a statement on the final report, which references the subcontractor laboratory/service. As part of the initial subcontractor approval process, a copy of the applicable certificates and scopes for subcontractor's accreditation/certifications is maintained as evidence of compliance.

#### 4.5.4 Subcontractor List

ESC maintains a list of all approved subcontract laboratories.

## **4.6 P PURCHASING SERVICES AND SUPPLIES**

### 4.6.1 Purchasing Policies and Procedures

ESC maintains SOP# 030210, *Materials Procurement for Analytical Processes*, which describes the purchasing process, including vendor selection and acceptance criteria, for the purchase, storage, and evaluation of supplies and services. Where specifications of outside services and supplies are relevant to the measurement integrity of analyses, ESC uses services and supplies of adequate quality. The various department supervisors are responsible for ordering supplies/chemicals that meet the method and relevant regulatory stated requirements.

### 4.6.2 Quality of Purchased Items

Where assurance of the quality of outside support services or supplies is unavailable, the laboratory uses these items only after they have been inspected or otherwise verified for adequate quality. Records of inspections, verifications, and suppliers are maintained in the laboratory.

### 4.6.3 Purchasing Documents

Purchasing documents contain data clearly describing the product and/or services.

### 4.6.4 Approved Supplier List

An approved list of material/service suppliers is maintained where products/services purchased affect the quality of data generated by the laboratory.

## **4.7 S SERVICE TO THE CLIENT**

The ESC Technical Service Department provides specific project service through the use of Technical Service Representatives (TSRs). The TSR is responsible for all contract requirements and laboratory/client communication, including information concerning schedules, delays, and major deviations in the testing process.

### 4.7.1 Meeting Client Expectations

The TSR works closely with the client to clarify the client's requests and to monitor the laboratory's performance in relation to the work requested, while ensuring confidentiality to other clients. The laboratory confidentiality policy prohibits divulging or releasing any information to a third party without proper authorization. See SOP# 010102, *Ethics, Data Integrity, and Confidentiality*. All electronic data (storage or transmissions) are kept confidential, based on technology and laboratory limitations, as required by client or regulation. All electronic transmissions contain a confidentiality notice that represents the following:

*Notice: This communication and any attached files may contain privileged or other confidential information. If you have received this in error, please contact the sender immediately via reply email and immediately delete the message and any attachments without copying or disclosing the contents. Thank you.*

For additional information see SOP# 020301, *TSR (Project Management)*.

#### 4.7.2 Client Feedback

Service related feedback is obtained from clients by survey. This feedback is used to improve the management system, quality system, testing and calibration activities and client services. The feedback is discussed in management reviews.

#### 4.7.3 Client Access

When requested, ESC provides reasonable access, as needed by outside parties, to relevant areas of the lab for witnessing capability and analytical performance. Confidentiality of all clients during this process is maintained.

#### 4.7.4 Client Project Information

Clients may be provided supplementary documents, as needed, to further strengthen the project information. This may include: preparation documents, packaging information, verification of calibrations, and certification information.

#### 4.7.5 Communication with the Client

ESC's Technical Service Representatives maintain good communication with outside parties and are able to provide sound advice/guidance in technical matters and opinions/interpretations based on results. Communication with the client, especially in large assignments, is maintained throughout the work. The client is informed of any delays or deviations in the performance of the tests and/or calibrations.

### **4.8 C COMPLAINTS**

The purpose of this section is to ensure that customer complaints are addressed and corrected. This includes requests to verify results or analytical data. All client concerns are initially addressed by the Technical Service Representatives. If further resolution is required, the Compliance Director (or designee) and other pertinent personnel, as deemed necessary by the depth of the problem, conduct needed investigations and provide client support. See SOP# 020302, *Client Complaint Resolution*.

#### 4.8.1 Investigation of Complaints

In the event of a complaint, negative audit finding, or any other circumstance, which raises doubt concerning the laboratory's competence or compliance with required procedures, the laboratory ensures that those areas of activity are promptly investigated. A resolution of the situation is promptly sought and, where necessary, re-testing is conducted.

#### 4.8.2 Causes and Corrective Actions

The personnel in the Quality Assurance Department examine all documents and records associated with complaints and the relevant Department Supervisor investigates audit findings and other circumstances. This investigation seeks to identify specific root causes and initiate any necessary corrective action.

#### 4.8.3 Documentation

Records of events and the actions taken by the laboratory to resolve issues and to prevent future occurrences are maintained (see Section 4.11).

### **4.9 C CONTROL OF NON-CONFORMING WORK**

#### 4.9.1 Policies and Procedures

A nonconformance is an event that does not meet the requirements of the governing documents. Nonconformances can include unacceptable quality control results (See SOP# 030208, *Corrective Action*) or departures from standard operating procedures or test methods. Requests for departures from laboratory procedures are approved by the QA Department, or designee, and documented.

Types of non-conformances are:

- Deviations from written procedures that were not pre-approved by the QA Department.
- Changes to an existing SOP that is not included in the current revision
- A single and/or continuous trend of inappropriate habits
- A single and/or continuous trend of unexpected bias in the QC results
- Unusual changes in detection limit
- Deficiencies identified during an internal/external audit
- Unacceptable results on performance testing samples
- Valid issues reported by clients, data reviewers, or auditors
- General activities that demonstrate the possibility of a negative impact to the quality of the data

A policy has been established to ensure the use of analytical techniques that do not conform to specified requirements are prevented. This control provides for identification, documentation, evaluation, segregation (when practical) and disposition of nonconforming tests/calibrations. The control also calls for notification to the appropriate laboratory divisions. Any non-conforming tests/calibrations are reported to the supervisor of the affected laboratory division who is responsible for corrective actions. Records are documented on corrective action requests.

#### 4.9.2 Correcting Nonconforming Work

The corrective action system is used to identify nonconforming tests and/or calibrations. See SOP 030208, *Corrective and Preventive Action*.

#### 4.9.3 Review and Disposition of Nonconforming Tests/Calibrations

Since the laboratory has adopted a continuous improvement philosophy, it has established a procedure for reviewing and disposing of nonconforming tests/calibrations. This procedure includes:

- Reworking the test/calibration to meet the requirements
- Rejecting the test/calibration
- Informing the client (if necessary)

#### 4.9.4 Release of Nonconforming Work

The laboratory allows the release of nonconforming data only with approval on a case-by-case basis by the Department Supervisor, or their designee. Planned departures from procedures or policies do not require audits or investigations. Permitted departures for nonconformances, such as QC failures, are fully documented and include the reason for the deviation and the impact of the departure on the data. Any bias indicated in nonconforming work is indicated by the presence of data qualifiers that alert the client to the possible bias.

### **4.10 IMPROVEMENT**

The laboratory continually improves the effectiveness of its management system through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive actions and management review.

### **4.11 CORRECTIVE ACTIONS**

ESC strives for the continual improvement of its organization and its services. Corrective action is the process used to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

ESC recognizes that the data supplied by the professional staff must be legally and technically defensible. The QA Department personnel continually monitor the quality assurance program to ensure that this goal is achieved. Each analyst is responsible for initiating corrective actions in their areas of expertise. The Compliance Director, or designee, administers corrective action approval. It is the Supervisor's responsibility to evaluate the Corrective Action, appoint the appropriate person within the department to be responsible for completion of the CAR and submit it to the Regulatory Affairs Department for processing.

#### 4.11.1 General

The initiation, management, tracking, and closure of corrective actions are described in SOP# 030208, *Corrective and Preventive Action*.

#### 4.11.2 Investigation of Corrective Actions

Each lab division is encouraged to take any corrective action to determine and eliminate the causes of actual nonconformances to the degree appropriate to the magnitude of problems and commensurate with the risks encountered.

#### 4.11.3 Selection and Implementation of Corrective Actions

In addition to SOP# 030208, *Corrective and Preventive Action*, more specific guidance can be found in each determinative method.

In general, the corrective action procedure includes:

- The effective handling of client complaints and reports of nonconformities
- Investigation of the root cause of nonconformities relating to process, service, and management systems, and recording of results
- Determination of the corrective action needed to eliminate the cause of nonconformities
- Application of controls to ensure that corrective action is taken and that it is effective.

#### 4.11.4 Monitoring of Corrective Actions

The closure and follow-up activities of corrective actions are approved and documented in ESC's tracking system to ensure that the actions have been effective in addressing and correcting the problem.

#### 4.11.5 Additional Audits

When the identification of nonconformances or the corrective action investigation casts doubt on compliance with policies and procedures or the management system, laboratory management ensures that appropriate areas of activity are

audited in accordance with Section 4.14.1. The results of corrective action are submitted for laboratory management review.

#### 4.11.6 Cessation and Restarting of Work

All laboratory personnel are capable of invoking a “stop work” order, in the event that a situation impacts data validity or safety. It is the responsibility of the following personnel to (1) evaluate a “stop work” order whenever a severe non-conformance warrants a cessation of analysis and (2) ensure that the cause of the stop work order has been satisfactorily resolved and approve the restarting of work:

- ESC President
- Laboratory Director
- Compliance Director
- Regulatory Affairs Department
- Quality Assurance Department
- Department Supervisor/Senior Analysts
- Technical Service Representative

Department Supervisors/Senior Analysts review corrective action reports and suggest improvements, alternative approaches, and amended/revised procedures, where needed. If the data reported are affected adversely by the nonconformance, the client is notified in writing. The discovery of a nonconformance for results that have already been reported to the client must be immediately evaluated for significance of the issue, its acceptability to the client, and determination of the appropriate corrective action.

#### 4.11.7 Other Sources that may Initiate Corrective Action

Deficiencies cited in external assessments, internal quality audits, data reviews, complaints, or managerial reviews are documented and require corrective action. Corrective actions taken are appropriate for the magnitude of the problem and the degree of risk.

Appendix II lists the current federal and state agencies that perform audits of ESC. This table also lists the required performance evaluations that may initiate corrective actions. ESC implements any reasonable corrective action deemed necessary by the regulatory Certification Officers. In addition, the following types of samples may also initiate corrective action: split samples sent to another qualified laboratory, monthly blind field duplicates, and client submitted QC samples.

#### 4.11.8 Corrective Action Documents

In general, corrective action documents are maintained by the Quality Assurance/Regulatory Affairs Departments. These documents include the following: corrective action resulting from both internal and external audits,

corrective action resulting from performance evaluation testing, corrective action resulting from discrepancies found in compliance data review, and any other corrective action as deemed necessary by the Quality Assurance/Regulatory Affairs Departments.

Corrective action resulting from analytical failure is kept with the analytical data and is recorded on the bench sheet or raw data. The Department Supervisor is responsible for making sure that suitable measures have been taken to ensure that the problem is identified and corrected and that corrective actions are incorporated into daily activities as needed to prevent/reduce further occurrences.

Corrective action involving sample receiving is recorded on a Nonconformance form and is then filed with the original Chain of Custody.

## **4.12 P REVENTIVE ACTIONS**

Preventive Action, rather than corrective action, aims at minimizing or eliminating issues that could lead to inferior data quality or other nonconformances through scheduled maintenance and review, before the actual nonconformance occurs.

### 4.12.1 Management of Preventive Actions

ESC Management encourages preventive action measures. Each staff member is empowered to make suggestions for improving or fool-proofing processes throughout ESC. Where process areas show potential for nonconformance, measures are taken to identify the problem and formulate a plan to implement the defined change needed. The Compliance Director, or designee, reviews any recommended changes before implementation to ensure the effectiveness of the modification.

4.12.2 SOP# 030208, *Corrective and Preventive Action*, is also employed for preventive actions.

In general, the procedure for preventive action includes:

- The use of appropriate sources of information, such as processes and work operations, which affect product or service quality, concessions, audit results, quality records, service reports, and client complaints to detect, analyze, and eliminate potential causes of non-conformities.
- Determination of the steps needed to deal with any problems requiring preventive action
- Initiation of preventive action and application of controls to ensure that it is effective.



Preventive action includes, but is not limited to, review of QC data to identify quality trends, regularly scheduled staff quality meetings, annual budget reviews, semi-annual managerial reviews, scheduled instrument maintenance, and other actions taken to prevent potential problems.

#### 4.12.3 Trend Analysis

A trend analysis is an investigation that involves the collection of data in a manner that reveals deviations over time. Examples of laboratory processes that can be analyzed for trend analysis are:

- Sample receipt or chain of custody discrepancies
- Sample storage or preservation errors
- Holding time violations
- Instrument calibration
- Control Charts – Charts that are generated from historical data that plot percent recovery vs. time
- Method QC failures and problems

### 4.13 CONTROL OF RECORDS

Records are usually data recordings that include annotations, such as daily refrigerator temperatures, posted to laboratory forms, lists, spreadsheets, or analyst notes on a chromatogram. Records may be on any form of media, including electronic and hardcopy. Records allow for the historical reconstruction of laboratory activities related to sample handling and analysis.

#### 4.13.1 General

Technical and quality assurance records are established and maintained to provide evidence of conformity to requirements and of the effective operation of the quality system. Mechanisms are established for records to remain legible, readily identifiable and retrievable. The laboratory maintains a record system appropriate to its needs, records all laboratory activities, and complies with applicable standards or regulations as required.

The laboratory has defined the length of time various records, pertaining to the management system and examination results, are to be retained. Retention time is defined by the nature of examination or specifically for each record. The laboratory retains all original observations, calculations and derived data, calibration records, chain of custody and a copy of the test report for a minimum of ten years, unless otherwise required by regulatory authority.

Documented records procedures SOP# 010103, *Document Control and Distribution Procedure*, and SOP# 020304, *Protection and Transfer of Records*, are established to define the means needed for the identification, storage, protection, retrieval, retention time, transfer, and/or disposition of records.

#### 4.13.2 Technical and Quality Records

**NOTE: ALL records/data are stored for a minimum of 10 years, unless otherwise noted.**

All hardcopy department logbooks, such as temperature, maintenance, and preparation logs are placed into storage boxes and archived via a unique numbering system, to the ESC storage facility. Additional information regarding reagents/standards can be found in the Standards Logger (Tree) digital archive system. This digital system is backed up according to the ESC IT backup procedure.

Archived information and access logs are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.

<b>Data Storage Criteria</b>	
<b>Data Type</b>	<b>Storage Criteria</b>
<b>Manual Data Wet Chemistry</b>	All manually generated data are stored in specific laboratory analysis workbooks. Each individual analysis is located in a separate notebook which contains all data relating to the test including, calibration curves/data, QC charts/limits, SOP, and completed analysis sheets. These notebooks are centrally located and contain completed data that is filed by analysis and date analyzed. Monthly – Data is removed from the notebook and placed in a dedicated filing cabinet. Semi-annually – Data is removed from the filing cabinet, placed in storage boxes and archived, via a unique numbering system, in the ESC storage facility
<b>Manual Data Prep Labs</b>	All logbooks utilized in manually recording sample preparation information are placed into storage boxes and archived, via a unique numbering system, in the ESC storage facility. This includes organic prep, metals prep, and TCLP.
<b>Manual Data Env. Micro, Mold</b>	All manually generated data is stored in specific laboratory files and notebooks. These files are centrally located and contain completed data that is filed by analysis and date analyzed. Data is placed into storage boxes and (when full) archived, via a unique numbering system, in the ESC storage facility.
<b>All Data Aquatic Toxicity</b>	All manually generated data is stored in specific laboratory files and notebooks. These files are centrally located and contain completed data that is filed by analysis and date analyzed. Data is placed into storage boxes and (when full) archived, via a unique numbering system, in the ESC storage facility. Final reports and Reference Toxicant results are also scanned into ESC's electronic document management system. The data storage device on which this data resides is backed up daily. Data files are archived on to magnetic tape and retained per laboratory policy.
<b>Computerized Data - Organic Dept.</b>	Injection logs are printed to PDF file and maintained with the data. The instrument data is printed to a secure server and remains in a format that cannot be changed after printed. Upon printing, the data in the original file is generated. This storage system is backed up nightly utilizing a seven-day rotation cycle. The data is immediately available for up to two years. After two years, raw instrument data files are archived onto a separate secure server and kept a minimum of ten years. Original raw data files cannot be edited.

<b>Data Storage Criteria</b>	
<b>Data Type</b>	<b>Storage Criteria</b>
<b>Computerized Data – Inorganic Metals Dept.</b>	All data produced by metals instrumentation is backed up to a secure drive, nightly, utilizing a seven-day rotation cycle. All data is archived on a network attached storage device and is immediately available for up to two years. After two years, raw instrument data files are archived on to a separate secure server and kept a minimum of ten years. Original raw data files cannot be edited.
<b>Final Report Storage - LIMS</b>	The LIMS facilitates access to any finished data and sample information by client code, sample number, and parameter run number. Furthermore, any data pertaining to a sample or client can be obtained. The LIMS also contains the information from the COC such as sample description, time and date collected, sampler ID, container type, preservative, sample receipt data, finished/approved analytical data, analyst, etc. The LIMS Oracle Database is backed up daily on tape. The back up tape is kept in secure storage. While all LIMS data are accessible, data older than six months is moved from the active production database and is available in an archive database.
<b>Final Report Storage - PDF</b>	Copies of all reports are stored according to client code in PDF format on a network attached storage device and are immediately available for up to ten years. After ten years data files are archived onto magnetic tape and kept an additional ten years. These reports include chain of custody forms, login confirmation reports, the final approved printed report, invoices and any other associated documents. Samples that require subcontract work also have a copy of the final report in the client file.
<b>Misc. Data Storage</b>	Company records that are not stored on a secure electronic device are placed in storage boxes and archived, via a unique numbering system, in the ESC storage facility. This includes quality records, such as audits, state certifications, PT results, internal audits, corrective actions, training files, logbooks, etc.

#### 4.13.3 Records Disposal

Records that have exceeded the required storage requirement are disposed of through the use of professional records destruction firm or as required by regulatory or client requirements. ESC retains the manifest of documents destroyed and files the verification receipt that is generated at the time of destruction. Additional guidance for records disposal is provided in the ESC SOP#020304, *Protection and Transfer of Laboratory Records*.

#### 4.13.4 Records Transfer

In the event that corporate ownership is transferred or that laboratory activities are terminated for any reason, all records become property of the transferee in accordance with ESC SOP# 020304, *Protection and Transfer of Laboratory Records*.

#### 4.13.5 Legal Chain of Custody Records

Evidentiary Sample Data are used as legal evidence. Procedures for evidentiary samples are documented in section 5.8.7.

## 4.14 A UDITS

### 4.14.1 Internal Audits

SOP# 010104, *Internal Audits*, addresses the implementation and maintenance procedure for a comprehensive system of annual internal audits to verify the on-going effectiveness of the management system.

4.14.1.1 The Quality Assurance Department is responsible for administering the internal audit system per the documented procedures. The department develops a schedule for internal audits according to management system requirements and conducts unscheduled audits (internal and external) when reasons for such audits exist.

4.14.1.2 Audits may be conducted utilizing documented checklists and/or audit plans. Audit results are documented in audit reports per established procedures. Copies of all audit reports including completed corrective action requests are forwarded to management of the audited area and maintained by the Quality Assurance department.

4.14.1.3 Audit plans are structured according to the following:

***State/Certifying Agencies*** - Internal audits are conducted according to the various requirements set forth by the state and international agencies that accredit/certify/approve ESC. In addition, work procured from non-certifying states, also determine other requirements set forth by the state of origin. The audits are conducted to maintain compliance with the following Quality Standards: AIHA LQAP, A2LA, ANSI/IEC/ISO 17025, TNI, and DOD QSM.

***Method Specific Criteria*** – Good Laboratory technique, technical compliance with analytical methods and standard operating procedures, and effectiveness are reviewed during the internal audit. ESC maintains compliance with methods as listed in section 2.1.3.

***Data Integrity and Analyst Ethics*** - In addition to established standard and method related criteria; the internal audit is designed to review the analytical data for integrity and defensibility. Any suspicion of ethical violations results in a confidential investigation involving only the Compliance Director, or designee, and any specialist personnel necessary to conduct a complete and thorough investigation. Investigations, of this type, are conducted in a timely manner and all details and supporting documentation are recorded and maintained for a period of at least 10 years. All investigations that result in findings of inappropriate activity are

documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications to clients.

**Support Systems** – The internal audit process is also designed to assess support systems that are not a direct part of analytical activities. This includes, but is not limited to, the following:

- Contract Review
- Procurement and Vendor Approval
- Inventory Control
- Document Control
- Subcontracting
- Environmental, Safety, Security, and Health (ESSH)

4.14.1.4 Audit personnel are qualified per documented procedures and do not have direct responsibility for or control over the area being audited.

4.14.1.5 Management personnel responsible for the audited area determine and implement timely corrective actions for any reported nonconformance. Follow-up audit activities include verification of the corrective actions taken and reporting of the results. Clients are notified promptly when audit findings cast doubt on the validity of the data.

#### 4.14.2 Performance Audits

Performance audits are conducted periodically. Examples of performance audits include Proficiency Test (PT) sample analysis, internal single-blind sample analysis, and the analysis of double-blind samples that are submitted through a provider or a client. Anything that tests the performance of the analyst and/or the method is considered to be a performance audit. Additional information for Proficiency Test (PT) samples are discussed in the next section below.

#### 4.14.3 Proficiency Testing

The laboratory participates in various proficiency testing samples (PT) as required by each accreditation, and obtains test samples from approved providers. Corrective action procedures are initiated for all failed PT samples. All studies are conducted independently and no attempts are made to compare or obtain results from other labs or the provider. Proficiency Testing (PT) or Proficiency Evaluation (PE) samples are treated as typical samples in the normal production process where possible, including the same preparation, calibration, quality control and acceptance criteria, sequence of analytical steps, number of replicates, and sample log-in. PT samples are not analyzed multiple times unless routine environmental samples are analyzed multiple times.

- **PT Studies**

<b><i>Study</i></b>	<b><i>Frequency</i></b>	<b><i>Vendor</i></b>
WP (Water Pollution)	Semi-annually	Phenova
WS (Water Supply)	Semi-annually	Phenova
Matrix – Soil RCRA	Semi-annually	Phenova
Matrix – UST Soil/Water	Semi-annually	Phenova
Matrix – Air Canisters	Semi-annually	Phenova
DMRQA – Chemistry	Annually	Phenova
DMRQA – Aquatic Tox.	Annually	Phenova
ELLAP	Quarterly	AIHA
IHLAP	Quarterly	AIHA
EMLAP – Direct Exam	Quarterly	AIHA
EMLAP – Fungal / Bacterial	Triannually	AIHA
Cryptosporidium / Giardia	Quarterly	WSLH

- **Blind Field Duplicates** – ESC collects blind duplicates periodically to evaluate field collection and laboratory precision. ESC routinely receives unmarked field duplicates from clients to evaluate sample batches.
- **Split Samples** – ESC periodically participates in split samples with outside laboratories to confirm analytical results. This is performed on a project specific basis.

#### 4.14.4 External Audits

It is the laboratory’s policy to cooperate and assist with all external audits, whether performed by clients or an accrediting authority. All external audits are fully documented and tracked to closure.

Management ensures that all areas of the laboratory are accessible to auditors as applicable and that appropriate personnel are available to assist in conducting the audit. Any findings related to an external audit follow corrective action procedures. Management ensures that corrective actions are carried out within the timeframe specified by the auditor(s).

ESC is subject to several external audits on an annual basis. The audits cover all disciplines, SDWA, CWA, CAA and RCRA/UST.

#### **SDWA**

The ESC laboratory (EPA No. TN00003) is certified by the State of Tennessee under the Safe Drinking Water Act. The State of Tennessee routinely audits the ESC laboratory procedures, quality control and methods and has found the laboratory practices to be consistent with EPA requirements. ESC is also audited under the Safe Drinking Water Act by Arizona, Iowa, North Carolina, New Jersey

- NELAP, and the A2LA. ESC maintains several other DW certifications, which have been granted in reciprocity.

#### **CWA/RCRA**

ESC is certified for wastewater and solid waste through audits by the following states/organizations: A2LA, Arizona, Iowa, Minnesota, New Jersey (NELAP), North Carolina, Ohio VAP, West Virginia, Wisconsin, and USACE. ESC maintains several other certifications, which have been granted in reciprocity.

#### **INDUSTRIAL HYGIENE**

The American Industrial Hygiene Association routinely audits ESC to maintain accreditation for analytical support of microbiological testing and limited inorganic exposure activities. ESC maintains the quality systems to satisfy the requirements necessary for accreditation in the following: Environmental Lead (air, soil, paint and wipes), Industrial Hygiene (multi-element metallic air filters and dust analysis), and Environmental Microbiology (fungal/bacterial testing and identification)

#### **CLIENT AUDITS**

Due to participation in a number of national contracts, ESC is audited by several clients; who are also ISO certified and are required to assess their suppliers.

### **4.15 M ANAGEMENT REVIEW**

#### **4.15.1 Items in Management Review**

Regular management review meetings take place semi-annually and cover the events from the preceding time period. The Compliance Director, Laboratory Director, and all Department Supervisors are responsible for attending each meeting. Guidance, including agenda items, is given in ESC SOP# 010105, *Management Review*.

#### **4.15.2 Records of Management Review**

The QA Department collects objective evidence on the effectiveness of the management system. This includes audit results, client feedback, contract performance data, nonconformance data, problem reports, changes affecting the management system and previous management review reports.

#### 4.15.3 Evaluation

On the basis of this input, the management system is tested for its effectiveness, for its relevance, and for its implementation. In particular, quality objectives and the objectives set within the management system are examined. Adjustments are considered due to changes in the conduct or scope of business.

#### 4.15.4 Improvement

Decisions are made regarding actions needed to improve the effectiveness of the quality management system.

#### 4.15.5 Procedure

Details of this review, how it is be performed and recorded and the associated responsibilities can be found in the procedure for ESC SOP# 010105, *Management Review*.



## **5.0 TECHNICAL REQUIREMENTS**

### **5.1 GENERAL**

- 5.1.1 ESC recognizes that many factors determine the correctness and reliability of the analyses performed by a laboratory. These factors include contributions from: human factors (5.2), accommodations and environmental conditions (5.3), analytical/calibration methods and method validation (5.4), equipment (5.5), measurement traceability (5.6), and sample management - handling of test/calibration items (5.8).
- 5.1.2 The extent to which the factors contribute to the total uncertainty of measurement differs considerably between types of analyses. ESC takes into account these factors in developing analytical procedures, in the training and qualifications of personnel, and in the selection and calibration of the equipment utilized.

### **5.2 PERSONNEL**

#### 5.2.1 General Personnel Management

ESC management ensures the competency of all who operate specific equipment, who perform analyses, and who evaluate results and approve data reports. Personnel performing specific tasks are qualified on the basis of appropriate education, training, experience, and/or demonstrated skills, as required.

#### 5.2.2 Training

All training and education requirements are outlined in SOP# 030205, *Technical Training and Personnel Qualifications*. Training requirements for safety and health are listed in the *Chemical Hygiene Plan*. When staff members undergo training, adequate and appropriate supervision by fully trained analysts is provided.

##### 5.2.2.1 Corporate Documents

All employees are required to read relevant corporate documents. At a minimum this includes:

- ESC Policy Manual
- ESC QA Manual
- Chemical Hygiene Plan
- SOPs (As specified/required for work area)

Records of verification are required for each individual and are retained on file for a minimum of 10 years.

#### 5.2.2.2 Specific Documents

Analysts are also required to undergo training specific to their position. This includes the following:

- Documented review & acknowledgement of Method Specific SOPs
- Documented review & acknowledgement of published methods related to the specific SOP
- Documented review & acknowledgement of other supporting methods related to the specific determinative SOP
- Certification Statement of acceptable performance of an Initial Demonstration of Capability (according to method criteria)
- Continuous acceptable performance on daily/batch control samples
- Performance Testing, where required, may be reviewed as continued verification of analyst proficiency.
- Educational/training courses are provided where required by the position.
- Certification Statement of acceptable performance of a Continuing Demonstration of Capability (according to method criteria)

Records of verification are required for each individual and are retained on file for a minimum of 10 years.

#### 5.2.2.3 Routine Training

Any routine training and re-training necessary for a person to perform a particular job effectively is specified in job descriptions, process procedures, maintenance procedures, etc., as appropriate.

#### 5.2.2.4 Special Training

Special training required as a result of new technologies, contracts, expanding markets, company-wide improvement programs, new method development, etc. is conducted as the need arises.

#### 5.2.2.5 Annual Training

An annual training plan is established by management and in conjunction with regulatory requirements. Managers ensure that the plan is implemented within their areas of responsibility. Further information is outlined in SOP# 030205, *Technical Training and Personnel Qualifications*.

### 5.2.3 General Responsibilities

See Organization Chart in Section 4.0 for more detailed information regarding company organizational structure.

#### **Chemist/Analyst:**

- Performs sample analyses
- Verifies detail and accuracy by performing primary analytical review
- Records pertinent information in laboratory notebooks
- Stores all data (files and discs)
- Updates QC charts – where applicable
- Prepares and completes benchsheets/raw data for review
- Perform secondary review and data approval of other analysts data packages

#### **Laboratory Director:**

The Laboratory Director is responsible for all operational laboratory activities. The Laboratory Director must approve the *Quality Manual* prior to implementation.

#### **Technical Director:**

The Technical Director is responsible for ensuring that analytical processes meet the needs and requirements of approved and accredited methods.

#### **Laboratory Group Leader, Department Supervisor:**

Day to day supervision of technical laboratory departments is the responsibility of these leaders who are full-time members of the staff and who assure reliable data through the following activities: monitoring quality control and corroborating the analysis performed. Additionally they certify that personnel, within their areas, possess appropriate educational and/or technical background to perform all analyses for which the laboratory is accredited. The laboratory group leader or supervisor oversees analytical raw data, ensures calculation/calibration correctness, and reviews instrument and sample preparation logs.

#### **Compliance Director**

The Compliance Director is responsible for ensuring that the laboratory is compliant with all regulations and requirements. The Compliance Director is also responsible for managing the implementation, monitoring, and development of the laboratory's Quality Assurance Systems; maintaining the laboratory's Quality Assurance Manual; ensuring all laboratory personnel are strictly adhering to the laboratory's ethics policy; and overseeing the Regulatory Affairs Department. The

Compliance Director has direct access to the laboratory's President and is independent of operations.

#### **Quality Assurance Manager**

The Quality Assurance Manager is responsible for managing the implementation, monitoring, and development of the laboratory's Quality Assurance Systems. The Quality Assurance Manager also oversees safety, waste management, internal and external audits, and new method implementation. The Quality Assurance Manager has direct access to the laboratory's President and is independent of laboratory operations.

#### **Quality Assurance Specialist/Technical Specialist**

Quality Assurance Specialists and Technical Specialists are a part of the Regulatory Affairs or Quality Assurance Departments. These individuals maintain a working knowledge of analytical methods, laboratory procedures, and policies; perform compliance data reviews/evaluations; identify non-conformances; assist in root cause analysis; help develop corrective actions as necessary; and provide general support to the laboratory's quality assurance program.

#### **Data Review Specialist (DRS)**

Each ESC analytical department employs the use of designated senior analysts/technicians as data reviewer. This individual has analytical experience in their assigned area and reports to the Department Supervisor. Working knowledge of the instrumentation, computerized systems, documentation, and processes are keys to successful approval of data being generated in each area. The data reviewer performs the secondary review of the data generated within the department and gives final approval of the data for reporting. The data reviewer is responsible for the review of data for method and procedure compliance. In addition, the application of qualifiers is verified and approved. If the data reviewer determines a result to be questionable, the data is returned to the analyst for appropriate correction. If necessary, the issue may be elevated to the Department Supervisor to initiate appropriate action based on the severity of the problem.

#### **Technical Service Representative (TSR)**

The TSR is responsible for final report review. Once the data has completed the laboratory validation steps, the final report is generated. The TSR reviews the data for client specific requirements, report completeness and any outstanding anomalies. If an error is suspected, the report is delayed until the appropriate Department

Supervisor can be contacted to resolve the question. Each TSR has laboratory experience in one or more departments.

#### 5.2.4 Job Descriptions

Employee qualification requirements are maintained by the Human Resources Department and are facilitated through the use of written job descriptions. Educational requirements and experience are included in the job description. The Department Supervisor and CEO determine specific education and experience requirements for individual positions within the laboratory based on the specific department needs.

#### 5.2.5 Training Records

Details of any employee training performed at ESC are recorded on training records. Procedural training records are maintained within each department, while policy records are maintained by Human Resources and/or Regulatory Affairs. Training on new or revised Standard Operating Procedures is maintained digitally in the protected and secure QA/QC Directory or via hardcopy with the Master copy of the procedure.

### **5.3 A ACCOMMODATION & FACILITY DESIGN**

#### 5.3.1 Laboratory Facilities

The design of the laboratory supports good laboratory practices and does not adversely affect measurement integrity.

#### 5.3.2 Environmental Conditions

All ESC laboratory facilities, analytical areas, energy sources, lighting, heating, and ventilation facilitate proper performance of calibrations and tests. The laboratory ensures that housekeeping, electromagnetic interference, humidity, line voltage, temperature, sound and vibration levels are appropriately controlled to ensure the integrity of specific measurement results and to prevent adverse effects on accuracy or increases in the uncertainty of each measurement.

Environmental conditions are recorded appropriately, when monitoring is required. The laboratory documents deviations and corrective actions when environmental conditions are not within specified conditions.

Environmental conditions maintained by the laboratory are within the limits recommended in **ANSI/AIHA Z9.5-2003**. Measurements are not made if environmental conditions deviate from those stated.

Laboratory staff ensures adequate conditions in the facility using the steps listed below:

- Verify that air conditioning, lighting, heating, and ventilation are controlled and monitored.
- Maintain good housekeeping practices to promote a clean, uncluttered laboratory.
- Have sufficient space to minimize the risk of injury to staff and/or damage to standards or equipment
- Maintain a convenient and efficient work environment with effective separation of incompatible activities.
- Limit the amount of paper products used or stored in sensitive and/or clean areas to prevent dust contamination.

### 5.3.3 Separation of Incompatible Activities

The ESC complex facilitates the physical separation of analytical activities to prevent possible contamination between departments.

Each laboratory structure is specifically designed for the type of analytical activity that it contains. The air handling systems, power supplies, and gas supplies are specific for each laboratory department.

The following areas are designated and maintained under proper conditions and security:

- Sample Receiving
- Sample/supply shipping
- Chemical Storage
- Waste storage/disposal
- Data Handling
- Data Archiving

Routinely, the departments are required to maintain cleanliness and exercise good housekeeping measures to further minimize potential for contamination that could adversely affect analytical processes.

### 5.3.4 Facilities Access Management

Entrance into any ESC building requires an electronic ID badge with appropriate assigned access. Access is controlled to each area depending on the required personnel, the sensitivity of the operations performed, and possible safety concerns. Chemical/receipt and storage is assigned to the purchasing department and is access controlled by an attendant who organizes and maintains the inventory.

#### 5.3.5 Good Housekeeping

ESC ensures good housekeeping practices in all facilities to maintain a standard of cleanliness necessary for analytical integrity and personnel health and safety. Where necessary, areas are periodically monitored to detect and resolve specific contamination and/or possible safety issues.

### **5.4 TEST METHODS AND VALIDATION**

Method Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

#### 5.4.1 General

5.4.1.1 ESC uses appropriate methods and procedures for all analyses within its scope. These include sampling, handling, transport, storage and preparation of items to be analyzed and/or calibrated, as well as statistical techniques for analysis of data and, where appropriate, an estimation of the associated measurement uncertainty.

5.4.1.2 ESC has instructions on the use and operation of all relevant equipment and on the handling and preparation of items for analysis, where the absence of such instructions could jeopardize the results. All instructions, standards, manuals and reference data relevant to the work of the laboratory are maintained current and are readily available to personnel (see section 4.3).

5.4.1.3 Deviations from methods occur only if the deviation has been documented, technically justified, authorized, and accepted by the client.

#### 5.4.2 Selection of Methods

5.4.2.1 The laboratory uses analytical methods, including methods for sampling, which meet the needs of the client and are appropriate for the analyses performed. Methods utilized are preferably those published as international, regional, or national standards. The laboratory ensures that it uses the latest valid edition of a method unless it is not appropriate or possible to do so or unless regulatory requirements dictate specific revision use. Methods are supplemented with Standard Operating Procedures that list additional details to ensure consistent application.

Where mandated, only approved procedures are used. ESC utilizes a number of method sources to accomplish project requirements. See Section 2.1.3 for a list of method references.

5.4.2.2 When the client does not specify the method to be used or if a client selects an inappropriate or out of date method, the laboratory selects appropriate and approved methods that have been designated by the project regulatory program. The client is informed as to the method chosen and client approval is required.

#### 5.4.3 Laboratory Developed Methods

5.4.3.1 Introduction of analytical methods developed by the laboratory for its own use is a planned activity and is assigned to qualified personnel equipped with adequate resources.

5.4.3.2 Plans are updated as development proceeds and effective communication is maintained with all personnel involved in the development process.

#### 5.4.4 Non-Standard Methods

5.4.4.1 When it is necessary to employ methods not addressed by approved industry standard methods, these are subject to agreement with the client and must include a clear specification of the client's requirements and the purpose of the analysis. The method developed must be validated appropriately before use.

5.4.4.2 For new analytical methods, procedures are developed prior to the analysis and contain at least the following information:

- appropriate identification
- scope
- description of the type of item to be analyzed
- parameters or quantities and ranges to be determined
- apparatus and equipment, including technical performance requirements
- reference standards and reference materials required
- environmental conditions required and any stabilization period needed
- description of the procedure, including:
  - affixing identification marks, handling, transporting, storing and preparing of items,
  - checks to be made before the work is started,
  - verifying equipment function and, where required, calibrating and/or adjusting the equipment before each use,
  - method of recording the observations and results
  - any safety measures to be observed;
- criteria and/or requirements for approval/rejection;
- data to be recorded and method of analysis and presentation;
- uncertainty or procedure for estimating uncertainty.



#### 5.4.5 Validation of Methods – ESC SOP #030211, *Method Validation*

##### 5.4.5.1 Validation Description

Validation is process of confirmation by examination and the provision of objective evidence that the stated requirements for a specific method/procedure are fulfilled.

##### 5.4.5.2 Validation Summary

The laboratory validates all methods, including the following: EPA, Standard Methods, NIOSH, OSHA, and program mandated methods, approved methods used outside their intended scope, non-standard methods and amplifications, and modifications of approved methods to confirm that the methods are fit for the intended use. The validation is as extensive as is necessary to meet the needs in the given application or field of application. The laboratory records the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

##### 5.4.5.3 Validation for Client Need

The range and accuracy of the values obtainable from validated methods (e.g. the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross sensitivity against interference from the matrix of the sample.) are assessed for the intended use as relevant to the clients' needs.

##### 5.4.5.4 Limits

Descriptions of analytes, preparative and analytical methods, matrices, accuracy and precision targets, and MDLs and RLs are presented in the QA Manual Appendices.

**Method Detection Limits (MDLs) – 40CFR, Part 136, Appendix B** - SOP# 030206, *Method Detection Limits*

Detection limits are determined annually (or after any major changes to the analytical system and/or procedures) and are comparable to those established by the EPA and are not typically lower than recommended detection limits. To determine whether the EPA detection limit is being achieved, an MDL study is performed according to 40 CFR Part 136, Appendix B or the currently accepted and approved guidance. When using the Appendix B guidance, the standard deviation of, at least, seven replicate standards at or near the expected detection limit is calculated. MDLs are determined such that the risk of reporting a false positive is less than 1%. The method detection limit (MDL) is calculated as follows:

$$\text{MDL} = \text{TS}$$

where: S is the Standard Deviation of replicate measurements and

T is the value of Student's T for n-1.

If the MDL is higher than the EPA-method-suggested MDL, the calculated value is used as a basis for establishing the reporting limit (RL) for reporting. MDLs are recalculated on an annual basis or sooner if a material change in the instrumentation or method is enacted, or a change in the calibration response factor is noted. Additional studies may also be conducted to enhance the program.

Published MDLs may be set higher than experimentally determined MDLs to: 1) avoid observed positive interferences from matrix effects or common reagent contaminants or 2) for reporting convenience (i.e., to group common compounds with similar but slightly different experimentally determined MDLs).

Method detection limit studies may also utilize additional study components to better reflect practices to produce a more realistic detection limit as approved by regulatory guidance/requirements. Blank background studies yielding a value for the blank contributions at low level quantitations during routine analysis may be utilized to calculate detection limits that further ensure that the incidence of reporting false positives and false negatives is greatly reduced in some applications.

Any alternate or modified method for the determination of MDL studies utilized at ESC must be approved for the application for which it is used and be technically justified in its use to provide an improvement in the data being generated within the study.

#### **Reporting Limits (RLs)**

Reporting Limits (RLs) are typically set 3 - 10 times the calculated MDL determined above. Because reporting level checks are required, ease of preparation of commercial analytical mixes may dictate, to some extent, the reported RL. Generally, the RL is set at less than 3 times the MDL to provide added confidence in the validity of the analytical data. The final RL is determined based on the matrix, method, and analyst experience. RLs are verified daily using a calibration standard at a level equal to or less than the established RL, when required.

#### 5.4.5.5 Demonstration of Capability

##### **Initial and Continuing Demonstration of Capability (IDOC & CDOC)** (General Testing Other Than Environmental Lead)

**NOTE:** All IDOC & CDOC records are kept on file by the laboratory. Supporting data is filed with each demonstration. Completion and approval is recorded on the form found in the 2003 NELAP Standard

Quality Systems Module, Appendix C. Records of verification are required for each individual and are retained for a minimum of 10 years.

General Requirements:

- A DOC is performed for each analyte whenever the method, analysts, analytes, or instrument type is changed.
- The Department Supervisor certifies that technical staff members in their area of expertise are trained and authorized to perform all analyses for which the laboratory is accredited by signing the DOC form. The QA department is the final approval of all IDOCs and CDOCs
- More specific information can be found in SOP# 030205: *Technical Training and Personnel Qualifications* and in SOP# 350355: *Technical Training and Personnel Qualifications for Biology*.

**IDOC**

An initial demonstration of capability (IDOC) must be made prior to using any analytical method, at any time there is a significant change in instrument or method, and when a new analyst is trained. An analyst can achieve the IDOC requirement for a specific method by using sample spike results. The following guide is a general outline of the IDOC requirements:

- The QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration or purchase as a quality control sample from an outside source.
- The analyte(s) is diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the method stated or laboratory-calculated method detection limit.
- At least four aliquots are prepared and analyzed according to the method either concurrently or over a period of days.
- Using all of the results, calculate the mean recovery (x) in the appropriate reporting units (such as µg/L) and the standard deviations of the population sample (n-1) (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence values in micro and mold analyses, the laboratory must assess performance against established and documented criteria.
- Compare the information from above to the corresponding acceptance criteria for precision and accuracy in the published method. If no method criteria exist, the IDOC performance must be compared to in-house QC limits for laboratory control samples (LCS). Where appropriate, limits may be compared to the criteria listed in DOD QSM. If all parameters meet the acceptance criteria, the analyst may begin independent analyses of actual client field samples. If any one of the parameters does not meet

the acceptance criteria, the performance is unacceptable for that parameter. The analyst completes further training before attempting the IDOC process again.

### CDOC

Continuing Demonstration of Capability (CDOC) are performed at least annually by documentation that technical personnel have read, understood and agreed to perform the most recent version of the analytical method (the approved method or standard operating procedure) and documentation of continued proficiency by at least one of the following once per year:

- Acceptable performance of a blind sample (single blind to the analyst);
- Another demonstration of capability using at least four consecutive laboratory control samples with acceptable levels of precision and accuracy
- Successful analysis of a blind performance study sample

### Initial and Continuing Demonstration of Capability (IDOC & CDOC) (Environmental Lead Only)

### IDOC

Analysts/Technicians in training complete a minimum of four independent test runs of sample preparation and/or instrumental analysis. Independent runs are defined as analytical runs consisting of at least five known samples, one of which is a certified reference material or proficiency testing material, separated by a period of time sufficient to evaluate the testing material.

- Sample Preparation and Analytical Personnel - the recoveries of the associated reference materials or proficiency training samples for each run must be within  $\pm 10\%$  of the certified value, 75% of the time.

**NOTE:** The reference/proficiency test samples utilized are: 1) similar to matrices the analyst encounters during routine sample analysis, 2) cover the sample mass range for which the analytical SOP has been designed and 3) cover the Lead (Pb) concentration for which the analytical SOP has been designed. In cases where there are several matrices of potential concern, four independent runs are sufficient to provide adequate demonstration of performance.

### CDOC

Annual demonstrations are performed by Analysts/Technicians involved in Lead (Pb) analyses to show continued ability to adequately analyze samples for Lead

(Pb) based on standard reference materials (SRMs) or certified reference materials. This demonstration is done at a minimum of every six months and can be a part of the analysis of proficiency testing materials or quality control samples associated with routine sample analyses.

#### 5.4.6 Measurement Uncertainty - ESC SOP# 030221, *Measurement of Uncertainty*

##### 5.4.6.1 Uncertainty Definition

Uncertainty is defined as a variable associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurement type. This definition of uncertainty focuses on the range of values that is relevant to the analytical technique being utilized for the analysis of field samples.

The uncertainty of testing results are calculated and documented in accordance with the requirements of ISO 17025 Clause 5.4.6. The Estimation of Uncertainty of Measurement Procedure is applied to all in-house analytical methods, where practical. The uncertainty of measurement determination is also required of all ESC subcontractors.

##### 5.4.6.2 Uncertainty Procedure

The Estimation of Uncertainty of Measurement Procedure is applied for estimating uncertainty of measurement, except when the analytical methods preclude such rigorous calculations. In certain cases it is not possible to undertake metrologically and statistically valid estimations of uncertainty of measurement. In these cases the laboratory attempts to identify all the components of uncertainty and make the best possible estimation, and ensure that the form of reporting does not give an exaggerated impression of accuracy. Reasonable estimation is based on knowledge of the performance of the method and on the measurement scope, and makes use of previous experience and validation data.

The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as:

- Requirements of the method
- Requirements of the client
- The existence of narrow limits on which decisions of conformance are based

In practice the uncertainty of the result may arise from many possible sources, including an incomplete definition, sampling, matrix effects and interferences, environmental conditions, uncertainties of weights and volumetric equipment, reference values, approximations and assumptions incorporated in the measurement method and procedure, and random variation.

In cases where a well-recognized method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied the estimation uncertainty of measurement by following the method and reporting instructions (see section 5.10).

#### 5.4.6.3 Uncertainty Determination

Where possible, ESC utilizes data from Laboratory Control Samples (LCS) to determine the minimal uncertainty estimates in each matrix. LCSs are matrix dependent and are consistent representatives of the method effects on the particular matrix of choice. Uncertainty is determined per analytical technique and matrix, where applicable, and is performed using a population of 20 or more data points. Since the uncertainty is essentially constant, for each method, across a given matrix, ESC's method of choice is to determine uncertainty at the 95% confidence interval.

Procedure Summary:

- Select a group of representative data, from a single analytical process and matrix. Data set must be 20 individual measurements or greater.
- Determine the relative standard deviation of recovery data
- Calculate the expanded uncertainty as two times the relative standard deviation

#### 5.4.6.4 Uncertainty Results

ESC does not report uncertainty measurements on the final report. However, uncertainty determinations are available for review, when specifically requested for a project. The measurements are only applicable to the specific analytical procedure and matrix. No effects of sampling activities or related processes, which are outside the scope of the laboratory's activities, are considered in this determination.

When reporting results with uncertainty, the sample value is reported as " $X \pm U$  at a 95% confidence limit,  $k=2$ ", where  $X$  is the sample result,  $U$  is the expanded uncertainty, and  $k$  indicates the confidence level. Other confidence limits may be utilized depending on client need and by changing the  $k$  factor used.

#### 5.4.7 Control of Data

##### 5.4.7.1 Transfer Checks

Calculations and data transfers are subject to appropriate checks in a systematic manner.

#### 5.4.7.2 Automated Acquisition

When computers or automated equipment are used for the acquisition, processing, recording, reporting, storage or retrieval of data, the laboratory ensures that:

- computer software developed by the user is documented in sufficient detail and suitably validated as being adequate for use
- procedures are established and implemented for protecting the data; such procedures includes, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission and data processing
- computers and automated equipment are maintained to ensure proper function and are provided with the environmental and operating conditions necessary to maintain the integrity of data.
- where available, audit trail software features are utilized

#### 5.4.7.3 Commercial Software

Commercial “off the shelf” software, e.g., word processing, database and statistical programs in general use within its designed application range may be considered sufficiently validated. However, laboratory software configuration/modifications are validated as in 5.4.7.2.

#### 5.4.7.4 ESC Software Systems

<b>Table 5.4.7.4a LIMS</b>	
<b>System Desc</b>	<b>ription</b>
LIMS	The LIMS is a computerized database for data management. Access to the system is protected by coded password and access is granted based on user need.
Security	Level 1. Login, lookup sample status, generates worksheets. General access, every station has access. Level 2. Enter data, proofread and change data. The data entry person has access to this level. Level 3. Review and validate data, generate reports. Access is limited to the TSR, lab supervisors, data reviewers, QA and Regulatory Affairs. Once data is approved in the LIMS, it cannot be altered. Only the status of the sample may be changed to either "reported" or "invoiced."
Hardcopy Records	All paper records are retained by ESC and/or are stored within ESC’s Document Management System (Cyberlab/Openlab) in pdf and/or excel format. As the pages become historical (prior to the current working range of log numbers), they are removed from the logbook, prep book, or workbook in sequential order and permanently bound for storage in banker's boxes and/or are stored within ESC’s Document Management System (Cyberlab/Openlab) in pdf and/or excel format. They are cross-referenced by sample log number, date and storage number.

<b>Table 5.4.7.4a LIMS</b>	
<b>System Desc</b>	<b>ription</b>
Data Records	<i>Data</i> is available on electronic media. <i>Revisions</i> to the LIMS software are documented within the code. Each revision indicates the change in function, programmer's initials, and date of change. Programming has limited access and is accessible only by approved individuals through the use of passwords.
Calculations	All calculations performed by the LIMS are approved and submitted by the Laboratory Supervisors. Each calculation is tested parallel to manual calculations to ensure proper function.
Automatic Data Transfer	Data is transferred electronically from instrumentation by way of ESC customized software (Tree) directly to the LIMS. Data is also transferred electronically by way of ESC customized software (Prep Data) that transfers/saves Prep Data directly into the LIMS Database. Once the data has been transferred, it undergoes a screen review to ensure it has been transferred properly.

<b>Table 5.4.7.4b AUXILIARY SOFTWARE</b>	
<b>System Description</b>	<b>ription</b>
Auxiliary	Auxiliary Computer and Software Used to Generate and Validate Data
General	Several instruments have their own dedicated single computer and manufacturer-designed software to run them. Instruction manuals and other documentation provided by each manufacturer are maintained. ESC receives updates as they become available from the manufacturer. All raw and filtered data is stored on media (with uniquely titled data files on floppy discs) and all associated printouts and paperwork is filed. The original raw data is not accessed again unless it is subjected to uncertainty.
Method Files	Creation of any method or analyte files, necessary to run the appropriate analyses is the responsibility of the Department Supervisor. The Supervisor verifies that the compounds, wavelengths, retention time windows, calculation criteria, and other relevant parameters are correctly input into the specific method file. Analysts may only use the method files that have been specifically generated by the Supervisor.
Supplier Info	All purchased software that is used in conjunction with software specific instruments is guaranteed by the supplier to function as required. The supplier of the software performs all troubleshooting or software upgrades and revisions.
Validation	Computer software is validated for proper performance. The result of the validation is recorded, when in-house programming is the source of the calculation.



## **5.5 E EQUIPMENT**

### 5.5.1 Usability

Laboratory standards, equipment, and associated apparatus are suitable for the validation of acceptable performance of analyses and are maintained in accordance with this quality manual to include protection from dirt, dust, corrosion, and other causes of deterioration. Laboratory personnel investigate any equipment or standards, which are suspect in contributing to out-of-control conditions. Records of corrective actions for discrepancies are maintained in the laboratory (see Section 4.11).

### 5.5.2 Calibration of Equipment

5.5.2.1 To maintain the integrity of standards, all maintenance operations are performed according to documented procedures and the laboratory standards are:

- Selected for use according to the level of precision, accuracy, and uncertainty required
- Limited in access and use, to trained and authorized laboratory staff only
- Handled and safely stored separately from samples and according to method requirements

5.5.2.2 Primary standards, directly traceable to NIST standards, are obtained from a vendor approved by the A2LA or ISO accredited and all certificates of analysis are maintained on file in the laboratory.

5.5.2.3 Secondary standards are also obtained from a vendor approved by the A2LA or ISO accredited and all certificates of analysis are maintained on file in the laboratory. They are calibrated by comparison to primary standards. Calibration reports are maintained on file in the laboratory.

5.5.2.4 Working standards are prepared from certified stock standards. Standard preparation logs are maintained electronically via the Standards Logger in the ESC LIMS.

5.5.2.5 Support Equipment Calibration: Including, but is not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, volumetric dispensing devices, and thermal/pressure sample preparation devices. All support equipment is maintained in proper working order and records are kept of all repair and maintenance activities, including service calls.

5.5.2.6 Equipment used with nominal values and corrections is verified by calibration labs having ISO 17025, or other suitable, accreditation. A calibration interval is established for the equipment (i.e., environmental equipment, balances). All balances and temperature-indicating devices are calibrated or verified by an outside vendor at least once annually. Verifications are performed on balances on each day of use using laboratory's reference weights.

5.5.2.7 Calibration of equipment is conducted at a frequency to ensure that the equipment remains in tolerance during its use in the laboratory. Frequency of calibration is based on a review of calibration, maintenance, and repair history. Reviews are conducted by the QA and records are maintained.

5.5.3 Equipment Operation and Maintenance – See Table 5.5.3.3 for General Information

5.5.3.1 ESC's preventative maintenance program provides guidelines to ensure that every effort is made to keep equipment well maintained and prepared for the next project. Most equipment is kept in duplicate and spare parts are kept in stock.

Instrument/equipment manuals are kept in each department for quick reference to aid in problem diagnosis. ESC maintains service contracts on major laboratory equipment, so that in the event of failure, repairs can be made within a few days. The appropriate Department Supervisor is consulted if an instrument repair is required. If a solution to the problem is not found immediately, a call may be placed to the instrument manufacturer or maintenance support provider for assistance in diagnosing the problem, determining the extent of repair needed and a possible timeframe for repairs to be completed.

5.5.3.2 If analyses are scheduled and it appears that the equipment may be down for a longer period, ESC arranges for analyses to be performed by another qualified lab. This action is utilized if client required definite turnaround time or sample holding times would be exceeded.

5.5.3.3 General Equipment (All Labs)

If method calibration requirements for a particular procedure are more stringent than those listed here, they are followed when that procedure is performed.

**Table 5.5.3.3a General Equipment Calibration**

Equipment Activity	Frequency	Record Type
<i>Balances</i>	Verified with Class I NIST traceable weights when used	Daily, before use Logbook – Located in each respective lab
<i>Balances</i>	<ul style="list-style-type: none"> <li>• Clean</li> <li>• Check alignment</li> <li>• Service Contract</li> </ul> Top-loading balances are allowed a tolerance of $\pm 1\%$ , while analytical balances are allowed a tolerance of $\pm 0.1\%$ .	At least once annually by a qualified vendor Certificates from contractor.
<i>Weights – Class I</i>	<ul style="list-style-type: none"> <li>• Only use for the intended purpose</li> <li>• Use plastic forceps to handle</li> <li>• Keep in case</li> <li>• Store in desiccator</li> <li>• Re-calibrate</li> </ul>	Checked for accuracy by an external source, at least every 5 years, or sooner if necessary. Certificates from contractor.
<i>pH meters</i>	Calibration: <ul style="list-style-type: none"> <li>• pH buffer aliquot are used only once</li> <li>• Buffers used for calibration bracket the pH of the media, reagent, or sample analyzed.</li> <li>• Check must perform within 0.05 pH units. Temperature correction is performed either automatically by the instrument or manually depending upon the instrument used. Automatic temperature compensation probes are verified annually.</li> </ul>	Before use Calibrations are recorded in a logbook.
<i>Automatic pipettes</i>	Verify for accuracy and precision using reagent water and analytical balance	In-house – Monthly Contract – Semi Annually Tolerance is set at 2.0%, (ASTM standard = 3%). Monthly verifications are recorded in a logbook. Semi-annual cal. is verified by certificates from the cal. service.
<i>Refrigerators, Freezers, Hot plates and BOD incubators</i>	<ul style="list-style-type: none"> <li>• Thermometers are immersed in liquid to the appropriate immersion line</li> <li>• The thermometers are graduated in increments of 1°C or less</li> <li>• Temperature ranges are listed in app. SOPs</li> </ul>	Temperatures are recorded each day in use Logbook
<i>Ovens</i>	<ul style="list-style-type: none"> <li>• Thermometers are immersed in sand to provide even measurement</li> <li>• The thermometers are graduated in increments of 1°C or less</li> </ul>	Temperatures are recorded each day in use Logbook

**Table 5.5.3.3a General Equipment Calibration**

<b>Equipment Activity</b>		<b>Frequency</b>	<b>Record Type</b>
<i>Thermometers</i>	ESC NIST-certified thermometers  All working thermometers	Calibrated at least every 5 years, or sooner if necessary by a NIST calibration service, accredited to ISO/IEC 17025 and ANSI/NCSL Z540-1.  Verified at least annually against NIST-certified thermometers by an outside service.	Calibration certificates from the calibration service.  “Accuracy Assurance Program Test Data Sheets” provided by the servicer. All thermometers are tagged with current tolerances. Internal daily checks are recorded in a logbook.
<i>DO Meter</i>	Calibrated according to manufacturer's specifications. Using the recorded temperature and barometric pressure the meter is calibrated to the air saturation of dissolved oxygen using a conversion chart provided by the manufacturer.	Before use	Calibration of each meter is recorded in a separate logbook.
<i>Specific Conductivity Meter</i>	The conductivity meter is calibrated according to manufacturer's specifications. Temperature correction is performed either automatically by the instrument or manually depending upon the instrument used. <ul style="list-style-type: none"> <li>• Biomonitoring, potassium chloride with a conductivity value of 100 and 1000µmhos/cm is used as the calibration standard.</li> <li>• Wet Lab, potassium chloride with a value of 1413µmhos/cm is purchased from NSI for calibration purposes.</li> </ul>	Before use	Calibration of each meter is recorded in separate daily logbooks.
<i>Fume Hoods</i>	Check quarterly and must meet the OSHA minimum recommended face velocity of 60 – 100fpm.	Quarterly	Electronic log

<b>Table 5.5.3.3b Class 1 Weight Tolerance</b>				
<b>Value</b>	<b>ASTM Class 1 Tolerance</b>	<b>Unit</b>	<b>ASTM Class 1 Tolerance</b>	<b>Unit</b>
1mg	0.01	mg	0.00001	g
2mg	0.01	mg	0.00001	g
3mg	0.01	mg	0.00001	g
5mg	0.01	mg	0.00001	g
10mg	0.01	mg	0.00001	g
20mg	0.01	mg	0.00001	g
30mg	0.01	mg	0.00001	g
50mg	0.01	mg	0.00001	g
100mg	0.01	mg	0.00001	g
200mg	0.01	mg	0.00001	g
300mg	0.01	mg	0.00001	g
500mg	0.01	mg	0.00001	g
1g	0.034	mg	0.000034	g
2g	0.034	mg	0.000034	g
3g	0.034	mg	0.000034	g
5g	0.034	mg	0.000034	g
10g	0.05	mg	0.00005	g
20g	0.074	mg	0.000074	g
30g	0.074	mg	0.000074	g

#### 5.5.4 Identification of Equipment

Each item of equipment is uniquely labeled, marked or otherwise identified. Maintenance and calibration records for equipment and standards are maintained.

#### 5.5.5 Records of Equipment

Equipment lists are department specific and are found in the associated appendices to the QA Manual.

#### 5.5.6 Equipment Handling, Storage, Use, and Maintenance

All laboratory equipment is maintained, stored, and used in accordance with manufacturer's instructions. Operation manuals and instructions for proper maintenance of equipment are available to the staff and located in the laboratory.

Equipment is used or operated only when in a safe and reliable condition, by personnel who have been trained and are qualified. User instructions are available.

<b>Table 5.5.6 - GENERAL PREVENTATIVE MAINTENANCE</b>	
<b>Type</b>	<b>Description</b>
<i>Glassware</i>	Routine laboratory glassware is washed in a non-phosphate detergent and warm tap water. Before washing, all writing and large deposits of grease are removed with acetone. Glassware is then rinsed with: tap water, "No Chromix" solution, tap water, and deionized (DI) water. Glassware is stored in designated drawers or on shelves, inverted if possible. All organic glassware is rinsed with the required solvent, prior to use. Inorganic glassware is rinsed with DI water prior to use, which is a precaution against airborne contamination
<i>Logbooks</i>	<p>Maintenance logs are kept on all major laboratory equipment. The logbook is updated and signed when maintenance is performed (i.e., new rings, column or septum change, etc.). Maintenance logbooks are located in the immediate area of the instrument or are maintained digitally in LIMS. All preventive maintenance is noted either in the maintenance logbook or in the run log notebook.</p> <p>At a minimum, all maintenance logs contain the following:</p> <ul style="list-style-type: none"> <li>• All entries in the maintenance logs must be initialed and dated by the person performing the maintenance.</li> <li>• The instrument ID number or serial number.</li> <li>• Make and model of the instrument.</li> <li>• A unique identifier for each notebook</li> </ul>
<i>Service Records</i>	<p>Maintenance that requires a service call from the vendor should contain the following:</p> <ul style="list-style-type: none"> <li>• Must state details when the problem began, and what the problem was.</li> <li>• When a service call was placed.</li> <li>• When the service engineer came to repair the instrument.</li> <li>• When the problem was solved.</li> <li>• How the problem was solved.</li> </ul> <p>To verify that the instrument is running properly after service has been performed, recalibrate and analyze QC samples before the service engineer leaves.</p>
<i>Additional Records – Misc. Monitoring</i>	<p>Additional records are kept, updated and signed when technicians are assigned to perform the following tasks:</p> <ul style="list-style-type: none"> <li>• Monitor laboratory devices such as air compressors, vacuum pumps, heaters, etc., to ensure that they are properly lubricated and in good working condition.</li> <li>• Monitor on a daily basis: general lab QC areas, such as BOD incubators, temperature, drying ovens, desiccators, deionized water, sample cooler temperature, etc., and record appropriate parameters in the assigned QC logbooks.</li> <li>• Monitor the supply and quality of purchased chemicals, reagents and glassware, and keep inventory at established levels. All chemicals are dated in relation to receipt and date opened.</li> </ul>

### 5.5.7 Equipment Out of Service

When equipment is found to be in unacceptable condition or has been subjected to overloading or mishandling or if an instrument gives suspect results or has been shown by verification or otherwise to be defective, the equipment is clearly marked as out-of-service. Only the analyst responsible for the repair, or the Department Supervisor, can place equipment back in service. Once repaired and validated by calibration, verification, or other appropriate reviews, and found to perform

satisfactorily, the equipment can be placed back in service. The laboratory examines the possible effect of defective equipment on any previous calibrations.

#### 5.5.8 Status of Calibration

When appropriate, each item of equipment is labeled, marked, or otherwise identified to indicate its calibration status.

All equipment used with nominal values and corrections is labeled indicating the calibration status. Examples of this equipment include thermometers, calibration weights, and balances.

#### 5.5.9 Equipment Returning to Service

When for any reason, equipment goes outside the direct control of the laboratory, the laboratory ensures that the function and calibration status of the equipment are checked and shown to be satisfactory before the equipment is returned to service within the laboratory.

#### 5.5.10 Calibration Checks

Analytical instruments are calibrated per method requirements. Calibration and calibration check requirements are described in the appendices of this document for each analytical area. Balances are calibrated or verified at least annually by a qualified vendor. The calibration of each balance is verified each day of use with reference weights. Working thermometers are compared with the reference thermometers at least annually.

#### 5.5.11 Calibration Factors

Where calibrations give rise to a set of correction factors, the laboratory has procedures to ensure that copies (e.g., in computer software) are correctly updated.

#### 5.5.12 Safeguarding of Equipment Integrity

Analytical and supporting equipment is protected from inadvertent adjustments that could affect the integrity of the laboratory results. Instruments are located in access-protected areas. Software is tested and approved before use. Spreadsheets used in the calculation of analytical results are tested, approved, and locked before being placed into service.

## 5.6 MEASUREMENT TRACEABILITY

### 5.6.1 Policy (See SOP# 030230, *Standards Logger – Tree Operation*)

5.6.1.1 Standards and equipment significantly affecting the measurement integrity of analyses conducted by the laboratory are monitored for stability as part of the measurement control program. Standards and equipment are calibrated and/or verified before use to ensure acceptable performance. Any standard or equipment that appears unreliable or has exceeded the calibration interval is evaluated and/or removed from service.

5.6.1.2 When standards, reagents, or other certified consumables are received, they are assigned a unique number. The number is recorded in the LIMS Standards Logger with other important information concerning receipt date, supplier, expiration date, description, and volume. The number is then placed on the item and the Certificate of Analysis. The Certificate of Analysis is maintained electronically. Each item is dated upon opening. Each laboratory appendix contains a list of standard sources, receipt, and preparation information. Field personnel obtain several field standards from the lab and the standards are NIST traceable.

### 5.6.2 Measurement Traceability

5.6.2.1 ESC has established a program of calibration and verification that is designed to ensure that the measurements made by the laboratory are documented and traceable to national standards.

5.6.2.2 To provide external evidence of traceability, the laboratory participates in measurement control programs, such as proficiency tests, and other interlaboratory and collaborative round robins, as required (See SOP# 030212, *PT Program*).

### 5.6.3 Calibration/Verification

#### 5.6.3.1 Standards (Calibration)

5.6.3.1.1 Primary standards are calibrated to the standards set forth by the National Institute of Standards and Technology (NIST) or by an ISO 17025-accredited provider.

5.6.3.1.2 Primary standards are verified by secondary standards and are monitored through the measurement control programs established in the laboratory.

5.6.3.1.3 Standards are re-calibrated if there is damage to the standards or any significant change is observed in the measurement control program.



### 5.6.3.2 Standards (Verification)

5.6.3.2.1 Continuous verification of standards, through the measurement control program, ensures required measurement integrity of testing and includes:

- Statistical data from check standards and/or control charts (See SOP# 030207, *Quality Control Charting and Tracking*)
- Results from interlaboratory comparisons and/or proficiency tests (See SOP# 030212, *PT Program*).

5.6.3.2.2 Measurement assurance procedures for verification of standards are maintained in the laboratory, according to the individual method SOPs.

### 5.6.3.3 Measuring and Test Equipment

5.6.3.3.1 Equipment used with nominal values and corrections is calibrated by calibration labs having ISO 17025 accreditation, other suitable accreditation, or mutual recognition. A calibration interval is established.

### 5.6.3.4 Standard/Reagent Sources, Records, & Preparation

#### **Standard /Reagent Selection**

Standards and reagents are selected according to the method requirements. A minimum of analytical reagent grade is used when not method stated. The Department Supervisors or designee(s) makes the actual determination concerning quality and manufacturer. The purchasing agent maintains a list of approved vendors that have been evaluated and approved as suppliers of critical consumables, supplies and services that may affect the quality of environmental testing and calibration. All supplies that are directly used for analysis are inspected and verified upon arrival at the Laboratory. ESC SOP# 030210, *Materials Procurement for Analytical Processes*, details the entire procedure.

#### **Standard/Reagent Inventory**

An inventory of consumables and reagents are stocked in the individual laboratory areas. Any overstock items are kept in a controlled area, maintained by the purchasing department. Items are taken from the inventory area to the laboratories upon request.

#### **Standard/Reagent Preparation**

When standards are prepared in-house, they are weighed on an analytical balance, calibrated against Class "T" weights, diluted in Class "A" glassware, and compared against an external reference standard. The standard is marked with concentration, then signed and dated by the analyst, and placed in the appropriate storage area.

All dilutions of stock standards are prepared in Class A volumetric glassware. Where dilutions are made to volume, TC (to contain) glassware is used. All volumetric pipettes are Class A and designated as TD (to deliver). If the intermediate or working standards are to be saved and used again, the standard container is marked with concentration, date, source standard, expiration, and the analyst's initials.

All purchased stock standards are kept in a designated area within the appropriate section. Each chemical is marked in relation to date received, date opened, and expiration date.

#### **Standard/Reagent Logbooks**

A standard log is kept digitally in Standards Logger, indicating date of preparation, which standard (by lot number, if applicable) used, the amount used to prepare the solution, when it was made and expiration date or the recommended holding time. Reagents are recorded in the same manner as standards. Reagents that are prepared on a daily basis are recorded directly onto the raw data sheet. The analyst preparing the reagent initials and dates the raw data sheet.

### **5.7 S AMPLING**

#### **5.7.1 Sampling Plan**

When the laboratory carries out sampling of substances, materials or products for subsequent testing or calibration, it has a sampling plan and procedure for sampling. The sampling plan as well as the sampling procedure are available at the location where sampling is undertaken. Sampling plans are, whenever reasonable, based on appropriate governing methods. The sampling process addresses the factors to be controlled to ensure the validity of the analytical results.

#### **5.7.2 Client Requirements**

ESC has no jurisdiction over client deviations from any sampling plan but clients are encouraged to maintain proper records and to include appropriate information in all documents and communications.

#### **5.7.3 Sampling Records**

See Appendix III for information regarding the records of relevant field data.

#### 5.7.4 Field Sampling - General Summary

##### **Sample Labels**

All sample labels contain the following information: Client name, project name or ID, site ID, sampling point, time collected, and date collected. In addition the label includes information regarding preservation and method assignment. The project ID number is a unique ID number that can be associated with the client overseeing the project. Clients are designated in the ESC LIMS by a unique name referred to as a COCODE. The COCODE always precedes the project ID so that ESC personnel can easily relate a project ID to a particular client. As samples are logged in, they are assigned a unique sequential number. NO login number can be used twice. When the samples are logged in, all field label information is entered. All sample information can be accessed by entering the LIMS and viewing the sample login number. ESC has the capability to access all samples with the same project ID and print a summary of the samples.

##### **Field Notebooks**

Field notebooks are an essential part of the COC. Every detail concerning the sampling event must be documented. All documentation must be written with waterproof ink. All records are signed and dated by the individuals responsible for making the entry. Errors made during the documentation process are deleted by a single line with the initials of the person who corrected it and the date the change was made.

Crucial information to be recorded in the field notebook includes:

- Site identification
- Sample location
- Date and time of sample collection.
- Names of individual(s) collecting and documenting each sample.
- Names of all individuals present at the time of collection.
- Pertinent field conditions, including weather, site, traffic, other events, problems, etc.
- A copy of the Shipping Batch Detail Report is included as an attachment to the COC with each kit prepared and shipped.
- Specific sampling equipment used for the collection of each individual sample or sample group (Unique equipment identification numbers can be used.)
- If field analyses are performed, calibrations and results are recorded in field workbooks.

- When sampling monitoring wells, the field notes (whether in notebooks or on standard forms) must also document:
  - *Well casing composition and diameter*
  - *Water table depth*
  - *Well depth*
  - *Calculations to determine the volume of water to be purged*
  - *The total volume of water purged and how accomplished*
  - *The date and time well was purged, beginning to end*
  - *Use of fuel-powered units, bailers, etc.*
- When collecting surface water samples, the field notes must include the depth at which the sample was taken and the type of sampling equipment used.
- When water samples are collected over a period of time, it is necessary to indicate the following information in the field notes:
  - Collection beginning and ending time and date
  - Specific equipment used (manual or automatic)
  - Abnormal conditions of the sampling location
  - Safety precautions taken.

#### **Field Chain of Custody (COC)**

All field records include the signature of the person(s) responsible for the collection of the samples.

COC forms are completed and returned with the samples collected by ESC personnel. COC forms are also made available to clients collecting their own samples. A copy of the COC is retained in pdf form along with a pdf copy of the final report in the LIMS. The original is returned to the client with the final report. The COC is signed by the sampling personnel in the space referred to as "Collected by:".

A sample label is affixed to the side of each sample container before or at the time of sample collection. Pertinent information on the label includes a unique field identification number, sample description, preservative, method requested, date and time the sample was collected.

#### 5.7.5 Field Quality Control Checks

Blanks collected in the field are considered to be specific quality control for a set of samples. Analytical data that is consequential from the blanks is used to assess the integrity of field sampling and cleaning operations. This data can be used to confirm the use of contaminant-free sample containers and preservation reagents, and/or successful equipment cleaning. Potential on-site contamination, personnel sample collection technique accuracy, and problems that may occur in sample storage and transportation may also be revealed. Field blanks are treated in the same manner as regular samples: preserved with the same reagents, stored and transported in the same containers with samples, etc. For soil or solid samples, deionized water is used for blanks in similar containers.

#### 5.7.5.1 Field/Equipment Blanks

The purpose of field blanks is to evaluate the purity of preservation or additive reagents. Field blanks also represent the collection techniques, general sample containers to be filled, and the effects of on-site environmental conditions and possible contaminants. Field blanks are prepared at sampling locations by filling sample containers with DI water, adding appropriate preservatives or additives, sealing the containers, and completing all paperwork required for the samples. Field blanks are stored in the same shipping containers with the samples for transportation back to the lab.

Field blanks are generally collected at a rate of one blank per parameter group per day, or 5% of the samples in the parameter group, whichever is greater.

Equipment blanks help measure the effectiveness of pre-cleaning and field cleaning of equipment. They are used to evaluate sources of contamination that may also be found in a trip blank. Equipment blanks are collected according to the frequency of one blank per parameter group per day, or 5% of the samples in the parameter group, whichever is greater. Equipment blanks are prepared by rinsing the equipment with analyte-free water collected in the same manner as used for sample collection. The equipment blank is placed in the appropriate containers with required preservatives, if any.

Blanks must be taken and preserved, where required, for each method group. The blanks are stored in the same shipping containers as samples for transportation back to the lab.

#### 5.7.5.2 Trip Blanks

Trip blanks are used when sampling for volatile organic compounds to evaluate the cleanliness of the sample container, purity of the blank source water, and the exposure of the sample to contaminants during storage and/or transportation to and from the laboratory. The Laboratory supplies the trip blank with the sampling kit order, according to the following:

- The trip blanks are filled with analyte-free water plus any appropriate preservatives. (Matrix specific trip blanks are provided where necessary)
- The containers are sealed, labeled, and transported to the field in the same coolers or boxes with the sample containers to be used for sample collection.
- Trip blanks are not opened in the field.
- The trip blanks must be handled in the same manner as the samples being collected and are transferred (if required) with other samples for storage and transportation to the laboratory.
- If additional blanks (field and equipment) are necessary the same source water as the trip blanks are used.

- One trip blank per parameter group per cooler are used in the sampling event.
- The client is notified if the trip blank does not return with the sample set and a nonconformance is issued.

#### 5.7.5.3 Field Duplicates

Field duplicates are collected for each analyte group and are required whenever five or more samples are being collected. If more than ten samples are to be collected, the field duplication rate is 10%.

#### 5.7.5.4 Field QC Check Samples

All field instruments are calibrated at the beginning of each sampling day. Calibration is checked following every 10 samples or at maximum intervals of 4 hours. Calibration is verified at the end of the day. Recalibration is required if the QC check samples do not meet calibration criteria. The pH meter is evaluated after every ten samples using a buffer different than the ones used to calibrate the meter. The conductivity meter is evaluated by measuring the performance of the standard and the result must not vary by more than 5% from the true value after applying the cell constant.

#### 5.7.5.5 Field Duplicate Analysis

All analyses run in the field have duplicates performed at a rate of 10% of the total samples.

## 5.8 S AMPLE MANAGEMENT

### 5.8.1 Sample Management Procedures

Procedures have been established for the transportation, receipt, handling, protection, storage, retention, and disposal of samples. These procedures include provisions necessary to protect the integrity of the samples, and to protect the interests of the laboratory and our customers. These procedures are discussed below and more information can be found in the following SOPs; 060105 *Sample Receiving*, 060106 *Sample Storage and Disposal*, 060108 *Return Sample Shipping*, 060110 *Sample Shipping*, and 060112 *Cold Storage Management*.

#### 5.8.1.1 Sample Transportation

When a sample is received by the laboratory, the method of transportation is recorded on the COC. ESC routinely uses FED-EX, UPS, USPS, Velocity Express and various air carriers. Locally collected samples are sometimes carried in by the clients sample collection personnel or by ESC courier. When ESC is involved in the actual sample collection, the samples are packed with ice on site and transported by ESC field personnel utilizing proper COC protocol.

#### 5.8.1.2 Sample Receipt

Upon receipt, all samples are inspected. All abnormalities and non-conformances are noted. Sample receipt procedures are discussed further in section 5.8.3 below.

#### 5.8.1.3 Sample Handling – Preparation

The LIMS keeps track of samples and their corresponding log numbers to be analyzed. The analysts responsible for sample preparation maintain preparatory documentation, whether organic or inorganic. The analyst asks the LIMS to generate a prep sheet for a specific prep code. The LIMS provides all samples assigned to that prep code and prints a worksheet to record the required information.

- Samples are mixed prior to taking sub-samples for analysis, with the exception of VOC analyses. Sub-sampling within the laboratory is performed according to SOP# 030220, *Sample Homogenization and Sub-Sampling*.
- ESC currently maintains the following prep information: wet chemistry, metal digestions, organic extractions (by method), and GC and GC/MS injection logs.
- The analyst preparing the samples, dates and initials the entry, records any non-standard procedure (e.g., an aliquot for metal digestion other than

100mL for a water sample) or unusual observation, and which samples are spiked or duplicated.

- The organic extraction digital prep logbook contains all details concerning the sample extraction procedure.
- When a preparation is complete, the chemist assigned to perform the analysis is notified and the prepped sample is placed in the appropriate holding area.
- Each extract/digestate/distillate is labeled to provide the following information: lab ID, date prepped, amount prepared (volume/weight), dilutions, etc.
- The various prep books, workbooks, digital logbooks and injection logs document every manipulation of the sample through receipt, preparation, and analysis.

#### 5.8.1.4 Sample Handling – Analysis

- Each chemist has been assigned primary analytical procedures.
- Before beginning analysis they request a Laboratory Run Preview sheet from the LIMS and receive a printed page for the specific analysis in the form of a bench sheet. This Run Preview sheet lists all sample log numbers, sample type, and due dates relating to the samples that are ready for analysis. At that time the analyst can then select "all" or choose certain samples. Once the samples have been selected they are assigned to a unique run number and are then printed to a run bench sheet.
- The bench sheet provides all necessary information to complete the analysis such as: date and initials, flask numbers (where applicable), standards ID, instrument readings, response factors, aliquots, dilutions, final results, and all QC spike and duplicate information.
- When all data is recorded and the calculations are complete, a second chemist designated as a data reviewer, performs a second analytical review. If all calculations and other performance objectives pass method criteria, the second reviewer dates and initials the data and then releases the data for final reporting.
- If the lab reviewer rejects the work, he discusses the corrective action measures with the analyst.

#### 5.8.1.5 Sample Protection and Storage

Samples are stored and protected to avoid any deterioration, loss or damage to the samples. Sample protection and storage procedures are discussed further in section 5.8.4 below.



#### 5.8.1.6 Sample Retention and Disposal

- Samples and related extracts/digestates are retained for 45 days.
- Non-hazardous samples containing preservative are neutralized and disposed through the conventional municipal waste system.
- Non-hazardous solids are heated at 450 degrees Fahrenheit for two minutes and disposed of in a commercial waste container.
- All other waste is disposed of according to Section 6.

#### 5.8.1.7 Sample Subcontracting

- When samples are transferred to a subcontracted facility, a COC accompanies the samples. The COC contains the following required information: sample collection date and time, ESC login ID number, quantity and type of container, and the requested analysis.
- A copy of the COC and the sub-contract lab report is filed for permanent record.
- A subcontracted analysis log records date sent, where sent, log number, analysis requested, price, date report received, and date invoice received.

#### 5.8.2 Sample Information and Labeling

A unique sample identification number is generated for each sample submitted to the laboratory and is used throughout the analytical and disposal cycle. A record of all samples is established and maintained. The samples are stored according to published method requirements and determinative SOP. While in storage, samples are stored by sample ID and analyses required.

- When samples are logged in, the information entered into the LIMS includes sample description, date and time collected, collector ID, field ID, project ID, date and time received, receiver's ID, analysis requested, specific QC requirements, type of container and preservative, sample type, due date, and remarks.
- Each sample is assigned a unique and consecutive log number. After a sample is entered into the LIMS database and assigned a specific number identifier, the LIMS login screen automatically presents the next consecutive number for logging in the subsequent sample. Log numbers are not available for reuse and cannot be altered, although descriptive information, as well as sample specific comments can be modified until the final report is issued.
- A sample label with the log number is printed by the LIMS and affixed to the sample. Each label contains a unique container ID, represents the sample ID number, and is clearly marked with preservative and requested analysis.

- If the sample requires special DOT labeling, the label remains with the sample through receiving and disposal. If the sampling personnel note any special handling or precautions due to the nature of the sample, it is recorded on the sample label. The login person, at that time, makes a note in the LIMS to ensure that all departments have the information.
- The importance of sample label review is stressed to all chemists/analysts and sample handling personnel.
- Duplicate samples, collected in the field, are logged with a separate laboratory ID. Laboratory personnel are typically unaware of field duplication.
- Replicate samples with multiple analyses and containers have the same login ID number.
- The login person records the sample numbers assigned onto the COC. The LIMS provides documentation on the person authorized to enter sample log information.

### 5.8.3 Sample Inspection and Receipt

All samples are verified upon receipt as meeting its description and being free from damage. In the event of a sample being lost, damaged or otherwise unsuitable for use, full details of the incident are recorded and reported to the client by the Technical Service Representative via a nonconformance form, prior to any analytical action being taken. Any further action taken is at the direction of the client.

Login Technicians are responsible for sample login and assessing sample container integrity, documentation, and identification. Samples are inspected and noted for temperature, pH using narrow-range pH paper, headspace, proper container type, container integrity (broken or leaking), and volume levels. Samples requiring thermal preservation at 4°C must arrive at the laboratory above freezing but  $\leq 6^{\circ}\text{C}$ . If the samples are not appropriately preserved, the problem is noted on a sample nonconformance form, the client is notified, and, if the lab is instructed to proceed, proper preservation is performed. The sample nonconformance sheet becomes a permanent part of the COC. Samples, which require refrigeration, are placed in a laboratory cooler immediately after login.

Login Technicians are trained to recognize analyses with immediate, 24-hour, and 48-hour holding times. Those samples are designated as “short-holds”. When short-hold samples arrive at the laboratory, the Login procedure for those samples takes place immediately. All analysts are trained to assess incoming samples for holding time limitations.

If a sample has a holding time limitation, the LIMS issues a due date on the bench sheet to ensure that the extraction or analysis is completed within the time allowed. In the event that a holding time is exceeded, the TSR contacts the client,

informs them of the situation, and requests further direction. If instructed by the client to proceed with the analysis, a qualifier is added to the benchsheet, which is then carried on to reporting. The final report bears the explanation in the form of a qualifier.

#### 5.8.3.1 Sample Objectives

ESC receives samples for analysis for a variety of reasons, such as planning, estimating, process control, treatability as well as permit compliance reporting, site investigation, and remediation. When general screening is the goal of the client/project, analysis of improperly preserved or collected samples may proceed provided that the client is notified. In this instance, the chemist is notified and the proper documentation is noted on the final report.

#### 5.8.3.2 Sample Rejection Criteria

Where the analytical results are to be used for regulatory or compliance purposes, samples are rejected under the following conditions:

- If there is insufficient sample volume and the regulatory DQOs cannot be reached using reduced sample volume.
- If the preservation and container requirements were not followed correctly
- If there is headspace in a sample collected for volatiles analysis
- If the COC is missing, incomplete, or completed in pencil
- If the holding time for the desired analysis has expired
- If the integrity of the sample container or custody seal has been violated, if samples are broken or leaking, or if apparent contamination has occurred.
- If the temperature is outside of the method stated requirement
- If the samples are known to contain high levels of chemicals that present a health/safety risk (i.e. dioxins, radioactivity above background, etc.)

#### 5.8.3.3 Nonconformance Issues

- If there are problems with the samples, the event details are documented on the sample nonconformance form/COC; then, the sampler and/or client is notified.
- If the client insists on proceeding with analyses, even with full knowledge of the possible invalidity of the sample, a qualifier detailing the problem is added in the LIMS and it is also noted on the nonconformance form.
- The TSR, affected chemists, and reporting personnel are also notified.

#### 5.8.3.4 Login Confirmation

- On a daily basis, login confirmations are printed and auto-emailed to the client. A pdf copy is maintained in the ESC LIMS.
- A dual check is performed by Login and the Technical Service Group to insure proper analytical login from the COC.
- The original COC is forwarded to the reporting personnel to be reviewed and included with the final report.

#### 5.8.4 Sample Storage and Protection

Samples are taken to the appropriate storage location immediately after sample receipt and login procedures are completed. All sample storage areas are located in limited access areas and are monitored to ensure sample integrity.

Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

Refrigerated storage areas are maintained at  $\leq 6^{\circ}\text{C}$  (but not frozen) and freezer storage areas are maintained at  $< -10^{\circ}\text{C}$  (unless otherwise required per method or program). The temperature of each storage area is checked and documented at least once for each day of use. If the temperature falls outside the acceptable limits, then corrective actions are taken and appropriately documented.

Laboratory facilities are operated under controlled access protocols to ensure sample and data integrity. Visitors must register at the front desk in Building 3 and be properly escorted at all times.

Samples are removed from storage areas by designated personnel and returned to the storage areas as soon as possible after the required sample quantity has been taken.

##### 5.8.4.1 Special Requirements

The following entities mandate any required needs for special handling, storage, packaging, preservation, shipping, and marking provisions:

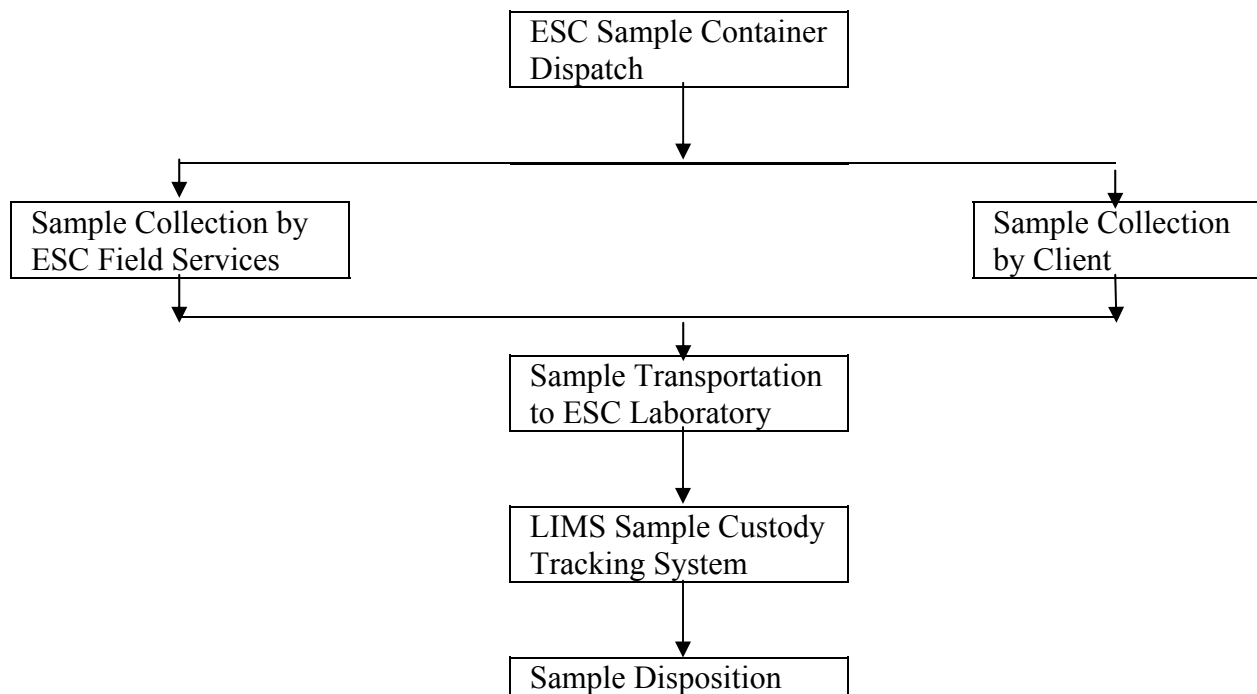
- EPA Approved Methods
- 40CFR Part 136.3
- 29 CFR (OSHA)
- 49 CFR (DOT)
- IATA (Dangerous Goods)

### 5.8.5 Chain of Custody

An important part of any sampling/analytical plan is ensuring sample integrity from collection to data reporting. Figure 5.8.5 below is a flow diagram that represents the sample custody process. All records and documentation required to track a sample from point of origin through disposal must be available. The documentation of the life of the sample is referred to as "chain of custody."

Formal chain of custody (COC) starts when the sample containers are requested. Such documentation includes container/shipping sheets, COC forms, field notebooks, field sample labels and custody seals, laboratory sample logs, sample extraction and digestion prep logs, analytical and instrument logs, QC data associated with the sample set, and the final report.

**FIGURE 5.8.5  
CHAIN OF CUSTODY PROCESS**



#### 5.8.5.1 Legal Chain of Custody

Legal COC involves all of the above, but actually begins in the laboratory with container preparation. All sample containers for collection purposes are purchased from the vendor as certified clean per EPA protocols. When a kit is prepared for delivery to the field a Shipping Batch Detail Report (BDR) is completed stating the number and type of bottles, required preservatives, date prepared, date sent, and person preparing kit. A copy of the Shipping Batch

Detail Report is generally kept beyond the estimated time of receipt of the kit back into the laboratory. The Shipping Batch Detail Report is sent with the kit for sampling guidance. The COC/Shipping BDR also represents the number of bottles sent to the client and the person preparing the kit. The containers are sent to the field in a portable cooler, with the COC/Shipping BDR inside, that is sealed by the person involved with the kit preparation and remains sealed until the recipient opens the kit. The individual receiving the containers for field use, signs the COC at the time the kit and containers are released for shipment to the laboratory. COC forms and sample container labels identify the analyses, dates, times, and individuals who remove samples.

The COC represents all persons who have the sample in their custody at a given time. The client designates common carriers on the COC when the sample is shipped back to the laboratory.

## 5.9 Q UALITY CONTROL

### 5.9.1 Quality Control Procedures

ESC has established quality control procedures for monitoring the validity of stated analytical methods. The resulting data are recorded in such a way that trends are detectable.

Monitoring of quality may include the following:

- regular use of certified reference materials and/or internal quality control using secondary reference materials;
- participation in inter-laboratory comparison or proficiency testing programs;
- replicate/duplicate analyses
- re-testing or re-calibration
- logic check or correlation of results from related analyses
- The identification and analysis of developing data trends by the use of control charts.

### 5.9.2 Quality Control Activities

Quality control data are analyzed using statistical techniques and, where they are found to be outside pre-defined criteria, planned action is taken to correct the problem and to prevent incorrect results from being reported.

### 5.9.3 Essential Quality Control Procedures

#### 5.9.3.1 Initial Calibration Verification (ICV) or Second Source Verification (SSV)

It is possible for a calibration curve to meet method criteria but still not have the ability to obtain accurate results because all calibration points are from the same source. To assess the accuracy of new calibration curves relative to the purity of the standards, a single standard from a secondary source is analyzed. This secondary source must be from an alternative vendor or from a different lot if the same vendor is used for the preparation of the calibration standards. The laboratory follows specific guidelines for ICV/SSV recoveries and further information can be found in the applicable laboratory SOP.

#### 5.9.3.2 Continuing Calibration Verification (CCV)

Analytical instrumentation is checked periodically to determine if the analytical response has changed significantly since the initial calibration was established. The values obtained from the analysis of the CCV are compared to the true values and a percent change calculated. The laboratory follows specific guidelines for CCV

frequency and recoveries. Further information can be found in the applicable laboratory SOP.

#### 5.9.3.3 Method Blank

A method blank is used to evaluate contamination in the preparation/analysis system and is processed through all preparation and analytical steps with its associated samples. A method blank is processed at a minimum frequency of one per batch of up to twenty samples.

The method blank consists of a matrix similar to the associated samples that is known to be free of analytes of interest. Method blanks are not applicable for certain analyses, such as pH, conductivity, flash point and temperature.

Each method blank is evaluated for contamination. The source of any contamination is investigated and documented corrective action is taken when the concentration of any target analyte is detected above the reporting limit and is greater than 1/10 of the amount of that analyte found in any associated sample. Some programs may require evaluating their method blanks down to ½ the reporting limit as opposed to the reporting limit itself. Corrective actions for blank contamination may include the re-preparation and re-analysis of all samples (where possible) and quality control samples. Data qualifiers must be applied to results that are considered affected by contamination in a method blank.

#### 5.9.3.4 Laboratory Control Sample

The Laboratory Control Sample (LCS) is used to evaluate the performance of the entire analytical system including preparation and analysis. An LCS is processed at a minimum frequency of one per batch of up to twenty samples.

The LCS consists of a matrix similar to the associated samples that is known to be free of the analytes of interest that is then spiked with known concentrations of target analytes. An LCS is not applicable for certain analyses where spiking procedures are not practical such as dissolved oxygen, odor, and temperature.

The LCS is evaluated against the method default or laboratory-derived acceptance criteria. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Any associated sample containing an 'out-of-control' compound must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier. An exception to this is when the acceptance criteria for the LCS are exceeded high and there are associated samples that are non-detects, then those non-detects can be reported. Another exception is when the acceptance criteria are exceeded low, those associated sample results may be reported if they exceed the maximum regulatory limit or decision level.



For LCSs containing a large number of analytes, it is statistically likely that a few recoveries will be outside of control limits. This does not necessarily mean that the system is out of control, and therefore no corrective action would be necessary (except for proper documentation). TNI has allowed for a minimum number of marginal exceedances, defined as recoveries that are beyond the LCS control limits (3X the standard deviation) but less than the marginal exceedance limits (4X the standard deviation). The number of allowable exceedances depends on the number of compounds in the LCS. If more analyte recoveries exceed the LCS control limits than is allowed (see below) or if any one analyte exceeds the marginal exceedance limits, then the LCS is considered non-compliant and corrective actions are necessary. The number of allowable exceedances is as follows:

<b>Number of Target Analytes</b>	<b>Allowable Marginal Exceedance Outliers</b>
>90	5 analytes allowed in the ME limit
71-90	4 analytes allowed in the ME limit
51-70	3 analytes allowed in the ME limit
31-50	2 analytes allowed in the ME limit
11-30	1 analytes allowed in the ME limit
<10	0 analytes allowed in the ME limit

#### 5.9.3.5 Matrix Spike

A matrix spike (MS) is used to determine the effect of the sample matrix on compound recovery for a particular method. The information from these spikes is sample or matrix specific and is not used to determine the acceptance of an entire batch. A MS consists of the sample matrix that is then spiked with known concentrations of target analytes.

A Matrix Spike/Matrix Spike Duplicate (MS/MSD) set is processed at a frequency specified in the applicable laboratory SOP or as determined by a specific customer request. Typically, an MS/MSD set is analyzed once per batch of up to twenty samples.

The MS/MSD set is evaluated against the method or laboratory derived criteria. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance, however, is based on method blank and LCS performance, not on MS/MSD recoveries. The spike recoveries give the data user a better understanding of the final results based on their site specific information.

#### 5.9.3.6 Sample Duplicate

A sample duplicate is a second portion of sample that is prepared and analyzed in the laboratory along with the first portion. It is used to measure the precision

associated with preparation and analysis. A sample duplicate is processed at a frequency specified by the particular method or as determined by a specific customer.

The sample and duplicate are evaluated against the method or laboratory derived criteria for relative percent difference (RPD). Any duplicate that is outside of these limits is considered to be 'out of control' and must be qualified appropriately.

#### 5.9.3.7 Surrogates

Surrogates are compounds that reflect the chemistry of target analytes, but are not expected to occur naturally in field samples. The purpose of the surrogates is to assess sample preparation, analytical efficiency, and to monitor the effect of the sample matrix on compound recovery.

The surrogates are evaluated against the method or laboratory derived acceptance criteria or against project-specific acceptance criteria specified by the customer. Any surrogate compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Samples with surrogate failures can be re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error.

#### 5.9.3.8 Internal Standards

Internal Standards are compounds not expected to occur naturally in field samples. They are added to every standard and sample at a known concentration prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. The laboratory follows specific guidelines for the treatment of internal standard recoveries and further information can be found in the applicable laboratory SOP.

#### 5.9.3.9 Proficiency Testing (PT) Studies

The laboratory participates in proficiency testing programs. PT samples are obtained from approved providers and analyzed and reported at a minimum of two times per year for the relevant fields of testing per matrix. PT samples are treated as typical customer samples. They are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

The laboratory does not share PT samples with other laboratories, does not communicate with other laboratories regarding current PT sample results, and does not attempt to obtain the assigned value of any PT sample from the PT provider.

The laboratory initiates an investigation and corrective action plan whenever PT results are deemed unacceptable by the PT provider. Additional PTs will be analyzed and reported as needed for certification purposes.

Additional information can be found in the SOP# 030212, *Proficiency Testing Program*

## 5.10 FINAL REPORTS/CERTIFICATES

### 5.10.1 General

The results of each analysis carried out by the laboratory are reported accurately, clearly, unambiguously, objectively, and in accordance with any specific instructions in the regulatory documents or standard operating procedures. The results are normally reported as a final client report and include all the information requested by the client and necessary for the interpretation of the analytical method results and all information required by the method of analysis.

### 5.10.2 Test Reports

In the case of a written agreement with the client, the results may be reported in a non-standard way and may not require the formalized information, but all associated analytical data is readily available and kept permanently on file for a minimum of 10 years. Specific programs or projects may require a longer data archive period.

Laboratory reports issued to the client for regulatory compliance analyses include, at a minimum, the following information:

- Title – “Report of Analysis”
- Laboratory name, address and phone number
- Client name, address, and contact
- Client name and/or site name
- Client or field identification number
- Collection personnel
- Analyte Name
- Method number for each sample analyses
- Analytical result for each analysis with applicable Data Qualifier as outlined in Table 5.14
- Dilution factor (where applicable)
- Method Detection Limit (when requested)
- Practical Quantitation Limit – designated on final report as RDL
- Date of sample preparation (when requested)
- Time of sample preparation if the holding time is <48 hours (when requested)

- Date of sample analysis
- Temperature at which pH measurements are made
- Date and time of sample collection from the Chain of Custody form
- Units of measurement
- Wet/Dry weight ID – Dry weight includes total solids value
- Identification of all laboratories providing analytical results in the report, including the appropriate laboratory certification numbers from all certifying agencies. The “S” qualifier is used when analyses have been subcontracted.
- Individual report statements: “The reported analytical results relate only to the sample submitted.” and “This report shall not be reproduced, except in full, without written approval from ESC”.
- Approval Signature
- Sequential page numbering with total pages identified.
- Date/Time Printed
- Revision date – if any
- Laboratory certification numbers as assigned by each certifying agency.
- In conjunction with Ohio VAP projects, a signed affidavit is also required.

An example of a final client report is presented in the following pages.

Figure 5.10.2: Example Final Client Report



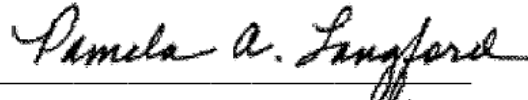
12065 Lebanon Rd.  
Mt. Juliet, TN 37122  
(615) 758-5858  
1-800-767-5859  
Fax (615) 758-5859  
Tax I.D. 62-0814289  
Est. 1970



<p style="text-align: center;"><b>Report Summary</b></p> <p>Tuesday [REDACTED]</p> <p>Report Number: [REDACTED]</p> <p>Samples Received: [REDACTED]</p> <p>Client Project:</p> <p>Description: NPDES Sample</p>
---

The analytical results in this report are based upon information supplied by you, the client, and are for your exclusive use. If you have any questions regarding this data package, please do not hesitate to call.

Entire Report Reviewed By:

  
Pam Langford, ESC Representative

**Laboratory Certification Numbers**

A2LA - 1461-01, AIHA - 100789, AL - 40660, CA - 01157CA, CT - PH-0197,  
FL - E87487, GA - 923, IN - C-TN-01, KY - 90010, KYUST - 0016,  
NC - ENV375/DW21704/BIO041, ND - R-140. NJ - TN002, NJ NELAP - TN002,  
SC - 84004, TN - 2006, VA - 460132, WV - 233, AZ - 0612,  
MN - 047-999-395, NY - 11742, WI - 998093910, NV - TN000032011-1,  
TX - T104704245-11-3, OK - 9915, PA - 68-02979, IA Lab #364, EPA - TN002

Accreditation is only applicable to the test methods specified on each scope of accreditation held by ESC Lab Sciences.

This report may not be reproduced, except in full, without written approval from ESC Lab Sciences. Where applicable, sampling conducted by ESC is performed per guidance provided in laboratory standard operating procedures: 060302, 060303, and 060304.



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Mt. Juliet, TN 37122  
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Fax (615) 758-5859  
Tax I.D. 62-0814289  
Est. 1970

REPORT OF ANALYSIS

[Redacted]

[Redacted]

Date Received : [Redacted]  
Description : NPDES Sample  
Sample ID : [Redacted]  
Collected By : [Redacted]  
Collection Date : [Redacted]

ESC Sample # : [Redacted]  
Site ID : [Redacted]  
Project # :

Parameter	Result	Det. Limit	Units	Method	Prep	PID	Analyzed	AID
BOD	BDL	5.00	mg/l	5210 B-20	01/31/15	1534	528 02/05/15 1025	ARM
Suspended Solids	4.4	2.5	mg/l	2540 D-20	02/03/15	0940	36 02/04/15 0847	MGM

BDL - Below Detection Limit  
Det. Limit - Practical Quantitation Limit (PQL)

Laboratory Certification Numbers:  
AIHA - 09227, AL - 40660, CA - I-2327, CT- PH-0197, FL - E87487, GA - 923, IN - C-TN-01  
KY - 90010, NC - ENV375, DW21704, ND - R-140, SC - 84004, TN - 2006, VA - 00109, WV - 233  
AZ -0612, MN - 047-999-395, NY - 11742, NJ - TN002, WI - 998093910

Notes:  
The reported analytical results relate only to the sample submitted  
This report shall not be reproduced, except in full, without the written approval from ESC.  
Reported: 02/06/15 11:33 Revised: 02/10/15 10:05

The following qualifier codes are routinely used by ESC when reporting data values that either meet the specified description outlined below or do not meet the quality control criteria of the laboratory:

**Table 5.10.2** ESC Qualifiers and Descriptions (Updated 4/15/15) *Subject to Revision*

U	Below Detectable Limits: Indicates that the analyte was not detected.
V	The sample concentration is too high to evaluate accurate spike recoveries.
V3	The internal standard exhibited poor recovery due to sample matrix interference. The analytical results will be biased high. BDL results will be unaffected.
B	The same analyte is found in the associated blank.
B1	The blank depletion was greater than the recommended maximum depletion of 0.2mg/L.
B2	The same analyte is found in the associated blank, the detection limit has been elevated.
E	The analyte concentration exceeds the upper limit of the calibration range of the instrument established by the initial calibration (ICAL).
H	The indicated analytical results were generated from a reinjection of the same sample extract or aliquot.
J	The identification of the analyte is acceptable; the reported value is an estimate.
J+	The associated batch QC was outside the upper control limits; associated data has a potential positive bias.
J-	The associated batch QC was outside the lower control limits; associated data has a potential negative bias.
J1	Surrogate recovery limits have been exceeded; values are outside upper control limits.
J2	Surrogate recovery limits have been exceeded; values are outside lower control limits
J3	The associated batch QC was outside the established quality control range for precision.
J4	The associated batch QC was outside the established quality control range for accuracy
J5	The sample matrix interfered with the ability to make any accurate determination; spike value is high
J6	The sample matrix interfered with the ability to make any accurate determination; spike value is low
J7	Surrogate recovery cannot be used for control limit evaluation due to dilution
O1	The analyte failed the method required serial dilution test and/or subsequent post-spike criteria. These failures indicate matrix interference.
P1	RPD value not applicable for sample concentrations less than 5 times the reporting limit.
W3	BOD cannot be determined due to apparent toxicity exhibited by the sample.
R	Rejected: Results have been rejected by the lab and should not be used.
Q	Sample was prepared and/or analyzed past recommended holding time. Concentrations should be considered minimum values.
W	Analysis was performed from an improper container.
T1	Sample(s) were received at greater than 6 degrees C.
T2	The laboratory analysis was performed from an unpreserved or inadequately preserved sample.
T4	Sample quantity was not sufficient.
T5	Sample quantity was not sufficient to complete analysis per recommended method guidelines.
T8	Sample(s) received past/too close to holding time expiration.

#### QUALIFIER REPORT INFORMATION:

ESC recognizes and utilizes sample and result qualifiers as set forth by the EPA Contract Laboratory Program. ESC firmly believes that relevant information pertaining to sample analysis be made available to the client. In addition to the EPA qualifiers adopted by ESC, the laboratory has implemented ESC qualifiers to provide more information pertaining to analytical results. Specific state regulatory agencies may utilize their own list of specific qualifiers for data associated with their program. In addition to those in the above table, ESC utilizes state specific qualifiers when required.

### 5.10.3 Optional Test Report Items

Where necessary, the final report contains a statement on the estimated uncertainty of measurement.

### 5.10.4 Calibration Certificates

ESC does not perform calibration activities for clients and therefore does not issue calibration certificates.

### 5.10.5 Opinions and Interpretations

Opinions and interpretations are allowed in final reports. They must be clearly marked as such, and the basis from which the opinions and interpretations have been made need to be documented.

### 5.10.6 Results from Subcontractors

ESC receives analytical reports from subcontracted laboratories. Results from subcontracted laboratories are clearly identified on the ESC client report.

### 5.10.7 Electronic Transmission of Results

In the case of transmission of analytical results by electronic means, the requirements set forth in this Quality Assurance Manual shall be met. All electronic transmissions contain a confidentiality notice that represents the following:

*Notice: This communication and any attached files may contain privileged or other confidential information. If you have received this in error, please contact the sender immediately via reply email and immediately delete the message and any attachments without copying or disclosing the contents. Thank you.*



### 5.10.8 Format of Reports

ESC client reports are designed to represent the analytical results unambiguously. Each client also has the option of using our web site to design a “custom” electronic report that will present results, historical data, and show trends in a format that is downloadable to a client database.

Client reports include the following information:

**Table 5.10.8 Data Package Contents**

<b>Level I Level II</b>	<b>Standard QC Data Package Provided Upon Request</b>
	<ul style="list-style-type: none"> <li>Final Analytical Report with qualifiers where necessary</li> <li>Sub-Contract Final Report if applicable</li> <li>Chain of Custody (COC) Form</li> <li>Method Blank</li> <li>Matrix Spike/Spike Duplicate Summary (MS/MSD) - with Control Limits</li> <li>Laboratory Control Sample Summary (LCS) - with Control Limits</li> <li>Reporting Limits listed on all reports</li> <li>Surrogate Recoveries for GC and GC/MS analyses (on final report)</li> <li>Case Narrative upon request</li> </ul>
<b>Level III</b>	<b>Data Package Provided Upon Request</b>
	<ul style="list-style-type: none"> <li>All QC Data Included in Levels I and II plus:</li> <li>MS/MSD analysis performed on specific sample upon request</li> <li>Initial and Continuing Calibration Information</li> <li>Instrument blank performance</li> </ul>
<b>Level III - Mod</b>	<b>Data Package Provided Upon Request</b>
	<ul style="list-style-type: none"> <li>All QC Data Included in Levels I, II and III plus:</li> <li>Chromatograms, including Batch QC, and Samples</li> </ul>
<b>Level III - Mod</b>	<b>Data Package Provided Upon Request</b>
	<ul style="list-style-type: none"> <li>Quantitation Reports</li> <li>Analysis Log</li> <li>Extraction Logs</li> </ul>
<b>Level IV</b>	<b>Data Package Provided Upon Request</b>
	<ul style="list-style-type: none"> <li>("CLP-Like" Validation Package)</li> <li>All QC Data Included in Levels I, II, III and III mod plus:</li> <li>Multiple Sample Dilutions Reported</li> <li>Before/After reports when manual integration is necessary (where requested)</li> <li>Initial and Continuing Calibration Chromatograms and Quantitation</li> <li>Surrogate, Tune, Internal Std &amp; Method Blank summary forms</li> <li>Standard Preparation Logs</li> </ul>

#### 5.10.9 Amendments to Reports

Reports that are amended after issue to the client, the amended report is clearly identified as such and a reference to the original report is made. The process is described in SOP 030223, *Report Revision*.

### 5.11 L LABORATORY DATA REDUCTION (*SOP 030201 Data Handling & Reporting*)

The primary analyst completes the majority of data reduction using the following:

- Spreadsheet calculation.
- Manual input of raw data for computer processing.
- Direct acquisition of raw data by computer.

#### 5.11.1 Spreadsheet Calculations

All data that are not captured by automatic acquisition are calculated using approved and controlled spreadsheets. No hand-calculations are performed. Any spreadsheets used are controlled, verified and locked to prevent unintentional changes.

#### 5.11.2 Manual Data Input

If data is manually entered for computer processing, a copy of the input and output is reviewed to ensure that no discrepancies exist. The persons entering the data and reviewing the data sign the data. The samples analyzed are evident. The data is identified by date analyzed or sample log number; in addition, a disc or tape backup is archived. Data files are uniquely identified by log number/parameter or date analyzed.

#### 5.11.3 Data Acquisition

If data is directly acquired from instrumentation and processed, the analyst reviews the following for accuracy: sample log numbers, calibration constants, response factors, reporting units, dilution factors, and established numerical values used for detection limits (if a value is reported as less than the MDL). The analyst signs and dates the resulting output.

Data that are produced by instrumentation such as calibration curves, absorbance responses, chromatograms, etc. are identified with the following information:

- Date of analysis and initials of analyst
- Initials of review analyst
- Instrument Identification
- Type of analysis

Instrument run logs can be cross-referenced by date to access information on instrument conditions.

#### 5.11.4 Analytical Data Records

Manual data entries are performed on hardcopy records with indelible ink. All errors are corrected with a single line strikethrough followed by initials and date. The corrected entry appears adjacent to the incorrect entry.

##### **Manual Data:**

All manual analytical data represents the following:

- Lab Sample ID
- Analysis Type and Method Number
- Date of analysis
- Prep Date/time
- Time of analysis (if holding time <72 hours)
- Instrument ID
- Calibration Date
- Analyst Initials
- Required QC
- Calculations
- Matrix
- Sample volume/amount
- Dilutions (if any)
- Units of measure
- Correlation coefficient
- Reagent ID – cross reference to preparation date/origin
- Standard ID – cross reference to preparation date/origin
- Calculations where required (manual)
- Qualifiers
- Comments where necessary
- Reviewer initials

##### **Instrument Data:**

The instrument printout and supporting data represents the following:

- Instrument ID – cross reference to maintenance log and instrument conditions
- Date/time of analysis
- Injection log/Sample run log
- Operator ID
- Instrument Responses
- Chromatograms/printouts (including manual integrations)

- Units of measure
- Sample amount/volume
- Dilutions
- Sample ID
- QC Samples
- Calibration Date
- Filename
- Comments
- Analyst Initials
- Review Initials
- Standard ID – cross reference to preparation date/origin
- Software version
- Method ID

### 5.12 D ATA VALIDATION PROCESS

<b>Table 5.12 DATA REDUCTION AND VALIDATION FLOW</b>		
<b>Primary Activity</b>	<b>Supporting Activity</b>	<b>Responsibility</b>
Review of COC	Login Confirmation to Client	Initially by Login Personnel and again by Technical Service Representative
Data Production and Reduction	Supporting documentation	Primary Analyst/Chemist
Review of Laboratory QC	Review of Data Completion and QC Limit Verification	Primary Analyst/Chemist
Approval of Laboratory QC	Review of Data Completion and QC Limit Verification	Data Reviewer/Senior Chemist
Approval of ESC Field QC and Data	Review of Field Records	Environmental Monitoring Manager
Data Entry to LIMS	Data Transfer	Analyst followed by Data Reviewer
Data Entry to LIMS	Data Transfer - Application of Qualifiers	Data Entry followed by Data Reviewer Verification
Final Report Review and Approval	TSR Approval/Signature	Technical Service Representative (TSR)

#### 5.12.1 Chain of Custody Review

One of the first steps in the validation process is review of the chain of custody (COC). The COC is reviewed first when the sample arrives. It is checked for completeness as well as time accountability. If the COC is complete and accurate, it is then processed through the system. If any irregularity is found, a non-conformance sheet is completed, with the TSR sign-off, etc. The samples are released for analysis upon approval of the COC.

### 5.12.2 Field Data

Field data must meet all calibration and continuing calibration requirements. All field data is reviewed for accuracy and completeness. The field data must be approved before it can be entered onto a report. The Environmental Monitoring Manager reviews recorded field data. Field QC criteria are explained in detail in Section 5.7 and in Appendix III.

### 5.12.3 Laboratory Data Review

All analytical data must undergo a multi-tiered review process prior to being reported to the customer. Data review is the process of examining data and accepting or rejecting it based on pre-defined criteria. These review steps are designed to ensure that reported data is free from errors and any non-conformances are properly documented. The laboratory's multi-tiered data review process is discussed below, and additional information regarding this process can be found in SOP #030227, *Data Review*.

**Primary Data Review** – Analysts performing the analysis have the primary responsibility for the quality of the data produced. The analysts initiate the data review process by reviewing and accepting/rejecting the data. This includes, but is not limited to; confirming all samples were prepared/analyzed according to the appropriate method and laboratory SOP, verifying dilutions are calculating properly, ensuring good chromatography, verifying proper spectral interpretations, evaluating quality control data, verifying that any customer/project specific requirements are met, and noting any non-conformances. The primary analyst is also responsible for compiling the initial data package for further data review.

**Secondary Data Review** – After the analyst have completed the primary data review process, the data package is then available for secondary data review that is performed by a qualified reviewer. This reviewer provides an independent technical assessment of the data. This includes, but is not limited to; confirming all samples were prepared/analyzed according to the appropriate method and laboratory SOP, verifying dilutions are calculating properly, ensuring good chromatography, verifying proper spectral interpretations, evaluating quality control data, verifying that any customer/project specific requirements are met, and noting any non-conformances. Reviews must also verify that all manual entries of raw data are accurate and there are no transcription errors.

**Final Administrative Review** – All final reports receive a final administrative review of some degree. Once the data have been technically reviewed and approved in the secondary data review process, authorization for release of the data from the analytical section is indicated in the LIMS. A Technical Service Representatives (TSR) will then perform a final administrative review of the data

which includes examining the report for method appropriateness, detection limit/QC acceptability, and any other apparent errors. If no errors are found, the TSR approves the report in LIMS and the client has the reports emailed to them. If errors are noted, the data is returned to the department for correction and resubmission to the TSR. In the case of DoD work, 100% of all packages must have a final administrative review to confirm that primary and secondary reviews were recorded properly and the data package is complete.

**Compliance Data Review** – Compliance data reviews are performed by the Quality Assurance/Regulatory Affairs department staff and are considered to be part of the overall internal audit program of the laboratory. These reviews are typically performed after the data has been released to the customer. A list is produced weekly from LIMS showing all methods run by the laboratory and how many batches were analyzed the previous week. Some of these data packages will undergo a compliance data review as per a schedule set by this department. For DoD work, at least 10% of all data packages will reviewed for technical completeness/accuracy. Findings from these compliance data reviews will be entered into the laboratory's corrective action system, and laboratory management will be informed of any major issues as soon as possible. Minor discrepancies will be discussed on a monthly basis by laboratory management in an effort to identify any trends or any underlying root causes.

## **6.0 WASTE MINIMIZATION/DISPOSAL AND REAGENT STORAGE**

ESC's sample disposal policy is founded on RCRA [40 CFR Part 261.4 (d)] and CWA [40 CFR Part 403 (Pretreatment)]. Part 261.4 (Figure 6.4) excludes a sample of waste while it is a sample; however, once no longer fitting the description of a sample, it becomes waste again. The policy is further strengthened by information found in "Less is Better" published by the ACS and developed by the ACS Task Force on RCRA.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. Refer to ESC SOP #030309, *Waste Management Plan* for detailed information.

### **6.1 Q QUARANTINED SOIL SAMPLES**

ESC maintains a permit to receive and analyze soils from foreign or quarantined areas. All non-hazardous soil samples are disposed of as originating from a quarantined area. All unconsumed soil samples and containers are sterilized in accordance with the current USDA regulations found in 7 CFR 301.81. Both container and contents are dry-heated at 450°F for two minutes, then crushed prior to disposal into a sanitary landfill. For further information refer to SOP# 030309, *Waste Management Plan*.

### **6.2 M OLD/BIOHAZARD SAMPLE DISPOSAL**

The laboratory has contracted a local licensed medical waste removal and disposal firm to remove all biohazard and medical waste generated by the laboratory. All waste arriving at the treatment facility is incinerated or steam sterilized complying with all Federal, State, County and local rules, regulations and ordinances. The medical waste containers are picked up at least weekly and confirmation records are available in the laboratory.

All wastes classified as non-biohazard are disposed of via the sanitary sewer following treatment with a disinfectant, such as Chlorox (hypochlorite). The disinfectant and waste liquid is one part disinfectant and five parts waste liquid. Waste disposal records indicating the disposal method are available in the laboratory.

### **6.3 REAGENTS, STORAGE AND WASTE DISPOSAL**

#### **6.3.1 Reagents:**

- All chemicals are at least ACS reagent-grade or better.
- All reagents and chemicals are checked for quality, purity and acceptability upon arrival in the laboratory.
- Each chemical container displays the following information: date opened and the expiration date.

- All reagent solutions prepared in-house are documented in Standards Logger and labels contain the following information: unique ESC identifier, date prepared, analyst initials, expiration date, and reagent name. In house reagents are recorded with the same information in Standards Logger.
- Purchased reagent solutions are labeled with a unique identifier assigned in Standards Logger when received, opened and with the expiration date.

### 6.3.2 Storage:

- Reagents requiring refrigeration are stored in the area of use in a suitable refrigerated storage that is separate from field sample storage.
- Reagents and standards used for volatile organic analysis are stored in a separate refrigerator and are not stored with field samples.
- See the following table for more information regarding reagent storage.

Item	Reagent Storage
Acids	Designated acid storage cabinets, in original container.
Organic Reagents - Flammables	Stored in flammables cabinet on separate air handling system from volatiles analysis.
Liquid Bases	Stored in designated cabinet, away from acids.
Solid Reagents	General cabinet storage.
Refrigerated Aqueous Reagents/Standards	Stored in walk-in cooler on designated shelves, away from field samples.
Stable Standard Solutions	Storage cabinet designated in each laboratory for standards.
Dehydrated Media	Dehydrated media is stored at an even temperature in a cool dry place away from direct sunlight. Media is discarded if it begins to cake, discolor, or show signs of deterioration. If the manufacturer establishes an expiration date, the media is discarded after that date. The time limit for unopened bottles is 2 years at room temperature. Where needed comparisons of recovery of newly purchased lots of media against proven lots, using recent pure-culture isolates and natural samples, are performed.
Pure Biological Cultures	All organisms are stored on Tryptic Soy Agar at 4°C in a dedicated refrigerator located in the biology department

### 6.3.3 Disposal:

- All excess, out of date or unneeded chemicals, reagents and standards are sent to the ESSH Office to ensure proper disposal. Excess chemicals designated as hazardous waste are lab packed and disposed of according to local, State and Federal regulations. Final disposal method is dependant on the classification of each individual chemical. Some sample extracts, chemicals or standards designated as hazardous waste may be disposed of into appropriate satellite accumulation areas.



- Any additional EPA waste codes resulting from addition of standard are applied to the satellite container, if applicable.
- ESSH prohibits the sink disposal of chemicals, the intentional release of chemicals through chemical fume hoods and mixing of nonhazardous lab trash with hazardous waste.
  - Sample and reagent/solvent disposal is handled in different ways according to toxicity.
    - Solvents, reagents, samples and wastes are segregated according to base/acid, reactive/non-reactive, flammable/non-flammable, hazardous/non-hazardous, soil/liquid etc. Samples are grouped together relevant to these categories and are disposed of accordingly.
    - Table 6.3 lists waste disposal methods for various test by-products.
  - Upon receipt and login, each sample is coded by sample matrix type. The codes divide samples into the following groups: air, industrial hygiene, wastewater, cake sludge, soil, drinking water and miscellaneous. As laboratory personnel review the data reported, the method of disposal is also determined.
  - The TSR is notified if samples are to be returned to the client.

## 6.4 CONTAMINATION CONTROL

### 6.4.1 Metals

The metals lab conducts quarterly lead wipe testing in order to ensure that the environment is contaminant free. All critical areas are included and a record is kept of the sampling plan (including locations) and results. Bench tops, balances, digestion equipment, and instrument areas are evaluated against the regulatory limit for lead contamination. Any detectable concentration must be less than the established regulatory limit for lead. If any detectable amount exceeds the established criteria, then the area must be cleaned and verified before analytical activities can resume in that location. See ESC SOP#: 340706, *Quarterly Monitoring for Lead Contamination*.

### 6.4.2 VOCs

The VOC Lab is physically separated from the Extraction Laboratory in order to eliminate contamination caused by the use of extraction solvents. Contamination is monitored daily through the use of instrument/method blanks. Refrigerator blanks are also used to ensure that cross contamination does not occur during volatile field sample storage.

### 6.4.3 Biological Lab

The aquatic toxicity testing, mold testing, and all other biological determinations are performed in the administrative building and are therefore physically separated from processes involving solvent or other chemical use that could negatively impact biological organisms. The mold lab conducts monthly analyses to ensure that the laboratory

environment is contaminant free. All critical areas are included and a record is kept of the sampling plan (including locations) and results.

**TABLE 6.4 - WASTE DISPOSAL**

**NOTE:** This information is a general guide and is not intended to be inclusive of all waste or hazardous samples.

PARAMETER WASTE	PRODUCTS	WASTE CLASSIFICATION	DISPOSAL METHOD
Acidity	slightly alkaline water	none	neutralize-sanitary sewer
Alkalinity	slightly acidic	none	neutralize-sanitary sewer
BOD, 5-day	Sample waste only	none	sanitary sewer
COD	acid waste, Hg, Ag, Cr+6	corrosive, toxic	dispose via haz waste regulations
Conductivity	Sample waste only	none	sanitary sewer
Cyanide, Total	acidic waste	corrosive	neutralize-sanitary sewer
Cyanide, Amenable	acidic waste	corrosive	neutralize-sanitary sewer
Flashpoint	Misc. Organic waste containing Chlorobenzene	Flammable	Dispose via haz waste regulations
Hardness, Total	pH 10.0 alkaline waste	none	neutralize-sanitary sewer
Extraction/prep	methylene chloride and hexane	toxic solvents	Reclaim for resale
Methylene Blue Active Sub.	Acidic Chloroform Waste	toxic & acidic	dispose via haz waste regulations
Nitrogen-Ammonia	alkaline liquids	corrosive	neutralize-sanitary sewer
Nitrogen-Total Kjeldahl	Trace Hg in alkaline liquid	corrosive toxic	neutralize-sanitary sewer
Nitrogen-Nitrate, Nitrite	mild alkaline waste	none	sanitary sewer
Oil & Grease and Petroleum/Mineral Oil & Grease	Hexane	Toxic solvent	dispose via haz waste regulations
pH	Sample waste only	none	sanitary sewer
Phenols	slightly alkaline, non-amenable CN-	none	sanitary sewer
Phosphate-Total and Ortho	combined reagent	listed	sanitary sewer
Reactive CN & S	Acidic waste	corrosive	Neutralize - sanitary sewer; waste is monitored for CN
Solids, Total/Suspended/Dissolved	Sample waste only	none	sanitary sewer
Turbidity	Sample waste only	none	sanitary sewer
Metals	acids, metal solutions	corrosive, toxic	highly toxic metal standards and samples - dispose via haz waste regulations
Volatile Organics	methanol	toxic solvents	dispose via haz waste regulations
Extractable Organics	solvents, standards	toxic solvents	dispose via haz waste regulations
Biological Non-biohazardous Waste	Gloves, plastic containers	none	Standard refuse

Subpart A-General Sec.

- 261.1 Purpose and scope.
- 261.2 Definition of solid waste.
- 261.3 Definition of hazardous waste.
- 261.4 Exclusions.
- 261.5 Special requirements for hazardous waste generated by conditionally exempt small quantity generators.
- 261.6 Requirements for recyclable materials.
- 261.7 Residues of hazardous waste in empty containers.
- 261.8 PCB wastes regulated under Toxic Substance Control Act.
- 261.9 Requirements for Universal Waste.

Sec.261.4 Exclusions.

(d) *Samples.* (1) Except as provided in paragraph (d)(2) of this section, a sample of solid waste or a sample of water, soil, or air, which is collected for the sole purpose of testing to determine its characteristics or composition, is not subject to any requirements of this part or parts 262 through 268 or part 270 or part 124 of this chapter or to the notification requirements of section 3010 of RCRA, when:

- (i) The sample is being transported to a laboratory for the purpose of testing; or
- (ii) The sample is being transported back to the sample collector after testing; or
- (iii) The sample is being stored by the sample collector before transport to a laboratory for testing; or
- (iv) The sample is being stored in a laboratory before testing; or
- (v) The sample is being stored in a laboratory after testing but before it is returned to the sample collector; or
- (vi) The sample is being stored temporarily in the laboratory after testing for a specific purpose (for example, until conclusion of a court case or enforcement action where further testing of the sample may be necessary).

(2) In order to qualify for the exemption in paragraphs (d)(1) (i) and (ii) of this section, a sample collector shipping samples to a laboratory and a laboratory returning samples to a sample collector must:

- (i) Comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements; or
- (ii) Comply with the following requirements if the sample collector determines that DOT, USPS, or other shipping requirements do not apply to the shipment of the sample:
  - (A) Assure that the following information accompanies the sample:
    - (1) The sample collector's name, mailing address, and telephone number;
    - (2) The laboratory's name, mailing address, and telephone number;
    - (3) The quantity of the sample;
    - (4) The date of shipment; and
    - (5) A description of the sample.
  - (B) Package the sample so that it does not leak, spill, or vaporize from its packaging.

(3) This exemption does not apply if the laboratory determines that the waste is hazardous but the laboratory is no longer meeting any of the conditions stated in paragraph (d)(1) of this section.

## 7.0 Common Calculations

- Percent Recovery (%REC)

$$\%REC = \frac{(MeasuredValue - SampleConc)}{TrueValue} * 100$$

where:

TrueValue = Amount spiked

MeasuredValue = Amount measured

SampleConc = Amount measured in source sample (Used for %REC in MS calculations)

NOTE: The SampleConc is zero (0) for LCS and Surrogate Calculations

- Relative Percent Difference (RPD)

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

where:

R1 = Result of Sample 1

R2 = Result of Sample 2

- Percent Difference (%D)

$$\%D = \frac{MeasuredValue - TrueValue}{TrueValue} * 100$$

where:

TrueValue = Amount spiked (can also be the CF or RF of the ICAL Standards)

Measured Value = Amount measured (can also be the CF or RF of the CCV)

- Percent Drift

$$\%Drift = \frac{CalculatedConcentration - TheoreticalConcentration}{TheoreticalConcentration} * 100$$

- Average

$$Average = \frac{\sum_{i=1}^n X_i}{n}$$

where:

n = number of data points

X<sub>i</sub> = individual data point

- Calibration Factor (CF)

$$CF = \frac{A_s}{C_s}$$

where:

$A_s$  = Average Peak Area over the number of peaks used for quantitation

$C_s$  = Concentration of the analyte in the standard.

- Response Factor (RF)

$$RF = \frac{(Conc_{.IStd})(Area_{Analyte})}{(Conc_{.analyte})(Area_{IStd})}$$

where:

$A_s$  = Response for analyte to be measured

$A_{is}$  = Response for the internal standard

$C_{is}$  = Concentration of the internal standard

$C_s$  = Concentration of the analyte to be measured

- Standard Deviation (S)

$$S = \sqrt{\frac{\sum_{i=1}^n (X_i - X_{ave})^2}{(n-1)}}$$

where:

$n$  = number of data points

$X_i$  = individual data point

$X_{ave}$  = average of all data points

- Relative Standard Deviation (RSD)

$$RSD = \frac{S}{X_{ave}} * 100$$

where:

$S$  = Standard Deviation of the data points

$X_{ave}$  = average of all data points

## 8.0 Revisions

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of the Quality Assurance Manual are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0	<p>General – Changed all references to Chief Regulatory Officer (CRO) and Quality Assurance Officer (QAO) to Compliance Director, and also Chief Operating Officer (COO) to Laboratory Director throughout this document to reflect the change in the job titles.</p> <p>Table 3.3a – Added additional terms and definitions</p> <p>Section 4.1.5.4 – Added Technical Director to the list of descriptions of management positions</p> <p>Section 4.1.5.5 – Revised Compliance Director to person currently holding this position</p> <p>Section 4.1.5.6 – Revised Quality Assurance Manager to person currently holding this position</p> <p>Figure 4.1 – Changed corporate org chart to the current lab wide org chart</p> <p>Section 4.2.5 – Removed availability of the procedure list via the ESC intranet</p> <p>Section 4.2.8 – Added policy for the use and control of electronic signatures</p> <p>Section 4.9.4 – Added Release of Nonconforming Work section which was previously located in section 4.11</p> <p>Section 4.11.8 – Added corrective action from compliance data reviews to the list of corrective action documents maintained by the Quality Assurance/Regulatory Affairs Department</p> <p>Section 4.14.2 – Reworded Performance Audit section for clarity</p> <p>Section 4.14.4 – Reorganized External Audit section for clarity and removed language about PT samples which was not applicable to external audits</p> <p>Section 5.2.2.5 – Clarified annual training section and added reference to SOP for further info</p> <p>Section 5.2.3 – Clarified responsibilities of Compliance Director and Quality Assurance Manager. Also added Quality Assurance Specialist and clarified the responsibilities of Technical Specialist.</p> <p>Section 5.4.7.2 – Added audit trail software functions are utilized where possible</p> <p>Table 5.4.7.4a – Added Cyberlab/Openlab to the hardcopy records management and added Tree and Prep Data software to the automatic data transfer</p> <p>Section 5.5.2.6 – Revised frequency of outside vendor verifications of balances and thermometers to at least annually.</p> <p>Table 5.5.3.3a - Revised frequency of outside vendor verifications of balances and thermometers to at least annually, and revised frequency of reference weights and thermometers to at least every five years. Revised frequency of fume hood checks to quarterly.</p> <p>Section 5.5.10 – Revised frequency of outside vendor verifications of balances and thermometers to at least annually</p> <p>Section 5.7.5.1 – Clarified frequency of Equipment Blanks and removed the applicable table</p> <p>Section 5.8 – Reorganized and reworded Sample Management section for clarity</p> <p>Section 5.8.1.6 – Revised temperature to 450°F for non-haz solid sample treatment prior to disposal</p> <p>Section 5.9 – Reorganized and reworded Quality Control section for clarity</p> <p>Section 5.10.5 – Reworded Opinions and Interpretations section for clarity and removed language about revisions to reports which is not applicable to opinions and interpretations</p> <p>Section 5.10.7 – Reworded Electronic Transmission of Results section and removed language about assembling data packages which is not applicable to this section</p> <p>Section 5.12 – Reorganized section and reworded laboratory data review section for clarity. Also added compliance data reviews.</p> <p>Section 6.1 – Added SOP reference for quarantine soil sample management</p> <p>Section 7 – New Section for common calculations which were in various other sections in the previous version of this document</p> <p>Section 8 – New Section to summarize revisions to the previous version of the QAM</p>

# **ESC Site Plan QUALITY ASSURANCE MANUAL**

## **APPENDIX I TO THE ESC QUALITY ASSURANCE MANUAL**

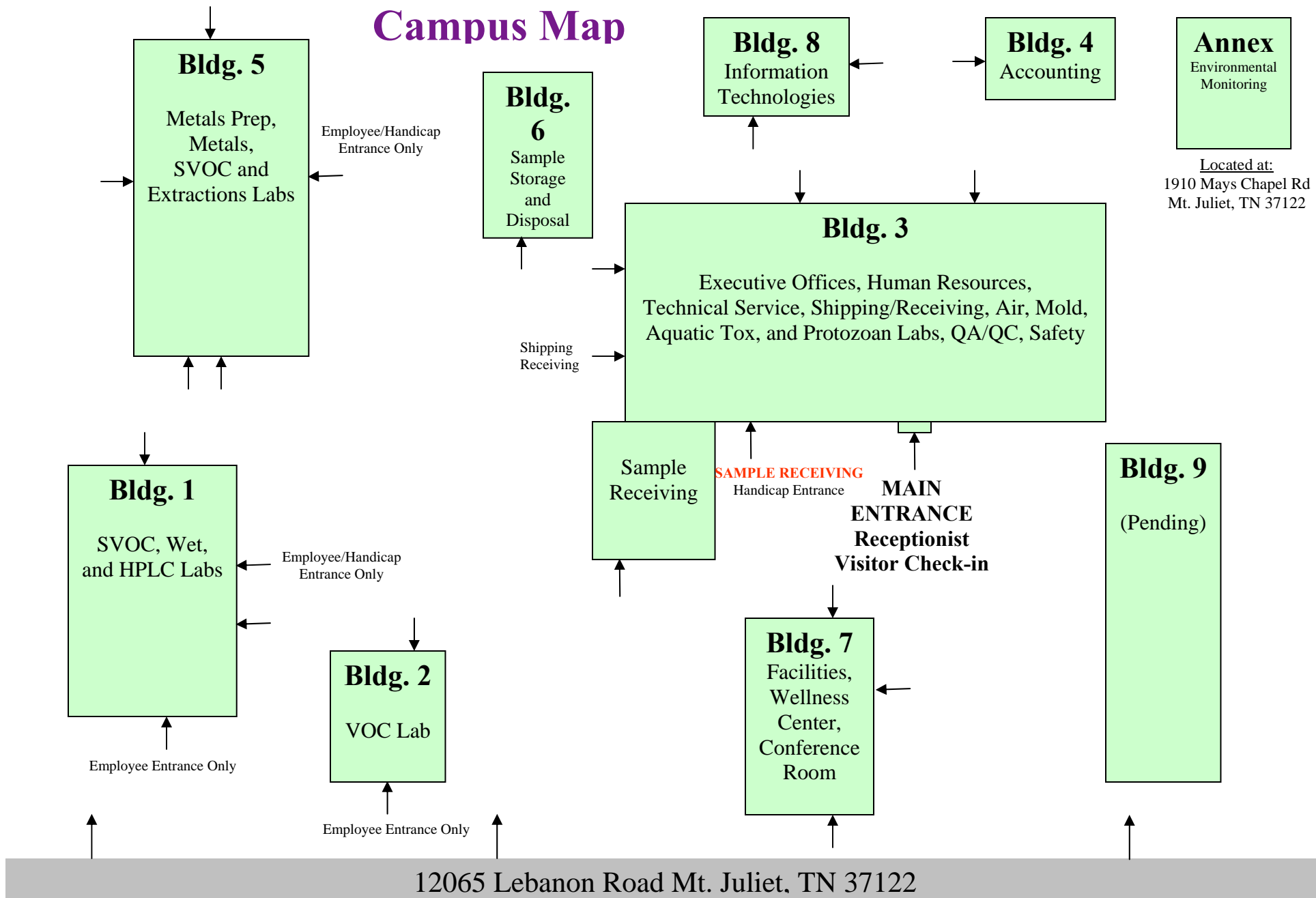
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# Campus Map





# **ESC Certifications QUALITY ASSURANCE MANUAL**

## **APPENDIX II TO THE ESC QUALITY ASSURANCE MANUAL**

for

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*Updated 8/27/15 (May be revised without notice)*

*Scopes of accreditation are on file in the Regulatory Affairs Department and are available upon request.*

State/Agency	Certificate Number	Expiration Date/Status	Certified Programs	Approved Programs <sup>8</sup>	Cert.Type	Cert. Authority
Alabama	40660	6/30/2016	DW	WW, RCRA, UST	Reciprocity	TN
Alaska	UST-080	1/11/2016	UST	UST	AK	AK
Arizona	AZ0612	6/25/2016	AIR, DW, WW, RCRA, UST		Audit	AZ
Arkansas	88-0469	1/21/2016	WW, RCRA, UST, Bioassay		NELAP	NJ
California	2932	8/31/2016	WW, RCRA, UST		NELAP	NJ
Colorado	None	3/31/2016	DW	WW, RCRA, UST	Reciprocity	TN
Connecticut	PH-0197	3/31/2017	DW, WW, RCRA, UST, Environmental Lead		Reciprocity	NJ, AIHA
Florida	E87487	6/30/2016	AIR, DW, WW, RCRA, UST		NELAP	NJ
Georgia DW	923	6/16/2016	DW		Reciprocity	TN
Georgia	None	9/30/2015	WW, RCRA, UST		NELAP	NJ
Georgia DW Crypto	923	6/30/2015	DW		Reciprocity	NJ
Idaho	TN00003	6/16/2016	DW	WW, RCRA, UST	NELAP	NJ
Illinois	200008	11/30/2015	DW, WW, RCRA, UST		NELAP	NJ
Indiana	C-TN-01	6/16/2016	DW	WW, RCRA, UST	Reciprocity	TN
Iowa	364	5/1/2016	WW, RCRA, UST		Audit	IA
Kansas	E-10277	10/31/2016	DW, WW, RCRA, UST		NELAP	NJ
Kentucky DW	90010	12/31/2015	DW	RCRA	Reciprocity	TN
Kentucky UST	16	11/30/2015	UST		Audit	A2LA
Kentucky WW	90010	12/31/2015	WW		Reciprocity	NJ
Louisiana	Agency ID 30792	6/30/2016	WW, RCRA, UST, AIR		NELAP	NJ
Louisiana DW	LA150002	12/31/2015	DW		NELAP	NJ
Maine	TN0002	7/5/2017	DW, WW, RCRA, UST		Reciprocity	TN, NJ
Maryland	324	12/31/2015	DW		Reciprocity	TN
Massachusetts	M-TN003	6/30/2016	DW, WW	RCRA, UST	Reciprocity	TN
Michigan	9958	6/16/2016	DW	WW, RCRA, UST	Reciprocity	TN
Minnesota	047-999-395	12/31/2015	WW, RCRA, UST		Audit	MN
Mississippi	None	6/16/2016	DW	WW, RCRA, UST	Reciprocity	NJ
Missouri	340	6/16/2016	DW	WW, RCRA, UST	Reciprocity	NJ
Montana	CERT0086	1/1/2016	DW	WW, RCRA, UST	Reciprocity	TN
Nebraska	NA	6/30/2015	DW	WW, RCRA, UST	Reciprocity	TN
Nevada	TN-03-2002-34	7/31/2016	WW, DW, RCRA, UST		NELAP	NJ
New Hampshire	2975	5/20/2016	DW, WW, RCRA, UST		NELAP	NJ
New Jersey - NELAP	TN002	6/30/2015	DW, WW, RCRA, UST, AIR		NELAP	NJ
New Mexico	None	Renewal	DW	WW, RCRA, UST	NELAP	NJ
New York	11742	4/1/2016	WW, RCRA, UST, AIR		NELAP	NJ
North C. Aquatic Tox	41	11/1/2015	Aquatic Toxicity		Audit	NC
North Carolina DW	DW21704	7/31/2016	DW		Audit	NC
North Carolina	Env375	12/31/2015	WW, RCRA, UST		Audit	NC
North Dakota	R-140	6/30/2015	DW, WW, RCRA		Reciprocity	TN, WI
Ohio VAP	CL0069	7/22/2017	WW, RCRA, UST, AIR		Audit	OH
Oklahoma	9915	8/31/2015	WW, RCRA, UST, BIOASSAY		NELAP	NJ
Oregon	TN200002	1/15/2016	DW, WW, RCRA, UST		NELAP	NJ
Pennsylvania	68-02979	12/31/2015	DW, WW, RCRA, UST	0	NELAP	NJ

State/Agency	Certificate Number	Expiration Date/Status	Certified Programs	Approved Programs <sup>8</sup>	Cert.Type	Cert. Authority
Rhode Island	221	12/30/2015	DW, Env. Lead	WW, RCRA, UST	Reciprocity	TN, AIHA
South Carolina	84004	6/30/2015	WW, RCRA, UST		NELAP	NJ
South Dakota	Pending	Pending				
Tennessee DW	2006	6/16/2016	DW	WW, RCRA, UST	Audit	TN
Tennessee DW Micro	2006	10/12/2015	DW Micro		Audit	TN
Texas - Env.	T 104704245-07-TX	10/31/2015	DW, WW, RCRA, AIR		Reciprocity	NJ
Texas - Mold	LAB0152	3/10/2017	MOLD		NA	TX
Utah	6157585858	7/31/2016	DW, WW, RCRA, UST		NELAP	NJ
Vermont	VT2006	1/5/2016	DW	WW, RCRA, UST	Reciprocity	TN
Virginia VELAP	460132	6/14/2016	DW, WW, RCRA, UST		NELAP	NJ
Washington	C1915	8/19/2016	DW, WW, RCRA, UST, AIR		Audit	A2LA
West Virginia	233	2/28/2016	WW, RCRA, UST		Audit	WV
West Virginia Crypto	9966 M	12/31/2015	DW		Reciprocity	NJ
Wisconsin	998093910	8/31/2016	WW, RCRA, UST, Bioassay		Audit	WI
Wyoming	A2LA	11/30/2015	UST	WW, RCRA	Audit	A2LA
A2LA <sup>1</sup>	1461.01	11/30/2015	DW, WW, RCRA, UST, AIR, MICRO		Audit	A2LA
AIHA <sup>2</sup>	100789	7/1/2016	IHLAP <sup>4</sup> , ELLAP <sup>5</sup> , EMLAP <sup>6</sup>		Audit	AIHA
DOD <sup>11</sup>	1461.01	11/30/2015	RCRA, UST		Audit	A2LA
EPA <sup>10</sup>	TN00003	None	Cryptosporidium		Audit	EPA
EPA <sup>10</sup> Region 8		7/15/2015	Drinking Water		Reciprocity	TN
USDA <sup>7</sup>	S-67674	9/27/2015	Quarantine Permit		Audit	USDA

(1) A2LA = American Association for Laboratory Accredited.  
 (2) AIHA = American Industrial Hygiene Association  
 (3) NELAP = National Environmental Laboratory Accredited. Program  
 (4) IHLAP = Industrial Hygiene Laboratory Accredited. Program  
 (5) ELLAP = Environmental Lead Laboratory Accredited. Program

(6) EMLAP = Environmental Microbiology Laboratory Accreditation Program  
 (7) USDA = United States Department of Agriculture  
 (8) Approved Programs = The state does not have a formal certification program.  
 (9) Pending = The state is processing our application.  
 (10) EPA = Environmental Protection Agency

1.0 SIGNATORY APPROVALS

# SAMPLING PROTOCOL QUALITY ASSURANCE MANUAL

## APPENDIX III TO THE ESC QUALITY ASSURANCE MANUAL


for


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### 3.0 SCOPE AND APPLICATION

This appendix discusses the standard practices and procedures utilized by ESC personnel for site selection and sample collection of various matrices. Topics addressed include field QA/QC procedures, together with equipment care and calibration for field sampling activities. Proper collection and handling of samples is of the utmost importance to insure that collected samples are representative of the sampling site. With this goal, proper sampling, handling, preservation, and quality control techniques for each matrix must be established and strictly followed. Precise identification of the collected samples and complete field documentation including a chain of custody are also vital.

ESC Lab Sciences does not provide sampling services for Industrial Hygiene and Environmental Lead analyses. We do require that all samples collected for these programs be sampled using the guidelines established by NIOSH, OSHA or other published protocol.

In addition, ESC Lab Sciences personnel do not conduct sampling in conjunction with the Ohio Voluntary Action Program (VAP).

### 4.0 LIST OF SAMPLING CAPABILITIES

• Parameter Group	• Sample Source
Extractable Organics	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Volatile Organic Compounds (VOCs)	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Metals	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Inorganic Anions	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Organics	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Physical Properties	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Cyanide	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge

• Parameter Group	• Sample Source
Microbiology	Surface water, groundwater, drinking water, wastewater
Macro Invertebrate Identification	Surface water, wastewater, sediments
Biotoxicity	Surface water and wastewater

## 5.0 GENERAL CONSIDERATIONS

The following procedures are used in all of ESC's sampling activities. These procedures must be considered in relation to the objectives and scope of each sampling event.

### 5.1 SELECTING A REPRESENTATIVE SAMPLING SITE

Selecting a representative sampling site is dependent upon the matrix to be sampled and type of analyses required. These matrix specific procedures are discussed in subsequent sections.

### 5.2 SELECTION AND PROPER PREPARATION OF SAMPLING EQUIPMENT

The type of sampling equipment to be used is specific to the sample matrix and the analyses to be conducted. These are discussed later in this section. Section 12.0 describes the equipment cleaning procedures utilized by ESC personnel.

### 5.3 SAMPLING PROCEDURES FOR INDUSTRIAL HYGIENE AND ENVIRONMENTAL LEAD SAMPLES

ESC does not provide sampling services for industrial hygiene and/or environmental lead analyses. Experienced laboratory personnel can assist with advice on sampling; however, the adequacy and accuracy of sample collection is the client's responsibility.

### 5.4 SAMPLING EQUIPMENT CONSTRUCTION MATERIALS

To prevent direct contamination or cross-contamination of the collected sample, great attention must be given to the construction material used for sampling equipment. Materials must be inert, non-porous and easy to clean. Preferred materials include Teflon<sup>®</sup>, glass, stainless steel and plastic. Plastics may not be used for collections where organics are the analytes of interest. Stainless steel may not be used where metallic compounds will be analyzed.

### 5.5 SELECTION OF PARAMETERS BEING ANALYZED

Parameters for analysis are usually dictated by and based on regulated monitoring conditions (i.e. NPDES or RCRA permits). If these do not apply, analyses are selected by ESC or the client based on federal regulations specific to the matrix being investigated.

## 5.6 ORDER OF SAMPLE COLLECTION

Unless field conditions demand otherwise, the order of sample collection is as follows:

1. Volatile organic compounds (VOCs)
2. Extractable Organics (includes Total Recoverable Petroleum Hydrocarbons [TRPH], Oil & Grease, Pesticides and Herbicides)
3. Total metals
4. Dissolved metals
5. Microbiological
6. Inorganic (includes Nutrients, Demand, and Physical Properties)
7. Radionuclides

## 5.7 SPECIAL PRECAUTIONS FOR TRACE CONTAMINANT SAMPLING

Many contaminants can be detected in the parts per billion or parts per trillion range and extreme care must be taken to prevent cross-contamination. Therefore, extra precautions apply where samples are collected for trace contaminants. These precautions include:

- A new pair of disposable latex gloves must be worn at each sampling location.
- Sample containers for samples suspected of containing high concentrations of contaminants are sealed in separate plastic bags immediately after collection and preservation.
- If possible, background samples and source samples should be collected by different field sampling teams. If different field teams are not possible, all background samples are collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated samples are not be placed in the same container as environmental samples. Ice chests or shipping containers for source samples or samples that are suspected to contain high concentrations of contaminants are discarded after use.
- If possible, one member of the field team should handle all data recording, while the other members collect samples.
- When sampling surface waters, water samples should always be collected before sediment samples are collected.
- Sample collection activities should proceed from the suspected area of least contamination to the suspected area of greatest contamination.
- ESC personnel uses equipment constructed of Teflon<sup>®</sup>, stainless steel, or glass that has been properly pre-cleaned (Sections 12.3 & 12.4) for collecting samples for trace metals or organic compounds analyses. Teflon<sup>®</sup>, glass, or plastic is preferred for collecting samples where trace metals are of concern. Equipment constructed of plastic or PVC are not be used to collect samples for trace organic compounds analyses.
- When fuel powered units are utilized, they are placed downwind and away from any sampling activities.
- Monitoring wells with free product are not sampled for trace contaminant analysis.



## 5.8 SAMPLE HANDLING AND MIXING

Sample handling should be kept to a minimum. ESC personnel must use extreme care to avoid sample contamination. If samples are placed in an ice chest, personnel should ensure that sample containers do not become submerged or tip over as this may result in cross-contamination. Small sample containers (e.g., VOCs or bacterial samples) are placed in airtight plastic bags to prevent cross-contamination.

Once a sample has been collected, it may have to be split into separate containers for different analyses. A liquid sample is split by shaking the container or stirring the sample contents with a clean pipette or pre-cleaned Teflon<sup>®</sup> rod. Then the contents are alternately poured into respective sample containers. Items used for stirring must be cleaned in accordance with the guidelines set forth in Section 12.0. Samples for VOCs, Cyanide, Total Phenol, and Oil & Grease must be collected as discrete grabs.

A soil sample may be split but must first be homogenized as thoroughly as possible to ensure representative sub-samples of the parent material. This is accomplished using the quartering method. The soil is placed in a sample pan and divided into quarters. Each quarter is mixed separately then all quarters are mixed together. This is repeated several times until the sample is uniformly mixed. If a round bowl is used, mixing is achieved by stirring the material in a circular fashion with occasional inversion of the material.

Soil and sediment samples collected for volatile organic compounds are not be mixed. The appropriate sample container should be filled completely, allowing little to no headspace.

Moisture content inversely affects the accuracy of mixing and splitting a soil sample.

## 5.9 QUALITY CONTROL SAMPLES

Quality control samples must be collected during all sampling events to demonstrate that the sample materials have not been contaminated by sampling equipment, chemical preservatives, or procedures relating to the sample collection, transportation and storage. A summary of the recommended frequency for collecting field quality control samples is presented in the following:

5.9.1 Quality Control Samples

Number of samples	Pre-cleaned equipment blank <sup>1</sup>	Field cleaned equipment blank	Trip blank (VOCs)	Duplicate
10 or more	minimum of 1 then 5%	minimum of 1 then 5%	one per cooler <sup>2</sup>	minimum one then 10% <sup>3</sup>
5 - 9	one	one	one per cooler <sup>2</sup>	one
less than 5	one	one	one per cooler <sup>2</sup>	Not required, but recommend a minimum of one. USACE projects require one. Client specific QAPP requirements must be considered.

<sup>1</sup> Pre-cleaned blanks are to be collected after the initial decontamination procedure has been completed but before the first sample is collected. Only one pre-cleaned or field-cleaned blank is required if less than 10 samples are collected. Only analyte-free water as defined in this document will be used in the preparation of any field and/or equipment blank.

<sup>2</sup> Where VOC methods are analyzed simultaneously, such as 601/602, only one (1) trip blank is required per cooler.

<sup>3</sup> Duplicate samples are collected for all VOC samples.

**5.10 VOLATILE ORGANIC COMPOUND SAMPLING**

**Water Samples**

Generally, groundwater, drinking water and wastewater samples for the analysis of volatile organic compounds are collected in duplicate pre-labeled 40mL vials. During bottle kit preparation in the laboratory, 200µL of concentrated HCl is added to each clean and empty vial. A Teflon® septum is placed in each cap and a cap is placed securely on each vial.

The sampler should check the water being sampled for residual chlorine content. This is done with residual chlorine testing strips. If no chlorine is present, the prepared vials may be filled as needed. If residual chlorine is present, add sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) to each vial prior to sampling.

To fill the vial properly, the sample is poured slowly down the inside wall of the vial until a convex meniscus is formed. Care should be taken to minimize turbulence. The cap is then applied to the bottle with the Teflon® side of the septum contacting the sample. Some overflow is lost; however air space in the bottle should be eliminated. Check for air bubbles by inverting the capped vial and tapping against the heel of the hand. This will dislodge bubbles hidden in the cap. If any bubbles are present, repeat the procedure using a clean vial and re-sample with a new preserved and septum. At a minimum, duplicate vials should always be collected from each sample location.

For analysis using EPA Method 524.2, samples that are suspected to contain residual chlorine, 25mg of ascorbic acid per 40mL of sample is added to each sample vial prior to sampling. Additionally, if analytes that are gases at room temperature (i.e. vinyl chloride, etc.) or any of the analytes in following table are not to be determined, 3mg of sodium thiosulfate is recommended for use to remove residual chlorine during sampling. If residual chlorine is present in the field sample at >5mg/L, then add additional 25mg of ascorbic acid or 3mg of sodium thiosulfate for each 5mg/L of residual chlorine present. Sample vials are filled as previously described. Following collection and dechlorination, Method 524.2 samples are adjusted to a pH of <2 with HCl.

Acetone	Acrylonitrile	Allyl chloride
2-Butanone	Carbon disulfide	Chloroacetonitrile
1-Chlorobutane	t-1,2-Dichloro-2-butene	1,1-Dichloropropanone
Diethyl ether	Ethyl methacrylate	Hexachloroethane
2-Hexanone	Methacrylonitrile	Methylacrylate
Methyl iodide	Methylmethacrylate	4-Methyl-2-pentanone
Methyl-tert-butyl ether	Nitrobenzene	2-Nitropropane
Pentachloroethane	Propionitrile	Tetrahydrofuran

For more detailed instructions, see the published method.

### **Soil Samples**

#### *Option 1 – Core Sampling Device*

Soil samples for volatile organic analysis are sampled using traditional core sampling methods. Once the core sample is collected, additional samples should be taken using an Encore™ sampler, either 5g or 25g, capped, sealed, and immediately cooled. The holding time for this method is 48 hours.

#### *Option 2 – Pre-weighed Vial*

In the other option for volatile soil sampling, 40mL vials with cap, Teflon® lined septum, preservative (5mL sodium bisulfate solution), and stir bar are pre-weighed, either by the user or the manufacturer. The vial is weighed on a balance capable of measuring to 0.01g and labeled with the pre-weighed value. In the field, place roughly 5g of sample into a pre-weighed vial, cap, and then immediately place on ice to achieve a temperature of ≤6°C. Exact soil weights can be measured using the pre-weight of the vial and the post-sampling weight. The difference represents the actual weight of the soil sample. The holding time for this method is 14 days.

Unless specifically permitted by the regulatory authority, VOC samples (liquid or solid) should never be mixed or composited.

## 5.11 OIL AND GREASE SAMPLING

Aqueous samples collected for oil and grease analyses must be collected as discrete grab samples. Sample containers should not be rinsed with sample water prior to sample collection and samples should be collected directly into the sample container. Intermediate vessels should only be used where it is impossible to collect the sample directly into the sample container and, in this case, only Teflon<sup>®</sup> beakers should be used. Samples should be taken from well-mixed areas.

## 5.12 CYANIDE SAMPLING

Cyanide is a very reactive and unstable compound and should be analyzed as soon as possible after collection. Samples are collected in polyethylene or glass containers and are pretreated and preserved in the manner specified in the following paragraphs.

### 5.12.1 Test for Oxidizing Agents

1. Test the sample with residual chlorine indicator strips.
2. Add a few crystals of ascorbic acid and test until negative.
3. Add an additional 0.6 grams of ascorbic acid for each liter sampled to remove residual chlorine.
4. Preserve the pretreated sample by to a pH > 12.0 with NaOH and cool to  $4 \pm 2^{\circ}\text{C}$ . Verify the pH of the samples as per Section 14.2.
5. Equipment blanks must be handled in the same manner as described in steps 1 through 4.

### 5.12.2 Test for Sulfide

1. Test the sample for sulfide using the sulfide test strip (formally HACH KIT).
2. If sulfide is not removed by the procedure below, the sample must be preserved with NaOH to pH > 12.0 and analyzed by the laboratory within 24 hours.
3. Sulfide should be removed by filtering visible particulate. Retain filter (filter #1).
4. Remove the sulfide by adding lead carbonate powder to the filtrate to cause the sulfide to precipitate out.
5. Test the filtrate for the presence of sulfide. If sulfides are present, repeat steps 1 and 4 until no sulfides are shown present.
6. The precipitate can now be filtered from the sample and this filter is discarded.
7. The sample is then reconstituted by adding the sediment collected on filter #1 back to the filtrate.
8. Preserve the pretreated sample to a pH > 12.0 with NaOH and cool to  $4 \pm 2^{\circ}\text{C}$ . Verify the pH of the samples as per Section 14.2
9. Equipment blanks must be handled in the same manner as described in steps 1 through 9.

### **5.13 BIOMONITORING SAMPLING**

Aqueous samples collected for Bioassay can be collected in either glass or HDPE plastic. There is no chemical preservation for this type of sample and the required volume varies with each type of analysis. Following sampling, all samples must be cooled to 0-6°C and can be held for a maximum of 36 hours from the time of collection. Grab and composite sample protocols are utilized for acute and chronic bioassays and are chosen according to permit requirements. Samples are collected with minimum aeration during collection and the container are filled allowing no headspace. Samples may be shipped in one or more 4L (1 gal.) CUBITAINERS® or unused plastic "milk" jugs. All sample containers should be rinsed with source water before being filled with sample. Containers are not reused. If the sample is a chlorinated effluent, total residual chlorine must be measured immediately following sample collection.

### **5.14 PROCEDURES FOR IDENTIFYING POTENTIALLY HAZARDOUS SAMPLES**

Any sample either known, or suspected, to be hazardous are identified as such on the chain of custody. Information explaining the potential hazard (i.e., corrosive, flammable, poison, etc.) are also be listed.

### **5.15 COLLECTION OF AUXILIARY DATA**

All auxiliary data are entered in the field records. Auxiliary data relative to a particular sampling location should be recorded concurrent with the sample event. Matrix specific auxiliary data are discussed later in this section.

### **5.16 TIME RECORDS**

All records of time are kept using local time in the military (24 hour) format and are recorded to the nearest minute.

### **5.17 REFERENCES**

ESC maintains copies of the various sampling references in the sample equipment room. Pertinent pages of these documents may be photocopied and taken to the field during sampling investigations. A bibliography of references used in the development of this section is presented in Section 17.

## **6.0 ANCILLARY EQUIPMENT AND SUPPLIES**

The equipment used to collect samples and conduct necessary purging activities is listed in subsequent sections for each type of sample. However, Section 6.1 lists some of the ancillary field equipment and instruments that may be required.

## 6.1 ANCILLARY EQUIPMENT AND SUPPLIES

Flow Measurement:	ISCO Continuous Flow Meters 3230, 3210, 2870; Flo-Poke pipe insert
Personal Protective Equipment:	Hard Hats, Face Shields, Rubber and Latex Gloves, Tyvex protective coveralls, rubber boots, safety glasses
Field Instruments:	Water Level Indicator, Continuous Recording pH Meter, Portable pH/Temperature Meters, Hach DR-100 Chlorine Analyzer, Hach CEL/700 Portable Laboratory, YSI Field Dissolved Oxygen/Temperature Meter w/ Submersible Probe, Portable Field Specific Conductance Meter, Hach 2100P Portable Turbidimeter
Chemical Supplies & Reagents:	Deionized Water, Tap Water, Liquinox Detergent, Isopropanol, Nitric Acid, Hydrochloric Acid, Sulfuric Acid, Sodium Hydroxide, Ascorbic acid, Sodium Thiosulfate, Ascorbic Acid, Zinc Acetate, pH calibration buffers (4.0, 7.0, and 10.0), Hach Sulfide Kit, lead carbonate powder, Specific Conductance Standard, Turbidity Standards
Tools:	Pipe Wrench, Bung Wrench, Crowbar, Hammer, Assorted Screwdrivers, Tape Measures, Channel Lock Pliers, Vise Grip Pliers, Duct Tape, Vinyl Pull Ties
Miscellaneous:	Cellular Phones, Pagers, Walkie Talkies, 12 Volt Batteries, Flashlights, Extension Cords, Brushes, Plastic sheeting, Fire extinguishers, Water Squeeze Bottles, First Aid Kit, lengths of rigid PVC conduit, aquatic sampling nets (Wilco)

## 7.0 WASTEWATER SAMPLING

### 7.1 SAMPLING EQUIPMENT

Type	Use	Materials	Permissible Parameter Groups
Continuous Wastewater Samplers- Peristaltic Pump	Sampling	Tygon tubing; glass or plastic sample container	All parameter groups except oil & grease, extractable organics, and VOCs
	Sampling	Teflon <sup>®</sup> tubing; glass sample container	All parameter groups except VOCs

## 7.2 GENERAL CONSIDERATIONS

The procedures used by ESC are generally those outlined in the *NPDES Compliance Inspection Manual*. Additional guidance is given in the *EPA Handbook for Monitoring Industrial Wastewater*. Some important considerations for obtaining a representative wastewater sample include:

- The sample should be collected where the wastewater is well mixed.
- Samples should not be collected directly from the surface/bottom of the wastestream.
- In sampling from wide conduits, cross-sectional sampling should be considered.
- If manual compositing is employed, the individual sample bottles must be thoroughly mixed before pouring the individual aliquot into the composite container.

## 7.3 SAMPLING SITE SELECTION

Wastewater samples should be collected at the location specified in the NPDES or sewer use permit if such exists. If the specified sampling location proves unacceptable, the project manager shall select an appropriate location based on site-specific conditions. An attempt should be made to contact the regulatory authorities for their approval. The potential for this type of issue highlights the need for a site inspection prior to the scheduled sampling event.

### 7.3.1 Influent

Influent wastewaters should be sampled at points of high turbulence and mixing. These points are: (1) the upflow siphon following a comminutor (in absence of grit chamber); (2) the upflow distribution box following pumping from main plant wet well; (3) aerated grit chamber; (4) flume throat; or (5) pump wet well when the pump is operating. Raw wastewater samples should be collected upstream of sidestream returns.

### 7.3.2 Effluent

Effluent samples should be collected at the site specified in the permit or, if no site is specified, at the most representative site downstream from all entering wastewater streams prior to final discharge.

### 7.3.3 Pond and Lagoon Sampling

Composite samples of pond and lagoon effluent are preferred over grabs due to the potential for ponds and lagoons to short circuit the projected flow paths. However, if dye studies or facility data indicate a homogeneous discharge, grab samples may be taken.

## 7.4 SAMPLING TECHNIQUES: GENERAL

The choice of a flow-proportional or time-proportional composite sampling program depends upon the variability of flow, equipment availability, sampling point configuration and accessibility. Flow metered sampling is necessary for complete wastewater characterization and should be utilized where possible. If not feasible, a time-proportional composite sample is acceptable.

A time-proportional composite sample consists of aliquots collected at constant time intervals and can be collected either manually or with an automatic sampler.

A flow proportional composite sample consists of aliquots collected automatically at constant flow intervals with an automatic sampler and a flow-measuring device. Prior to flow-proportional sampling, the flow measuring system (primary flow device, totalizer, and recorder) should be examined. The sampler may have to install flow measurement instrumentation if automatic sampling is to be used.

## 7.5 USE OF AUTOMATIC SAMPLERS

### 7.5.1 General

Automatic samplers are used when several points are sampled at frequent intervals, with limited personnel, or when a continuous sample is required. Automatic samplers used by ESC must meet the following requirements:

- Must be properly cleaned to avoid cross-contamination from prior sampling events.
- No plastic or metal parts shall come into contact with the sample when parameters to be analyzed could be impacted by these materials.
- Must be able to provide adequate refrigeration. Commercially available ice is placed in the sampler base and packed around the container approximately half way up the sample container.
- Must be able to collect a large enough sample for all required analyses. Composite sample containers (glass or plastic) hold up to 10 liters.
- A minimum of 100 milliliters should be collected each time the sampler is activated.
- Should provide a lift of at least 20 feet and be adjustable so that sample volume is not a function of pumping head.
- Pumping velocity must be adequate to transport solids without settling.
- The intake line must be purged a minimum of one time before each sample is collected.
- The minimum inside diameter of the intake line should be 1/4 inch.
- Have a power source adequate to operate the sampler for 48 hours at 15-minute sampling intervals.
- Facility electrical outlets may be used if available.



- Facility automatic samplers may be used for conventional parameters if they meet ESC QA/QC criteria.

Specific operating instructions, capabilities, capacities, and other pertinent information for automatic samplers presently used by ESC are included in the respective operating manuals and are not presented here.

All data relative to the actual use of automatic equipment on a specific job is recorded in sampling logbooks.

## 7.5.2 Equipment Installation

### 7.5.2.1 Conventional Sampling

Automatic samplers may be used to collect time-proportional composite or flow-proportional composite samples. In the flow-proportional mode, the samplers are activated by a compatible flow meter. Flow-proportional samples can also be collected using a discrete sampler and a flow recorder and manually compositing the individual aliquots in flow-proportional amounts.

Installation procedures include cutting and installing the proper length of tubing, positioning it in the wastewater stream, and sampler programming. All new tubing (Dow<sup>®</sup> Corning Medical Grade Silastic, or equal, in the pump and Tygon<sup>®</sup>, or equal, in the sample train) will be used for each sampler installation.

For a time-proportional composite, the sampler should be programmed to collect 100mL samples at 15-minute intervals into a refrigerated 10L plastic or glass jug, as appropriate for the particular parameters being analyzed.

For a flow-proportional composite, the sampler should be programmed to collect a minimum of 100mL for each sample interval. The sampling interval should be based on the flow of the waste stream.

### 7.5.3 Automatic Sampler Maintenance, Calibration, and Quality Control

To ensure proper operation of automatic samplers, the procedures outlined in this section are used to maintain and calibrate ESC automatic samplers. Any variance from these procedures is documented.

Proper sampler operation is checked by ESC personnel prior to each sampling event. This includes checking operation through three cycles of purge-pump-purge; checking desiccant and replacing if necessary; checking charge date on NiCad batteries to be used; and repairing or replacing any damaged items.

Prior to beginning sampling, the purge-pump-purge cycle is checked at least once. The sample volume is calibrated using a graduated cylinder at least twice, and the flow pacer that activates the sampler is checked to be sure it operates properly.

Upon return from a field trip, the sampler is examined for damage. The operation is checked and any required repairs are performed and documented. The sampler is then cleaned as outlined in Section 12.

## 7.6 MANUAL SAMPLING

Manual sampling is normally used for collecting grab samples and for immediate in-situ field analyses. Manual sampling may also be used when it is necessary to evaluate unusual waste stream conditions. If possible, manually collected samples are collected in the actual sample container that is submitted to the laboratory. This minimizes the possibility of contamination from an intermediate collection container.

Manual samples are collected by (1) submerging the container neck first into the water; (2) inverting the bottle so that the neck is upright and pointing into the direction of wastewater flow; (3) quickly returning the sample container to the surface; (4) shake to rinse. Pour the contents out downstream of sample location; (5) collect sample as described in steps 1, 2, and 3; pour out a few mL of sample downstream of sample collection. This allows for addition of preservatives and sample expansion.

Exceptions to the above procedure occur when preservatives are present in the sampling container or when oil & grease, microbiological, and/or VOC analyses are required. In these cases, samples are collected directly into the container with no pre-rinsing.

If the water or wastewater stream cannot be physically or safely reached, an intermediate collection container may be used. This container must be properly cleaned (Section 12) and made of an acceptable material. A separate collection container should be used at each sampling station to prevent cross-contamination between stations. The sample is collected by lowering a properly cleaned Teflon<sup>®</sup>, plastic, or glass collection vessel into the waste stream. The intermediate vessel may be lowered by hand, pole or rope.

## 7.7 SPECIAL SAMPLE COLLECTION PROCEDURES

### 7.7.1 Trace Organic Compounds and Metals

Due to the ability to detect trace organic compounds and metals in extremely low concentrations, care must be taken to avoid contamination of the sample. All containers, composite bottles, tubing, etc., used in sample collection for trace organic compounds and metals analyses should be prepared as described in Section 12.

Personnel handling the sample should wear a new pair of disposable latex gloves with each set of samples collected to prevent cross-contamination. A more detailed discussion is given in Section 5.7 under special precautions for trace contaminant sampling.

#### 7.7.2 Bacterial Analysis

Samples for bacterial analysis are always collected directly into the prepared glass or plastic sample bottle. The sample bottle should be kept closed until immediately prior to sampling and never rinsed with sample. When the container is opened, care should be taken not to contaminate the cap or the inside of the bottle. The bottle should be held near the base and plunged, neck downward, below the surface and turned until the neck points upward and upstream. The bottle should be filled to within one-inch of the top and capped immediately.

Section 14 presents preservation procedures and holding times. As holding times are limited to 6 hours for microbiological analyses, special arrangements may be required to ensure that these samples reach the laboratory within this timeframe.

#### 7.7.3 Immiscible Liquids/Oil and Grease

Oil and grease may be present in wastewater as a surface film, emulsion, solution, or a combination of these forms. A representative sample for oil and grease analysis is difficult to collect. The sampler must carefully evaluate the location of the sampling point to find the area of greatest mixing. Quiescent areas should be avoided.

Because losses of oil and grease will occur on sampling equipment, collection by composite sampler is not practical. Intermediate sampling vessels should not be used if possible. If intermediate collection vessels are required they should be made of Teflon<sup>®</sup> and be rinsed with the sample three times before transferring any sample to the sample container. Sample containers, however, should never be rinsed.

#### 7.7.4 Volatile Organic Compounds Analyses

Water samples to be analyzed for volatile organic compounds are collected in 40mL pre-preserved (200uL of concentrated HCl) vials with screw caps. A Teflon<sup>®</sup>-silicone septum is placed in each cap prior to the sampling event. The Teflon<sup>®</sup> side must be facing the sample.

Sampling containers with preservatives are pre-labeled prior to any field activities to reduce the chances of confusion during sampling activities. A complete list of sample preservatives, containers, holding times, and volumes is found in Section 14.

The sampler should check the water to be sampled for chlorine. This is done with residual chlorine indicator strips. If no chlorine is found, the vials may be filled. If residual chlorine is present, the sampling and preservation procedures listed in Section 5.10 of this manual must be performed.

## 7.8 AUXILIARY DATA COLLECTION

While conducting wastewater sampling, the following information may also be gathered:

- Field measurements -- pH, DO, conductivity, temperature
- Flows associated with the samples collected -- continuous flows with composite samples and instantaneous flows with grab samples
- Diagrams and/or written descriptions of the sample locations
- Photographs of pertinent wastewater-associated equipment, such as flow measuring devices, treatment units, etc.
- Completion of applicable forms required during specific investigations.

All observations, measurements, diagrams, etc., are entered in field logbooks or attached thereto.

## 8.0 SURFACE WATER AND SEDIMENT SAMPLING

### 8.1 EQUIPMENT

Equipment Type	Use	Material	Permissible Parameter Groups
<b>Surface Water Sampling</b>			
Kemmerer Sampler	Depth sampling	PVC	All parameter groups except extractable organics, VOCs, and oil & grease
Automatic Samplers	Sampling	Teflon <sup>®</sup>	All parameter groups except VOCs, oil & grease, & micro
	Sampling	PVC	All parameter groups except extractable organics, VOCs, oil & grease, and micro
Sample Collection Container	Sampling	Stainless steel	All parameter groups
Bailers	Sampling	Teflon <sup>®</sup>	All parameter groups
	Sampling	PVC	All parameter groups except extractable organics, VOCs, and oil & grease
<b>Sediment Sampling</b>			
Hand Augers	Sampling	Carbon Steel	Demand, nutrients, and extractable organics (for hard packed soils only)
Sediment Core Sampler	Sampling	Stainless Steel, Teflon <sup>®</sup>	All parameter groups
Encore <sup>™</sup>	Sampling	Teflon <sup>®</sup>	VOC Sediment/soil
Scoops	Sampling	Teflon <sup>®</sup> coated	All parameter groups

Equipment Type	Use	Material	Permissible Parameter Groups
Mixing Bowl	Compositing	Glass	All parameter groups except VOCs
Spoons, spatula	Sampling, compositing	Stainless Steel	All parameter groups

## 8.2 GENERAL

Selection of surface water sampling locations for water quality studies are determined by the objective of the study and waterway type. Factors that impact and alter water quality and characteristics (dams, bridges, discharges, etc.) must be considered. Accessibility is important.

## 8.3 SAMPLE SITE SELECTION

Fresh water environments are commonly divided into two types: (1) rivers, streams, and creeks; and (2) lakes, ponds, and impoundments. Since these waterways differ considerably in general characteristics, site selection must be adapted to each.

Prior to conducting a sampling event, an initial survey should be conducted to locate prime sampling points. Bridges and piers provide ready access to sampling points across a body of water. However, they should only be used when found not to be detrimentally impacting stream characteristics.

If wading for water samples must be done, caution should be used to avoid disturbing bottom deposits that could result in increased sediment in the sample. Shallow areas may be best for sediment sampling.

### 8.3.1 Rivers, Streams, and Creeks

Sampling sites should be located in areas possessing the greatest degree of cross-sectional homogeneity. Such points are easily found directly downstream of a riffle or rapid. These locations are also good for sediment sampling. In the absence of turbulent areas, a site that is clear of immediate point sources, such as tributaries and effluent discharges, may be used.

Typical sediment deposition areas are located at the inside of river bends and downstream of islands or other obstructions. Sites immediately upstream or downstream from the confluence of two streams or rivers should be avoided due to inadequate mixing of the combining flows. Also, backflow can upset normal flow patterns.

Great attention should be given to site selection along a stream reach:

- Sites should be spaced at intervals based on time-of-water-travel. Sampling sites may be located at about one-half day time-of-water-travel for the first three days downstream of a waste source for the first six sites and then approximately one day for the remaining distance.

- If the study data is for comparison to previous study data, the same sampling sites should be used.
- Sites should be located at marked physical changes in the stream channel.
- Site locations should isolate major discharges as well as major tributaries.

Dams and weirs usually create quiet, deep pools in river reaches that would otherwise be swift and shallow. When times of travel through them are long, sites should be established within them.

Some structures, such as dams, permit overflow that may cause significant aeration of oxygen deficient water. Sites should be located short distances upstream and downstream of these structures to measure the rapid, artificial increase in dissolved oxygen (DO), which is not representative of natural aeration.

A minimum of three sites should be located between any two points of major change in a stream, even if the time-of-travel between the points of change is short. Major changes include, but are not limited to, a waste discharge, a tributary inflow, or a significant change in channel characteristics. Sampling three sites is also important when testing rates of change of unstable constituents. Results from two of three sites will usually support each other and indicate the true pattern of water quality in the sampled zone. If the effect of certain discharges or tributary streams of interest is desired, sites should be located both upstream and downstream of these points.

Due to the tendency of the influent from a waste discharge or tributary to slowly mix, cross-channel, with the main stream, it is nearly impossible to measure their effect immediately downstream of the source. Thus, samples from quarter points may miss the wastes and only indicate the quality of water above the waste source. Conversely, samples taken directly in the stream portion containing the wastes would indicate excessive effects of the wastes with respect to the river as a whole.

Tributaries should be sampled as near the mouth as possible. Often, these may be entered from the main stream for sampling by boat. Care should be taken to avoid collecting water from the main stream that may flow back into the tributary as a result of density differences created by temperature, salinity, or turbidity differences.

Actual sampling locations vary with the size and amount of turbulence in the stream or river. Generally, with streams less than 20 feet wide, well mixed areas and sampling sites are readily found. In such areas, a single grab sample taken at mid-depth at the center of the channel is adequate. A sediment sample can also be collected at the center of the channel. For slightly larger streams, at least one vertical composite should be taken from mid-stream. It should be composed of at least one sub-surface, mid-depth, and above the bottom sample. Dissolved oxygen, pH, temperature, conductivity, etc. should be measured on each aliquot of the vertical composite. Several locations should be sampled across the channel width on the larger rivers. Vertical composites across the channel width should be located

proportional to flow, i.e., closer together toward mid-channel where flow is greater and less toward the banks where the flow proportionally lower.

The field crew will determine the number of vertical composites and sampling depths for each area. They should base their decisions upon two considerations.

1. The larger the number of sub-samples, the more nearly the composite sample will represent the water body.
2. Taking sub-samples is time consuming and expensive, and increases the chance of contamination.

A number of sediment samples should be collected along a cross-section of a river or stream to adequately characterize the bed material. The normal procedure is to sample at quarter points along the cross-section of the site. When the sampling technique or equipment requires that the samples be extruded or transferred at the site, they can be combined into a single composite sample. However, samples of dissimilar composition should not be combined. They should be kept separate for analysis in the laboratory. To ensure representative samples, coring tubes are employed. The quantity of each sub-sample that is composited shall be recorded.

### 8.3.2 Lakes, Ponds, and Impoundments

Lakes, ponds, and impoundments have a much greater tendency to stratify than rivers and streams. This lack of mixing requires that more samples be obtained from the different strata. Occasionally, extreme turbidity differences occur vertically where a highly turbid river enters a lake. This stratification is caused by temperature differences where the cooler, heavier river water flows beneath the warmer lake water. A temperature profile of the water column and visual observation of lake samples can detect these layers. Each layer of the stratified water column should be sampled.

The number of sampling sites on a lake, pond, or impoundment is determined by the objectives of the investigation dimensions of the basin. In small bodies of water, a single vertical composite at the deepest point may be sufficient. Dissolved oxygen, pH, temperature, etc., should be conducted on each vertical composite aliquot. In naturally formed ponds, the deepest point is usually near the center; in impoundments, the deepest point is usually near the dam.

In lakes and larger impoundments, several vertical sub-samples should be composited to form a single sample. These vertical sampling locations should be along a transection or grid. The field crew will determine the number of vertical composites and sampling depths for each area. In some cases, separate composites of epilimnetic and hypolimnetic zones may be required. Additional separate composite samples may be needed to adequately represent water quality in a lake possessing an irregular shape or numerous bays and coves. Additional samples should always be taken where discharges, tributaries, agriculture, and other such factors are suspected of influencing water quality.

When collecting sediment samples in lakes, pond, and reservoirs, the sample site should be as near as possible to the center of the water mass, especially for impoundments of rivers or streams. Generally, coarser grained sediments are deposited at the headwaters of a reservoir, and the finer sediments are near the center. The shape, inflow pattern, bathymetry, and circulation affect the location of sediment sampling sites in large bodies of water.

### 8.3.3 Control Sites

The collection of samples from control sites is necessary to compile a basis of comparison of water quality. A control site above the point of interest is as important as the sites below, and must be chosen with equal care. Two or three sites above the waste inflow may be necessary to establish the rate at which any unstable material is changing. The time of travel between the sites should be sufficient to permit accurate measurement of the change in the material under consideration.

## 8.4 SAMPLING EQUIPMENT AND TECHNIQUES

### 8.4.1 General

Any equipment or sampling techniques used to collect a sample must not alter the integrity of the sample and must be capable of providing a representative sample.

### 8.4.2 Water Sampling Equipment/Techniques

The physical location of the collector dictates the type of equipment needed to collect samples. Surface water samples may be collected directly into the sample container when possible. Pre-preserved sample containers shall never be used as intermediate collection containers. Samples collected in this manner use the methods specified in Section 7.6 of this manual. If wading into the stream is required, care should be taken not to disturb bottom deposits, which could be unintentionally collected, and bias the sample. Also, the sample should be collected directly into the sample bottle and **up current** of the wader. If wading is not possible or the sample must be collected from more than one depth, additional sampling equipment may be used. If sampling from a powerboat, samples must be collected upwind and upstream of the motor.

#### 8.4.2.1 Sampling Procedure Using a Teflon<sup>®</sup> or PVC Bailer

If data requirements of surface water sampling do not necessitate sampling from a strictly discrete interval of the water column, Teflon<sup>®</sup> or PVC constructed bailers can be used for sampling. The type bailer used is dependent on the analytical requirements. A closed top bailer utilizing a bottom check valve is sufficient for many surface water studies. Water is continually displaced through the bailer as it is lowered down through the water column until the specified depth is attained. At this point, the bailer is retrieved back to the surface. There is the possibility of



contamination to the bailer as it is lowered through the upper water layers. Also, this method may not be successful in situations where strong currents are found or where a discrete sample at a specified depth is needed.

If depth specific, discrete samples are needed and the parameters do not require Teflon<sup>®</sup> coated sampling equipment, a standard Kemmerer sampler may be used. A plastic bucket can also be used to collect surface samples if parameters to be analyzed do not preclude its use. The bucket shall always be rinsed twice with the sample water prior to collection and the rinse water be disposed of downstream from the sample collection point. All field equipment will be cleaned using standard cleaning procedures.

#### 8.4.2.2 Sampling Procedure Using a Kemmerer Sampler

Due to the PVC construction of the Kemmerer sampler, it shall not be used to collect samples for extractable organics, VOCs, and/or oil & grease analysis. The general collection procedure is as follows:

1. Securely attach a suitable line to the Kemmerer bottle.
2. Lock stoppers located at each end of the bottle on the open position. This allows the water to be drawn around the bottom end seal and into the cylinder at the specified depth.
3. The bottle is now in the set position. A separate "messenger" is required to activate the trip mechanism that releases the stopper and closes the bottle.
4. When the bottle is lowered to the desired depth, the messenger is dropped. This unlocks the trip mechanism and forces the closing of both end seals.
5. Raise the sampler, open one of the end seal, and carefully transfer the sample to the appropriate sample container.

#### 8.4.2.3 Sampling Procedures Using Sample Collection Containers

In most cases, sample collection containers are used to collect surface water from easily accessible sampling points. This means that the sample is collected manually, always upstream of the sampling person's position. An extension may be added to the container to make the sampling point more accessible for manual sampling. Extensions can be constructed of aluminum, PVC, steel, or any other suitable material. The sample container is normally attached to the extension using a clamp, vinyl pull ties, or duct tape. Samples collected in this way are done so in the following manner:

1. Place the inverted sample container into the water and lower to the desired depth. Never use a pre-preserved container as an intermediate sample collection device.
2. Re-invert the container with the mouth facing into the direction of flow and at the appropriate depth to collect the desired sample.
3. Carefully bring the container to the surface and transfer to the appropriate container.

#### 8.4.3 Sediment Sampling Equipment/Techniques

A variety of methods can be used to collect sediment samples from a streambed. ESC utilizes corers and scoops. Precautions must be taken to ensure that the sample collected is representative of the streambed. These methods are discussed in the following paragraphs.

##### 8.4.3.1 Sediment Core Samplers

Core sampling is used to collect vertical columns of sediment from the stream or lakebed. Many types of coring devices are available for use depending on the depth of water from which the sample is obtained, the type of bottom material, and the length of the core to be collected. Some devices are weight or gravity driven while others are simple hand push tubes. These devices minimize the loss of fine particles and should always be used when collecting sediment samples from flowing waters.

Coring devices are particularly useful in pollutant monitoring because the shock wave created by sampler descent is minimized and the fines at the sediment-water interface are only slightly disturbed. The sample can be withdrawn primarily intact removing only the layers of interest. Core liners manufactured of Teflon<sup>®</sup> or plastic can be purchased. These liners reduce the possibility of contamination and can be delivered to the laboratory in the tube they were collected in. Coring devices sample small surface areas and small sample sizes and often require repetitive sampling to obtain a sufficient amount of sample. This is the primary disadvantage to these devices but they are recommended in the sampling of sediments for trace organic compounds or metals analyses.

When sampling sediments in shallow water, the direct use of a core liner is recommended. Stainless steel push tubes are also used because they provide a better cutting edge and higher tensile strength than Teflon<sup>®</sup> or plastic. One advantage to using the Teflon<sup>®</sup> or plastic tubes is the elimination of possible metals contamination of the sample from the core barrels or cutting heads. The length of the corer tube should correspond to the desired depth of the layer being sampled. In general, soft sediments adhere better to the inside of the tube and a larger diameter tube can be used. Coarser sediments require the use of a smaller diameter tube of two inches or less to prevent the sample from falling out of the tube. The inside bottom wall of the tube can be filed down to allow easier entry into the substrate.

When samples are obtained by wading, caution should be used to minimize disturbance in the area sampled. Core tubes are pushed directly down into softer substrates until four inches or less of the tube is above the sediment-water interface. A slight rotation of the tube may be necessary to facilitate ease of entry into harder substrates and reduce compaction of the sample. The tube is then capped and slowly extracted and the bottom of the corer is capped before it is pulled above the water surface.

Sub-sampling is performed for VOC samples using an Encore™ type sampling device. This device is used to collect soil/sediment samples, while preventing container headspace. Once the core sample is collected, additional samples should be taken using an Encore™ type sampler, either 5g or 25g, capped, sealed, and immediately chilled to 4°C. The holding time for this sampling method is 48 hours. Alternatively, weigh 5g of sample into a pre-weighed vial (with a Teflon® lined screw cap) containing, 5mL sodium bisulfate solution and a magnetic stir bar, cap, and then ice to 4°C. The holding time for this method is 14 days.

#### 8.4.3.2 Scooping Samples

The easiest and quickest way to collect a sediment sample in shallow water is with a Teflon® coated scoop or stainless steel spoon. This type of sampling should be limited to quiescent (i.e., non-flowing) waters such as lakes or reservoirs.

#### 8.4.3.3 Mixing

As specified in Section 5.8, sediment samples, collected for chemical analysis, should be thoroughly mixed (except for volatile organic compounds analysis) before being placed in the sample containers.

## 8.5 SPECIAL SAMPLE COLLECTION TECHNIQUES

### 8.5.1 Trace Organic Compounds and Metals

Samples for trace pollutant analyses in surface water should be collected by dipping the sample containers directly into the water. Sometimes samples are split for enforcement or quality control purposes. A sufficient volume of sample for all containers should be collected in a large glass container and then, while mixing, be alternately dispensed into the appropriate bottles. This cannot be done for volatile organic compound samples due to potential loss of target analytes.

Only Teflon® or stainless steel should be used in sediment sampling for trace contaminant analyses. Teflon® coring tubes are the preferred technique.

### 8.5.2 Bacterial Analysis

Samples for bacteriological examination must be collected in sterilized bottles and protected against contamination. The preferred technique is to collect sample directly into the sample bottle. Hold the bottle near the base and plunge, neck downward, below the surface. The container is then turned with the neck pointed slightly upward and the mouth directed toward the current. The bottle is filled to about ½ inch from the top and recapped immediately. While the bottle is open, extreme care should be used to protect both the bottle and stopper against contamination. The ½ inch air space is left in the bottle to facilitate subsequent shaking in the laboratory.

If sampling with an intermediate sampling device (i.e. bailer), the device shall be thoroughly rinsed with sample water prior to collecting the sample. For this reason, microbiological samples are among the final samples collected from a sampling site. Begin pouring sample out of the sampling device before collecting into the sterilized container. Continue pouring sample out of the device, place the container under the flowing stream, and fill the container to ½ inch from the top. Flow should remain continuous before and during the filling process.

When sampling from a bridge, the sterilized sample bottle can be weighted and lowered to the water on a rope. Collectors must be careful not to dislodge debris from the bridge that could fall into the bottle.

## 8.6 AUXILIARY DATA COLLECTION

A field logbook is used to record data pertinent to sampling activities. This data describes all sampling locations and techniques, lists photographs taken, visual observations, etc. Visual observations of sample site conditions, including weather and overall stream conditions, recorded during the investigation can be valuable in interpreting water quality study results.

## 8.7 SPLIT AND DUPLICATE SAMPLE COLLECTION

Split samples measure variability between analysts, methods, and laboratories and are taken as subsamples from a single sample. This is unlike duplicate samples that measure variability inherent in the collection method or waste stream and are obtained in close succession during the same sampling event.

### 8.7.1 Split Sample Collection

Split samples are collected as follows:

1. Sample must be collected in a properly cleaned container constructed of acceptable materials. The volume should be more than twice the volume required for one sample.
2. Add appropriate preservative where required.
3. Mix thoroughly.
4. Alternately, decant sample into subsample containers in increments of approximately 10% of total subsample volume until containers are full.
5. Seal the sample containers with appropriate, airtight caps.
6. Label each sample container with a field number and complete a chain of custody.

**NOTE:** Volatile organic samples are not collected in this manner. Samples for VOC's must be collected as simultaneous, discrete grab samples.

### 8.7.2 Duplicate Sample Collection

1. Collect two samples in rapid succession.
2. Preserve where required.
3. Mix thoroughly.
4. Seal the sample containers with appropriate, airtight caps.
5. Label each sample container with a field number and complete a chain of custody.

## 9.0 GROUNDWATER AND DRINKING WATER SAMPLING

### 9.1 GROUNDWATER AND DRINKING WATER SAMPLING EQUIPMENT

Equipment type	Purpose	Component(s)	Allowable Parameter Groups
Bailers (disposable and non-disposable)	Purging	Teflon <sup>®</sup> & SS	All parameter groups
	Sampling	Teflon <sup>®</sup>	All parameter groups
Peristaltic Pump <sup>1</sup>	Purging <sup>2</sup>	Tygon Tubing	All parameter groups except organics
	Purging	Teflon <sup>®</sup>	All parameter groups
		Silastic Rubber	All parameter groups except organics
ISCO Bladder Pump <sup>3</sup>	Sampling	Stainless Steel, Teflon <sup>®</sup>	All parameter groups

<sup>1</sup> New or dedicated tubing must be used at individual monitoring well sites.

<sup>2</sup> If sample is not collected immediately after evacuation, tubing shall be withdrawn from the well prior to pump being turned off to prevent back flowing into the well.

<sup>3</sup> Pump will be cleaned after each use.

### 9.2 GENERAL GROUNDWATER SAMPLING

Groundwater sampling is necessary for a number of purposes. These include, but are not limited to, evaluating potable or industrial water sources, mapping contaminant plume movement at a land disposal or spill site, RCRA compliance monitoring (landfills), or examining a site where groundwater contamination may have or may be occurring.

Normally, groundwater is sampled from a permanent monitoring well. However, this does not exclude collection of samples from a sinkhole, pit, or other drilling or digging site where groundwater is present.

Monitoring wells are not always at the optimum. In these situations, additional wells may need to be drilled. Experienced, knowledgeable individuals (hydrologists, geologists) are needed to site the well and oversee its installation so that representative samples of groundwater can be collected.

ESC utilizes the procedures being reviewed in this section. Further guidance is available in the *RCRA Groundwater Monitoring Technical Enforcement Guidance Document (TEGD)*; ESC field personnel, at a minimum meet, and when possible exceed, the requirements of this document.

### 9.3 MEASUREMENT OF WELL WATER LEVEL AND STAGNANT WATER VOLUME CALCULATION

The sampling and analysis plan provides for measurement of standing water levels in each well prior to each sampling event. Field measurements include depth to standing water surface and total depth of the well. This data is then utilized to calculate the volume of stagnant water in the well and provide a check on the integrity of the well (e.g., silt buildup). The measurement should be taken to 0.01 foot when possible. A battery powered level sensor is used to measure depth to the surface of the groundwater. Equipment shall be constructed of inert materials and will be cleaned per sample equipment cleaning procedures prior to use at another well. Field data is recorded on the Monitoring Well Data Sheet (Figure 2).

#### 9.3.1 Procedure for Water Level Measurement

1. Clear debris from area around well or lay plastic sheathing around well pad.
2. Remove protective casing lid.
3. Open monitoring well lid.
4. Lower the clean water level indicator probe down into the well. A beep will sound upon contact with the water surface. False readings can be made from the wetted side of the well so it is necessary to check the level several times until a consistent reading is achieved. Record the distance (to the nearest 0.01 ft.) from the top of the well casing to the water surface on the Monitoring Well Data Sheet.
5. Continue to lower the probe until it reaches the well bottom. Record the distance (to the nearest 0.01 ft) from the top of the well casing to the bottom of the well on the Monitoring Well Data Sheet.
6. All water level and well depth measurements are made from the top of the well casing unless specified otherwise by the project manager or DER.
7. The wetted depth is obtained by subtracting total well depth from the surface level depth.

### 9.3.2 Calculating Water Volume

Total volume of standing water in a well is calculated by the following formula:

$$V = \pi r^2 h \times 7.48 \text{ gallons/ft}^3$$

where;

V	=	volume of standing water in the well (gallons)
r	=	radius of well (ft)
h	=	depth of water column in the well (ft)
$\pi$	=	3.14
7.48	=	conversion factor

## 9.4 WELL EVACUATION: WELLS WITHOUT IN-PLACE PLUMBING

Water standing in a well may not be representative of actual groundwater conditions. The standing water in a well should be removed to allow representative formation water to supplant the stagnant water. The evacuation method depends on the hydraulic characteristics of the well but the following general rules apply.

The total amount of water purged must be recorded. Therefore, the volume must be measured during the purging operation. This may be determined by:

1. Collecting the water in a graduated or known volume container (i.e., bucket);
2. Calculate the volume based on the pump rate; however pump rate may not be constant and field personnel should be aware of this;
3. Record the time that the actual purging begins in the field record.

Purging is considered complete if any one of the following criteria is satisfied:

1. Three well volumes are purged and field parameters (pH, temperature, conductivity) stabilize within 5% in consecutive readings at least 5 minutes apart. If field parameters have not stabilized after 5 well volumes, the purging is considered complete and sampling can begin.
2. Five well volumes are purged with no monitoring of field parameters.
3. At least one fully dry purge. A second dry purge may be necessary in some situations.

**FIGURE 2  
 MONITORING WELL DATA SHEET**

Site location:

ESC Project name/##: \_\_\_\_\_

Well Number	Depth to water surface (ft)	Depth to bottom of well (ft)	Length of water column (ft)	Volume of water evacuated (gal)	Time/date

Well Number	Temperature (°F)	pH (S.U.)	Conductivity (Tmho/cm)	Time/Date

Well casing material / diameter:

Sampled by / signature:

NOTES / CALCULATIONS:

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Except for low recovery wells, all wells are sampled within 6 hours of purging. Low recovery wells may be sampled as soon as sufficient sample matrix is available or up to 10 hours after purging. Wells that do not recover sufficiently within 10 hours should not be sampled.

Purging equipment includes Teflon<sup>®</sup> or stainless steel bailers or a peristaltic pump. Any fuel-powered pumping units are placed downwind of any sampling site. If purging equipment is reused, it is cleaned following standard procedures. Disposable latex gloves are worn by sampling personnel and changed prior to starting work at each sampling site.

If bailed water is determined to be hazardous, it should be disposed of in an appropriate manner.

The Florida Department of Environmental Regulation requires that during purging of the well, the purging device should be placed just below the surface of the water level and be lowered with the falling water level. For high yield wells, three casing volumes should be evacuated prior to collecting samples. Purging should be conducted at a rate to minimize agitation of the recharge water. Conductivity, pH, and temperature measurement during purging is necessary to monitor variability of the groundwater. **Samples should be collected within 6 hours of purging high yield wells.**

Low-yield wells (incapable of yielding three casing volumes) should be evacuated to dryness at a rate that does not cause turbulence. When the well recovers sufficiently, the first sample should be analyzed for pH, temperature, and conductivity. When recovery exceeds two hours, the sample should be collected as soon as sufficient volume is available. **If recovery is longer than 10 hours, the well should not be tested.** The project manager may wish to review available information to determine if obtaining a representative sample is possible.

#### 9.4.1 Procedure for Well Evacuation: Teflon<sup>®</sup> Bailer

1. Clear the area around the well pad; cover with plastic if necessary.
2. Slowly lower the bailer to the water surface and remove it when full.
3. Reel or pull bailer to the surface using caution to not allow the lanyard (cable or string) to touch the ground.
4. Use the bailer volume and number of bails removed to determine volume of water removed. Excess hazardous material should be poured into a container for later disposal.
5. Repeat steps 2 and 3 until 1.5 well volumes have been removed.
6. Begin monitoring for pH, temperature, and conductivity. Record values on the Monitoring Well Data Sheet. Discard the sample into the collection pail. Purge until the change between samples of each parameter is less than 5%.
7. Continue until at least three well volumes have been evacuated and the parameters pH, temperature, and conductivity are within 5 percent, or until a low yield well has been evacuated to dryness.
8. Record date and time the well was purged on the Monitoring Well Data Sheet.

**NOTE: For wells sampled in the State of Florida, three well volumes are purged prior to pH, temperature, and conductivity screening. Following evacuation of three well volumes, purge water is screened for these parameters at regular intervals until two consecutive measurements are within 5 percent. The intervals may be time-based (at least 5 min) or represent a portion of the well volume (at least 0.5 well volume).**

**Compliance with more stringent local, State, or Regional guidelines is observed where required.**

#### 9.4.2 Procedure for Well Evacuation: Peristaltic Pump

1. Clean area around the well pad.
2. Install the appropriate length of Tygon<sup>®</sup> or Teflon<sup>®</sup> tubing into the pump mechanism.
3. Insert the uncontaminated sampling end of the tubing into the well surface.
4. Connect the pump to the power supply.
5. Operate the pump at a flow rate that does not cause excessive agitation of the replacement water.
6. Determine the pump flow rate.
7. Purge until 1.5 well volumes have been evacuated.
8. Collect samples at a rate of one per well volume evacuated. Monitor these samples for pH, temperature, and conductivity. Record these measurements on the Monitoring Well Data Sheet. Monitor until the difference in each parameter is less than 5 percent.
9. Continue purging until three well volumes have been evacuated and the parameters pH, temperature, and conductivity are within 5 percent, or until a low yield well has been evacuated to dryness.
10. Record the date and time the well was purged on the Well Sampling Field Data Sheet.

## 9.5 PURGING TECHNIQUES: WELLS WITH IN-PLACE PLUMBING

### 9.5.1 General

The volume to be purged depends on whether the pumps are running continuously or intermittently and how close to the source samples can be collected. If storage/pressure tanks are present, a volume must be purged to totally exchange the volume of water in the tank.

### 9.5.2 Continuously Running Pumps

For continuously running pumps, the well should be purged by opening the valve and allowing it to flush for 15 minutes, if the well volume is unknown. If the sample is collected after a holding tank, the volume of the tank should also be purged.

### 9.5.3 Intermittently Running Pumps

Wells are purged at the maximum rate for at least 15 minutes. Monitoring of field parameters continues until two consecutive measurements within 5% are measured at 5-minute intervals.

## 9.6 SAMPLE WITHDRAWAL

Technique for withdrawal is dependent on the parameters to be analyzed. To collect a representative sample and minimize the possibility of sample contamination:

- Use Teflon<sup>®</sup> or stainless steel sampling devices when organics are an analyte of concern.
- Use dedicated tubing or samplers for each well. If a dedicated sampler is not available, clean the sampler between sampling events. Analyze equipment blanks to ensure cross-contamination has not occurred.

The preferred sample collection order is as follows (decreasing volatility):

1. Volatile organic compounds (VOCs)
2. Extractable Organics (includes Total Recoverable Petroleum Hydrocarbons [TRPH], Oil & Grease, Pesticides and Herbicides)
3. Total metals
4. Dissolved metals
5. Microbiological
6. Inorganics (includes Nutrients, demands, and Physical Properties)
7. Radionuclides

The following items are acceptable sampling devices for all parameters:

- A gas-operated, Teflon<sup>®</sup> or stainless steel squeeze pump (also referred to as a bladder pump with adjustable flow control) should be dedicated or completely cleaned between sampling events. If it is dedicated, the protocols on use, flow rates, and flow controls should be discussed.
- A Teflon<sup>®</sup> bailer with check valves and a bottom emptying device. Dedicated or disposable bailers should not be cleaned between purging and sampling operations.

ESC generally supplies sampling devices for wells sampled by ESC. However, some clients have wells equipped with dedicated sampling devices. All dedicated equipment is cleaned between sampling events with the exception of dedicated pump systems or dedicated pipes that are never removed. ESC evaluates the device and the project manager approves/disapproves of the dedicated device prior to sampling.

If sampling includes dissolved parameters, samples are filtered in the field in the following manner:

1. Use a one piece, molded, in-line high capacity disposable 1.0 micron filter when collecting samples for dissolved trace metals analysis. Use a 0.45 micron filter when sampling for all other (i.e., orthophosphorous, silica, etc.) dissolved parameters.
2. Filter material should be non-contaminating synthetic fibers.
3. Filter should be placed on the positive pressure side of the peristaltic pump.
4. If well is deeper than 25 feet; a submersible bladder pump may be necessary to bring the sample to the surface. Samples shall not be collected in an intermediate container.
5. At least one filtered equipment blank, using deionized water, must be collected and analyzed.
6. The sample is preserved as required following filtration.
7. Unfiltered samples are collected in conjunction with filtered samples.

**NOTE:** Filtered samples are collected only at the request of DER and will not be collected for turbid samples only.

#### 9.6.1 Sample Removal: With In-Place Plumbing

Samples should be collected following purging from a valve or tap as near to the well as possible, and ahead of all screens, aerators, filters, etc. Samples shall be collected directly into the sampling containers. Flow rate should not exceed 500 mL/min.

#### 9.6.2 Sample Removal: Without In-Place Plumbing

1. Following purging, collect the sample and pour it directly from the bailer into the sample container. If a peristaltic pump is used, pump the sample directly into the container. Collect the samples in order of decreasing volatility.
2. Measure the conductivity, pH, and temperature of the samples and record the results on the Monitoring Well Data Sheet.
3. If a bailer is not dedicated, clean field equipment using standard procedures. Collect blanks at a rate of one per type of equipment cleaned. If a piece of equipment is cleaned more than twenty times, collect blanks at a rate of 10 percent. An equipment blank must be taken and preserved for each analyte method group.
4. If a bailer is used to collect samples, replace the bailer string. Take precautions not to allow the string to touch the ground. Dispose of the used string properly. If Teflon<sup>®</sup> or stainless steel cable is used, clean according to standard procedures and do not let it touch the ground.
5. Replace the well cap and close and lock the protective casing lid.

## 9.7 SPLIT AND DUPLICATE SAMPLE COLLECTION

Split samples measure variability between analysts, methods, and laboratories and are taken as subsamples from a single sample. Duplicate samples measure variability inherent in the collection method or waste stream and are obtained in close succession during the same sampling event.

### 9.7.1 Split Sample Collection

1. Collect sufficient volume in a container constructed of appropriate materials. The volume should be more than twice the volume required for one sample.
2. Preserve as necessary.
3. Mix well.
4. Alternately decant 10% of the sample volume into each container and mix well.
5. Continue until each container is filled with an adequate sample volume.
6. Seal the containers, assign a field number, and complete the chain of custody.

### 9.7.2 Duplicate Sample Collection

1. Collect two samples in rapid succession into separate containers.
2. Preserve as necessary.
3. Mix well.
4. Seal the containers, assign a field number, and complete the chain of custody.

## 9.8 DRINKING WATER SAMPLING

### 9.8.1 General Concerns

Containers and preservatives must be selected prior to sampling.

- Containers and preservatives shall comply with Tables 1 and 2.
- It is recommended that the appropriate preservative be added to the container by the laboratory.

### 9.8.2 Sampling Drinking Water Wells

1. Purging and sampling should be from a spigot closest to the wellhead.
  - The spigot should be located before the holding tank and filters. If this is not possible, the holding tank must also be purged.
  - All aerators and filters should be removed if possible.
2. Depending on the running schedule of the well and the placement of the pressure tank, the system is purged as described in Section 9.5.
3. If volume of the pressure tank is not known, the well is purged for at least 15 minutes at maximum rate.
4. The flow is reduced to approximately 500 mL/minute.

5. Sample containers with no preservatives:
  - The interior of the cap or the container should not come in contact with anything.
  - The sample container is rinsed and the water is discarded.
  - Containers are not rinsed if collecting for oil and grease, total recoverable hydrocarbons, volatile organics (including trihalomethanes) or microbiologicals.
  - The container should be tilted to minimize agitation.
6. Sample containers with preservatives:
  - The above protocol is followed but **DO NOT** rinse the container.
  - The open end of the container should be held away from the face while filling.
  - The container should be gently tipped several times to mix the preservatives.
7. Place the bottle in a plastic bag and cool to 4°C.

#### 9.8.3 Sampling Drinking Water within a Facility/Residence for the Lead/Copper Rule

1. The appropriate sampling point depends on whether the sample is being taken to monitor compliance with Drinking Water Regulations for Lead and Copper. If so, the sample must be taken from a cold water tap in the kitchen or bathroom of residential housing or from an interior tap where water is used for consumption in a non-residential building.
2. Samples must be collected after the water has stood in the pipes for at least six hours.
3. THE SYSTEM SHOULD NOT BE FLUSHED.
4. The first flush should be collected immediately into the sample container. DO NOT RINSE THE CONTAINER PRIOR TO COLLECTING THE SAMPLE.
5. The container should be tilted to minimize agitation.
6. If the container contains preservative, hold the open end away from the face.
7. If the container does not contain preservative, add preservative as needed.
8. Replace cap and gently tip the container several times to mix the preservatives.
9. Place in a plastic sample bag.

#### 9.8.4 Sampling a Lead Service Line in a Facility/Residence for the Lead/Copper Rule

1. When sampling for compliance, the sampling point is normally designated by the permit or the municipality.

2. For Lead & Copper samples, each sample shall have stood in the line for at least six hours and shall be collected in one of the following ways:
  - a. At the tap, after flushing the volume of water between the tap and the lead service line. The volume of water shall be calculated based upon the inner diameter and length of the pipe between the tap and the service line.
  - b. By tapping directly into the service line.
  - c. In a single-family residence, allow the water to run until a significant temperature change indicates water standing in the service line is being sampled.
3. The flow shall be reduced to less than 500 mL/min before collecting samples.
4. Test for the presence of residual chlorine using residual chlorine indicator strips or a Hach DR-100 chlorine analyzer.
5. If residual chlorine is present and the parameter being analyzed requires removal of chlorine, collect the sample in the appropriate sample container(s) using the required preservatives.
  - a. Add 0.008% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> or 100mg of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> per 1L of sample water directly into the sample container.
  - b. After replacing the cap, tip the container several times to mix the preservative.

## 10.0 SOIL SAMPLING

Soil samples are preserved as per Section 14. When compositing subsamples, the quantity of each subsample used is measured and recorded in the field logbook.

### 10.1 SAMPLING EQUIPMENT

Type	Use	Materials	Allowable Parameter Groups <sup>1</sup>
Hand Auger (Bucket type)	Sampling	PVC	All parameter groups except VOC's, extractables and organics
Encore™ Sampler	VOC soil subsampling	Teflon®	VOC's only
Split Spoons	Sampling	Carbon Steel	All parameter groups
Trowel, Spatula	Sampling and Compositing*	Chrome-Plated Steel	All parameter groups
Spoons	Sampling and Compositing*	Stainless Steel	All parameter groups
Shovel	Sampling	Carbon Steel	All parameter groups
Mixing Pan	Compositing*	Pyrex & Aluminum	All parameter groups except metals in aluminum pan

- <sup>1</sup> Carbon steel & Chrome-plated steel tools may be used for collecting soils where trace metal concentrations are not a concern. When these tools are used, samples should be taken from soils not in contact with the tool surface.
- \* Compositing is not suitable for VOC's

## 10.2 HAND AUGER SAMPLING PROCEDURE

This procedure is used when only relatively shallow samples are required or when the use of heavy equipment is not practical. The hand auger may be used to collect samples of soils or other materials at various depths by adding extensions as necessary.

1. Remove surface debris from the location of the sampling hole using a clean shovel or spoon.
2. Disturbed portions of soil should be discarded and not used as part of the sample.
3. Using a clean auger, drill to the desired sample depth. Confirm depths using a tape measure or other appropriate device.
4. Use a clean planer auger to clean and level the bottom of the boring.
5. All grab samples should be mixed thoroughly prior to placement in containers (except VOCs).
6. Using a clean auger, extract the desired sample. Subsampling is performed for VOC sample collection using an Encore™ sampling device. Once the core sample is collected, additional samples should be taken using an Encore™ sampler, either 5g or 25g, capped, sealed, and immediately cooled to 4°C. The holding time for this method is 48 hours. Alternatively, weigh 5g of sample into a pre-weighed vial (with a Teflon® lined screw cap) containing 5mL sodium bisulfate solution and a magnetic stir bar, cap, and then ice to 4°C. The holding time for this method is 14 days.
7. If less than the collected volume of material is desired or if multiple containers are required, subsampling shall be conducted. The collected material shall be placed in a clean mixing pan and thoroughly mixed using a clean, stainless steel spoon. The mixed material will then be quartered, removed and recombined before samples are collected. For clay soils, representative aliquots of the entire sample should be removed from the auger using stainless steel spoons. Samples for chemical analyses shall not be collected from auger flights or cuttings from hollow stem auger flights. Samples used for vapor meter determinations will not be used for trace contaminant analyses.
8. Samples should then be labeled. The depth range from which the samples were taken should be included in the sample description.
9. Repeat steps (2) through (6) as necessary to obtain samples at all desired depths.
10. When preparing composite samples, the quantity of each subsample shall be measured and recorded in the field logbook.



### 10.3 SPLIT AND DUPLICATE SAMPLE COLLECTION

Split samples measure variability between analysts, methods, and laboratories and are taken as subsamples from a single sample. This is unlike duplicate samples that measure variability inherent in the collection method or waste stream and are obtained in close succession during the same sampling event. True split samples are difficult to collect for soils, sediment, and sludge under field conditions. Split samples for these materials are therefore considered duplicate samples.

The collection procedure is as follows:

1. Collect the appropriate volume of sample into a clean disk constructed of a non-reactive material.
2. Mix the material with a clean utensil and separate into 4 to 10 equal portions.
3. Alternate placing a portion of the subdivided material into each container.
4. Repeat until each container is filled.
5. Assign each container a field sample number and complete the chain of custody.

## 11.0 WASTE SAMPLING

### 11.1 SAMPLING EQUIPMENT

Type	Use	Materials	Allowable Parameter Groups <sup>1</sup>
Shovel	Sampling	Carbon Steel	All parameter groups except metals
Split Spoons	Sampling	Carbon Steel	All parameter groups except metals
Trowel, Spatula	Sampling and Compositing*	Stainless Steel	All parameter groups
Spoon	Sampling and Compositing*	Stainless Steel	All parameter groups
Drum Pump	Sampling	Polypropylene	All parameter groups
Mixing pan	Compositing*	Pyrex or aluminum	All parameter groups except metals in aluminum pan
Coliwasa	Sampling	Glass	All parameter groups

<sup>1</sup>Carbon steel tools may be used for collecting wastes when trace metal concentrations are not a concern.

\*Compositing is not suitable for VOC's

### 11.2 GENERAL

This section discusses the collection of samples from drums, tank trucks, and storage tanks, and samples from waste piles and landfills. All ESC personnel consider sampling from closed containers as a hazardous operation.

#### 11.2.1 Specific Quality Control Procedures for Sampling Equipment

Sampling equipment used during waste sampling must be cleaned as specified in Section 12 of this manual before being returned from the field to minimize contamination.

Contaminated disposable equipment must be disposed of as specified in the sampling plan.

All field equipment is cleaned and repaired before being stored at the conclusion of a field study. Special decontamination procedures may be necessary in some instances and is developed on a case-by-case basis. Any deviation from standard cleaning procedures and all field repairs is documented in field logbooks. Equipment that has not been properly cleaned must be tagged and labeled.

#### 11.2.2 Collection of Supplementary Information

The collection of supplementary data is important when collecting waste samples. Any field analyses are recorded in field logbooks. Sketches of sampling locations and layout are documented in the logbooks. Photographs are used extensively.

### 11.3 OPEN AND CLOSED CONTAINER SAMPLING

#### 11.3.1 General

When sampling containers, open containers should be sampled first since they generally present less of a hazard. Closed containers must be considered as extremely hazardous. Due to the dangers involved with container sampling, the sampling of drums or other containers containing either unknown materials or known hazardous materials are considered a hazardous duty assignment.

One problem with container sampling is stratification and/or phase separation. Care must be taken to ensure that the sample collected is representative. If only one layer or phase is sampled, this should be noted when interpreting analytical results.

If no stratification is present, representative samples may be composited by depth. When a drum or cylindrical container is standing vertically, depth compositing provides a good quantitative estimate of the containers contents. In other cases where containers are tipped, horizontal, deformed, etc., and stratification may not be present, vertical compositing provides at least a qualitative sample.

#### 11.3.2 Sampling Equipment

The following equipment is available for use in collecting waste samples: barrel bung wrenches, adjustable wrenches, etc.; coliwasa samplers for drum sampling; and peristaltic pumps for liquid waste sampling from containers.

### 11.3.3 Sampling Techniques

Containers containing unknown materials or known hazardous materials are opened using only spark proof opening devices from a grounded container.

The coliwasa sampler is a single use glass sampler, consisting of an outer glass tube with one end tapered and a separate inner glass tube with a small bulb on one end. The outer tube is slowly lowered into the drum, tapered end first. Slowly lowering the tube allows the liquid phases in the drum to remain in equilibrium. The inner glass tube is inserted into the outer tube. After both inner and outer tubes are inserted into the drum to be sampled, the inner tube bulb end is pressed gently against the tapered end of the outer tube, forming a seal. Both tubes are withdrawn from the drum and the ends of the tubes are held over the sample container.

Drum samples can also be collected using a length of glass tube (1/2-inch or less inside diameter). The tube is inserted into the drum as far as possible and the open end is sealed to hold the sample in the tube. The sample is then placed in the appropriate container. Sample volumes are the absolute minimum required.

Tank truck and storage tank samples may be collected from access ports on top of these tanks or trucks using the above techniques. Tank trucks are often compartmentalized, and each compartment should be sampled. Sampling from discharge valves is not recommended due to stratification possibilities and possibilities of sticking or broken valves. If the investigator must sample from a discharge valve, the valving arrangement of the particular tank truck being sampled must be clearly understood to ensure that the contents of the compartments of interest are sampled. The investigator must realize that samples obtained from valves may not be representative.

If stratification or phase separation of waste samples is suspected, the sample collected should be representative of container contents. Samples should be depth composited when possible and number and types of layers shall be noted when interpreting analytical results.

## 11.4 WASTE PILES AND LANDFILLS

### 11.4.1 General

Waste piles consist of sludge and other solid waste, liquid waste mixed with soil, slag, or any type of waste mixed with construction debris, household garbage, etc. The sampling personnel must be aware that landfills were not and are often still not selective in the types of materials accepted. Sampling at landfills could involve sampling operations that are potentially dangerous to sampling personnel.

#### 11.4.2 Sampling Locations

Sampling locations should be selected that yield a representative sample of the waste. Exceptions are situations in which representative samples cannot be collected safely or when the team is purposely determining worst-case scenarios.

##### 11.4.2.1 Waste Piles

A representative sample from a small waste pile can be obtained by collecting a single sample. Collecting representative samples from large waste piles requires a statistical approach in selecting both the numbers of samples and sample location. A discussion of statistical methods is outlined in the *Test Methods for Evaluating Solid Waste (SW-846)* issued by the EPA Office of Solid Waste and Emergency Response.

##### 11.4.2.2 Landfills

Representative samples from landfills are difficult to achieve to due to the heterogeneous nature of the wastes. A statistical approach should be used in selecting both the number of samples and the sample location. Statistical methods are given in *Test Methods for Evaluating Solid Waste (SW-846)* issued by the EPA Office of Solid Waste and Emergency Response. Landfills often generate leachate at one or more locations downgradient of the fill material that can provide some insight into the materials contained in a landfill that are migrating via groundwater.

#### 11.4.3 Sampling Techniques

All samples collected should be placed into a Pyrex<sup>®</sup> or aluminum mixing pan and mixed thoroughly. Samples for volatile organic compounds analyses must not be mixed or composited. Stainless steel spoons or scoops should be used to clear away surface materials before samples are collected. Near surface samples can then be collected with a clean stainless steel spoon. Depth samples can be collected by digging to the desired depth with a carbon steel shovel or scoop and removing the sample with a stainless steel spoon.

## 12.0 STANDARD CLEANING PROCEDURES

### 12.1 GENERAL

#### 12.1.1 Introduction

ESC personnel use the procedures outlined in this section to clean field equipment prior to use. Ideally, a sufficient amount of clean equipment is carried to the field so that the project can be conducted without the need for field cleaning. This is not always the case. ESC's policy regarding cleaning field equipment is as follows:

1. Equipment used in the field must be thoroughly cleaned in a controlled environment using prescribed procedures. This minimizes the potential for contaminants being transferred to equipment, vehicles, and the laboratory.
2. All equipment is rinsed immediately with tap water after use, even if it is to be field cleaned for other sites.
3. If equipment is used only once (i.e., not cleaned in the field), it is labeled as “dirty” or “contaminated equipment” in the field and transported separately from clean equipment.
4. All cleaning procedures are documented. Field decontamination is documented in the field records. These records specify the type of equipment cleaned and the specific protocols that are used. In-house cleaning records must identify the type of equipment, date it was cleaned, SOP used, and person that cleaned it.
5. Unless justified through documentation (i.e., company written protocols and analytical records) and historic data (i.e., absence of analytes of interest in equipment blanks), the protocols in Sections 12.1.2 through 12.7.11 are followed without modification.
6. All field sampling equipment is pre-cleaned in-house.

#### 12.1.2 Cleaning Materials

Use a phosphate-free, laboratory detergent such as Liquinox<sup>®</sup>. The use of any other detergent is noted in field logbooks and summary reports.

Ten percent nitric acid solution is made from reagent-grade nitric acid and deionized water.

The standard cleaning solvent used is pesticide-grade isopropanol. Other solvents (acetone and/or hexane) may be substituted as necessary. The use of other solvents must be documented in field logbooks and summary reports.

Tap water may be used from any potable water system. Untreated water is not an acceptable substitute for tap water.

Deionized water is tap water that has been passed through a deionizing resin column and should contain no inorganic compounds at or above analytical detection limits. Organic-free water is tap water that has been de-ionized and treated with activated carbon. Organic-free water should contain no detectable levels of organic compounds, and less than 5 ug/L of VOCs.

Analyte-free water is water in which all the analytes of interest and all interferences are below the method detection limits. Analyte-free water is always used for blank preparation and for the final in-house decontamination rinse.

Substitution of a higher grade water (i.e., deionized or organic-free water for tap water) is permitted and need not be recorded. Solvent, nitric acid, detergent, and rinse water used to clean equipment shall not be reused.

#### 12.1.3 Marking Clean Equipment

Equipment that is cleaned by these methods is marked with the date and time that the equipment was cleaned.

#### 12.1.4 Marking Contaminated or Damaged Field Equipment

Field equipment that needs repair is tagged and repairs or symptoms noted on the tag. Field equipment that needs cleaning **will not** be stored with clean equipment. All wrapped equipment not used in the field may be placed back in stock after equipment is inspected to ensure that contamination has not taken place.

#### 12.1.5 Decontamination of Equipment Used With Toxic or Hazardous Waste

Equipment used to collect hazardous or toxic wastes or materials from hazardous waste sites, RCRA facilities, or in-process waste streams is decontaminated prior to leaving the site. This decontamination procedure consists of washing with laboratory detergent and rinsing with tap water. More stringent procedures may be required depending on the waste sampled.

If equipment is heavily contaminated, an acetone or acetone/hexane/acetone pre-rinse may be necessary prior to regular decontamination procedures. It is not recommended that this type of cleaning be performed in the field.

#### 12.1.6 Disposal of Cleaning Materials

See Section 16.

#### 12.1.7 Safety Procedures for Cleaning Operations

All applicable safety procedures are followed during cleaning operations. The following precautions are taken during cleaning operations:

- Safety glasses or goggles, gloves, and protective clothing are worn during all cleaning operations.
- Solvent rinsing operations are conducted under a hood or in an open, well ventilated area.
- No eating, smoking, drinking, chewing, or hand to mouth contact is permitted during cleaning operations.

#### 12.1.8 Storage of Field Equipment

All clean field equipment is stored in a designated, contaminant-free area.

## 12.2 QUALITY CONTROL PROCEDURES FOR CLEANING

### 12.2.1 General

This section establishes quality control methods to monitor the effectiveness of the equipment cleaning procedures. The results of these methods are monitored by the ESC Quality Assurance Department. All quality control procedures are recorded in a logbook and maintained in a quality assurance file. If contamination problems are detected, the ESC QA Department determines the cause(s) of the problem(s) and takes immediate corrective action.

### 12.2.2 Rinse Water

The quality of water used is monitored once per quarter by placing water in standard, pre-cleaned sample containers and submitting them to the ESC laboratory for analysis. Organic-free water is also submitted for analyses of the various organic compounds.

## 12.3 PROCEDURES FOR CLEANING TEFLON<sup>®</sup> OR GLASS EQUIPMENT USED IN THE COLLECTION OF SAMPLES FOR TRACE ORGANIC COMPOUNDS AND/OR METALS ANALYSES

1. Equipment is washed with laboratory detergent and hot water using a brush to remove any particulate matter or surface film. If oil, grease, or other hard to remove residues are present on the equipment, an acetone/hexane/acetone pre-wash and/or steam cleaning may be necessary.
2. Rinse the equipment with hot tap water.
3. Rinse or soak, if necessary, equipment with a 10% nitric acid solution. If nitrogen-containing compounds are analytes of concern, hydrochloric acid must be used as a substitute or subsequent equipment rinse.
4. Rinse equipment with tap water.
5. Rinse equipment with deionized water.
6. Rinse equipment twice with solvent and allow to dry.
7. If equipment cannot be cleaned effectively, discard properly.
8. Wrap equipment in aluminum foil. Seal in plastic and date.

## 12.4 PROCEDURES FOR CLEANING STAINLESS STEEL OR METAL SAMPLING EQUIPMENT USED IN TRACE ORGANIC AND/OR METALS SAMPLE COLLECTION

1. Equipment is washed with laboratory detergent and hot water using a brush to remove any particulate matter or surface film. If oil, grease, or other hard to remove materials are present, a acetone/hexane/acetone pre-wash and/or steam cleaning may be necessary.

2. Rinse equipment with hot tap water.
3. Rinse equipment with deionized water.
4. Rinse equipment twice with solvent and allow to dry.
5. If equipment cannot be cleaned effectively, discard properly.
6. Wrap equipment in aluminum foil. Seal in plastic and date.

## 12.5 CLEANING PROCEDURES FOR AUTOMATIC SAMPLING EQUIPMENT

### 12.5.1 General

All automatic wastewater samplers are cleaned as follows:

- The exterior and accessible interior portions of automatic samplers is washed with Liquinox and rinsed with tap water.
- The electronics casing are cleaned with a clean damp cloth.
- All vinyl sample tubing is discarded after each use.
- Teflon<sup>®</sup> tubing is cleaned using procedures found in Section 12.6.2.
- Silastic pump tubing is cleaned after each use, if possible. Tubing is cleaned using cleaning procedures specified in Section 12.6.1 of this document. Tubing is checked on a regular basis and will be changed if it has become discolored or loses elasticity.

### 12.5.2 Reusable Glass Composite Sample Containers

1. If containers are used to collect samples that contain hard to remove materials (i.e., oil and grease) it is rinsed as necessary with reagent grade acetone prior to the detergent wash. If material cannot be removed, the container is discarded.
2. Wash containers thoroughly with hot tap water and Liquinox and rinse thoroughly with hot tap water.
3. If metals are to be sampled, rinse with 10% nitric acid. If nutrients are to be sampled, follow with a 10% hydrochloric acid rinse.
4. Rinse thoroughly with tap water.
5. Rinse thoroughly with DI water.
6. If organics are to be sampled, rinse twice with isopropanol and allow to air dry for 24 hours or more. Cap the container with the decontaminated Teflon<sup>®</sup> lined lid.
7. After use, rinse with tap water in the field and cover to prevent drying of material onto the interior surface.
8. Containers that have a visible scale, film, or discoloration after cleaning or were used at a chemical manufacturing facility should be properly discarded at the conclusion of the sampling activities.



### 12.5.3 Reusable Plastic Composite Sample Containers

1. Wash containers with hot tap water and laboratory detergent using a bottlebrush to remove particulate matter and surface film.
2. Rinse containers with hot tap water.
3. Rinse containers with 10% nitric acid. If nitrogen containing compounds are analytes of concern, hydrochloric acid must be used as a substitute or subsequent equipment rinse.
4. Rinse containers with tap water.
5. Rinse containers with deionized water.
6. Cap with aluminum foil.
7. Plastic sample containers used at facilities that produce toxic compounds will be properly disposed of at the conclusion of the sampling activities. Containers that have a visible film, scale, or other discoloration remaining after cleaning will be discarded.

### 12.5.4 Plastic Sequential Sample Bottles for Automatic Sampler Base

1. Rinse bottles in field with potable or de-ionized water when possible.
2. Wash in dishwasher at wash cycle, using laboratory detergent cycle, followed by tap and deionized water rinse cycles. Alternatively, handwash using the same procedure.
3. Rinse with 10% nitric acid. If nitrogen containing compounds are analytes of concern, hydrochloric acid must be used as a substitute or subsequent equipment rinse.
4. Rinse with tap water.
5. Replace bottles in sampler base; cover with aluminum foil before storing.

## 12.6 CLEANING PROCEDURES FOR SAMPLING TUBING

### 12.6.1 Silastic Rubber Pump Tubing Used In Automatic Samplers

Silastic pump tubing used in automatic samplers need not be replaced in pumps where the sample does not contact the tubing, where the sampler is being used solely for purging purposes (i.e., not being used to collect samples). Tubing must be changed on a regular basis, if used for sampling purposes, and should be cleaned in this manner:

1. Flush tubing with laboratory grade detergent and hot tap water
2. Rinse thoroughly with hot tap water
3. Rinse thoroughly with DI water
4. If used to collect metals samples, the tubing is flushed with 1+5 nitric acid, followed by a thorough rinsing with DI water
5. Install the tubing in the automatic wastewater sampler
6. Cap both ends with aluminum foil or equivalent

Tubing should always be replaced at automatic sampler manufacturer's recommended frequencies. If tubing cannot be adequately cleaned, it is discarded.

#### 12.6.2 Teflon<sup>®</sup> Tubing

New Teflon<sup>®</sup> tubing is pre-cleaned as follows:

1. Rinse outside of the tubing with pesticide-grade solvent.
2. Flush interior of the tubing with pesticide-grade solvent.
3. Let dry overnight in drying oven or equivalent.
4. Wrap tubing in aluminum foil and seal in plastic.

Reused tubing is transported to the field in pre-cut and pre-cleaned sections. Field cleaning of Teflon<sup>®</sup> is not recommended. The following steps describe in-house cleaning procedures:

1. Exterior of tubing must be cleaned first by soaking in hot, soapy water in a stainless steel or non-contaminating sink. Particulate may be removed with a brush.
2. Clean inside of tubing ends with a small bottlebrush.
3. Rinse surfaces and ends with tap water.
4. Rinse surfaces and ends with nitric acid, tap water, isopropanol, and analyte-free water.
5. Place on fresh aluminum foil, connect all sections with Teflon<sup>®</sup> couplings.
6. Cleaning configuration:
  - a. Cleaning solutions are placed in a clean, 2-liter glass jar.
  - b. Place one end of tubing in the solution, the other in the **INFLUENT** end of a peristaltic pump.
  - c. Effluent from the pump can be recycled through the glass cleaning solution jar. All cleaning solutions can be recycled EXCEPT the final isopropanol and analyte-free water rinses.
7. The above configuration is used as follows:
  - a. Pump generous amounts of hot, soapy water through the tubing.
  - b. Follow this with tap water, 10% nitric acid, tap water, isopropanol, and analyte-free water.
  - c. The nitric acid and isopropanol rinses should be allowed to remain in the tubing for 15 minutes with the pump shut off then continue with subsequent rinses
  - d. Leave any couplings in and connect or cover the remaining ends.
8. After cleaning the interior, rinse the exterior with analyte-free water.
9. The cleaned lengths are wrapped in aluminum foil and stored in a clean, dry area until use.

## 12.7 FIELD EQUIPMENT CLEANING PROCEDURES

### 12.7.1 General

It is the responsibility of field personnel to properly clean equipment in the field. The following procedures are observed when cleaning equipment in the field.

### 12.7.2 Conventional Equipment Use

Remove deposits with a brush if necessary. If only inorganic anions are of interest, equipment should be rinsed with analyte-free water and with the sample at the next sampling location prior to collection. Clean equipment for the collection of samples for organic compounds or trace inorganic analyses according to Section 12.7.3.

### 12.7.3 Equipment Used to Collect Organic Compounds and Trace Metals Samples

1. Clean with tap water and laboratory detergent. If necessary, use a brush to remove particulate and surface films then rinse with tap water.
2. Rinse with 10 to 15% nitric acid solution followed by 10% hydrochloric acid rinse (unless equipment is made of metal) followed by tap water and DI water.
3. Rinse twice with solvent.
4. Rinse with organic-free water and allow to air dry.
5. If organic-free water is unavailable, let air dry. Do not rinse with deionized or distilled water.
6. Wrap with aluminum foil or plastic.

### 12.7.4 Teflon<sup>®</sup>, Glass, Stainless Steel or Metal Equipment Used to Collect Samples for Metal Analyses

1. Remove particulate matter and surface films. Clean with laboratory detergent and tap water.
2. Rinse with tap water.
3. Ten percent nitric acid solution (skip 3 and 4 if equipment is made of metal and/or stainless steel).
4. Rinse with tap water.
5. Rinse with deionized water then let air dry.

### 12.7.5 Instruments Used to Measure Groundwater Levels

1. Wash with laboratory detergent and tap water.
2. Rinse with tap water.
3. Rinse with deionized water.
4. Allow to dry.

#### 12.7.6 Field Filtration Apparatus

1. A new, disposable filtration unit will be used for each site. Filter pore size is dependent on parameter being monitored as per Section 9.6.
2. The peristaltic pump is cleaned as described in Section 12.7.7.
3. Silastic pump tubing is cleaned as described in Section 12.6.1.
4. If Teflon<sup>®</sup> tubing is used, it is cleaned as described in Section 12.6.2.
5. Other tubing types must be cleaned following the appropriate regimen described in Section 12.6. In general, non-Teflon<sup>®</sup> type tubing (e.g., HDPE) will not be re-used.

#### 12.7.7 Flow Meters, Above Ground Pumps, Bladder Pumps and Other Field Instrumentation

The exterior of equipment such as flow meters should be washed with a mild detergent and rinsed with tap water before storage. The interior of such equipment may be wiped with a damp cloth.

Other field instrumentation should be wiped with a clean, damp cloth. Meter probes should be rinsed with deionized water before storage.

Equipment desiccant should be checked and replaced as necessary.

Peristaltic pumps used for purging must be free of oil and grease on the exterior. They must be cleaned on the outside with Liquinox and rinsed with tap water followed by DI water.

#### 12.7.8 In-Field Decontamination For Submersible Purging Pump and Tubing

ESC uses the submersible bladder pump listed in Section 9.1 only for purging and not for sample collection. The pump and tubing is decontaminated between wells in the following manner:

1. Interior of the pump and tubing is thoroughly flushed with a soapy water solution.
2. Wipe or scrub the exterior of the pump and tubing as necessary with the appropriate soap solution.
3. Rinse exterior and interior of pump and tubing thoroughly with tap water followed by a deionized water rinse.
4. Allow remaining water to drain from tubing and pump and allow to air dry as long as possible in a contaminant free area before purging the next well.

#### 12.7.9 Shipping Containers

All reusable shipping containers are washed with laboratory detergent, rinsed with tap water, and air dried before storage or re-use. Extremely contaminated shipping containers are cleaned as thoroughly as possible and properly disposed.

#### 12.7.10 Analyte Free Water Containers

Analyte-free water containers can be made of glass, Teflon<sup>®</sup>, polypropylene, or high density polyethylene (HDPE). Inert glass or Teflon<sup>®</sup> are recommended for holding organic-free sources of water. Polypropylene can be used when organics are not analytes of concern. HDPE is not normally recommended but is acceptable for use. Water should not be stored in these containers for extended periods. Containers of water should only be used for a single event and should be disposed of at the end of the sampling day. The procedure for cleaning analyte-free water containers is as follows:

1. For new containers, follow instructions in Section 12.3 of this manual. Delete the solvent rinse if containers are made of plastic.
2. Cap with Teflon<sup>®</sup> film, aluminum foil, or the Teflon<sup>®</sup> lined bottle cap (aluminum foil or Teflon<sup>®</sup> film may also be used as a cap liner).

If water is being stored in reused containers, the following cleaning procedures should be followed:

1. After emptying, cap the container.
2. Wash exterior of the container with Liquinox and rinse with DI water.
3. Rinse the interior twice with isopropanol unless the container is made of plastic.
4. Rinse the interior thoroughly with analyte-free water.
5. Invert and allow to dry.
6. Fill the container with analyte-free water and cap with aluminum foil, Teflon<sup>®</sup> film, or a Teflon<sup>®</sup> lined bottle cap.
7. Water is not stored prior to a sampling event for more than 3 days.

#### 12.7.11 Vehicles

Field vehicles used by ESC personnel should be washed at the conclusion of each sampling event. This should reduce the risk of contamination due to transport on a vehicle. When vehicles are used at hazardous waste sites or on studies where pesticides, herbicides, organic compounds, or other toxic materials are known or suspected to be present, a thorough interior and exterior cleaning is mandatory at the conclusion of the site visit.

Vehicles are equipped with trash containers. ESC personnel are responsible for cleanliness of each vehicle.

### **13.0 SAMPLE HISTORY**

Sample chronology is recorded and kept on the ESC chain of custody, field logbooks and laboratory notebooks. These are discussed in detail in Section 9.0.

### **14.0 SAMPLE CONTAINERS, PRESERVATION METHODS AND HOLDING TIMES**

#### **14.1 GENERAL CONSIDERATIONS**

The following section contains information regarding sample containers, preservation methods, and holding times. Refer to SW-846, Table II-1 and Chapter 3, Page 3 for solid waste and RCRA projects and 40 CFR Part 136, Table II for water and wastewater projects.

The provisions of 40 CFR Part 136, Table II take precedence over requirements given in any approved method when sampling in the State of Florida for water and wastewater.

Proper sample preservation is the responsibility of the sampling team and it is their responsibility to assure that all samples are preserved according to 40 CFR Part 136. For the purposes of this manual, "immediately" is defined as within 15 minutes.

Sample preservation is accomplished either by obtaining pre-preserved containers from an acceptable source or by adding preservatives in the field.

It is the responsibility of the field team accepting pre-preserved containers to make sure that the proper preservatives are used and desired results are achieved. The laboratory also supplies additional preservatives from the same source in suitable containers.

#### **14.2 SAMPLE PRESERVATION**

The following protocols apply for sample containers preserved in the field after the sample has been added:

1. Preservatives are at least reagent grade or higher. The acid for metals is suitable for trace metals analyses.
2. Fresh preservatives are obtained prior to each sampling event. Remaining preservatives that are not sealed must be discarded in an acceptable manner.
3. Preservatives are transported in pre-measured glass ampules and added directly to the sample.
4. A corresponding amount of preservative is added to the associated equipment blanks.
5. The pH is checked on all pH preserved samples with the exception of VOC, oil and grease, and TRPH.

Effectiveness of pH adjustment is made in the following manner:

1. Narrow range pH paper is used to test a small aliquot of the preserved sample.
2. A small portion of sample is placed into a container, checked with pH paper, and compared against the color chart.
3. Discard the aliquot properly, but do not pour back into the sample container.
4. If pH is acceptable, document in field log and prepare for transport to laboratory.

If pH is unacceptable, continue to add additional preservative in measured increments using the methods described above until an acceptable pH has been reached. Record the total amount of preservative used in the field log. Always use additional preservative from the same source as the initial preservation attempt.

In some cases, an extra dummy sample can be used to test pH preservation. Content should be suitably discarded.

If equipment blanks or field blanks are used, the maximum amount of preservative that was used to preserve any single sample in the set is added to the equipment or field blank.

Samples requiring temperature preservation are cooled to 4°C. The cooler will be checked to ensure that the ice has not melted.

### **14.3 SAMPLE CONTAINERS**

ESC does not clean and re-use sample containers. ESC purchases all sample collection containers precleaned. All used sampling containers are discarded after use. The cleaning criteria of all containers must meet EPA analyte specific requirements.

QEC provides written certification that containers do not contain analytes of concern above method detection levels

ESC maintains records for these containers (lot numbers, certification statements, date of receipt, etc.) and intended uses are documented.

### **14.4 FIELD REAGENT HANDLING**

Reagents, cleaning materials, and preservatives that are maintained by a field team will be stored, transported, and handled in such a way as to prevent and/or minimize contamination. The following storage and use protocols will be observed:

1. Chemicals are stored in-house and transported to the field segregated by reactivity.
2. Acids are stored in an acid storage cabinet and solvents are stored in a vented, explosion proof solvent storage cabinet.
3. All chemicals transported to the field are stored in bottles and packed to avoid breaks.

4. When reagents are transferred from an original container, the transport container must be pre-cleaned and of compatible material as the original container.
5. Chemicals are separated from sample containers and samples to avoid reaction and possible contamination.
6. Analyte free water is segregated from solvents to prevent contamination.

#### 14.4.1 Reagent and Standard Storage

Chemical	Method of Storage
Nitric acid	Stored separated from other acids in original container in vented cabinet.
Sulfuric acid	See above
Hydrochloric acid	See above
Isopropanol	Stored in original glass container in vented and explosion proof solvent storage cabinet.
pH calibration buffers, turbidity standards, conductivity standards	Stored in cabinet designated for standard and reagent storage. Stored in temperature-controlled area of laboratory.
Sodium hydroxide	Stored in original container in designated cabinet in laboratory.
Sodium thiosulfate, zinc acetate, ascorbic acid, lead acetate	Stored in original containers in designated area of laboratory. Reagent solutions made fresh prior to use.

## 14.5 SAMPLE TRANSPORT

In the majority of situations, samples are delivered directly to the laboratory by the field sampling team or field courier following standard chain of custody protocols. Samples are preserved immediately (i.e., within 15 minutes) and packed with ice prior to transport. The field team relinquishes custody to the login sample custodian upon arrival at the laboratory.

Certain situations require that the field sampling team ship samples to the laboratory utilizing common carrier (UPS, FEDEX, etc.). If samples are sent by common carrier, all documentation (transmittal form, chain of custody, field data, analyses request, etc.) is placed in a ziplock bag and placed inside the sample container. The container is then sealed closed and sent to the laboratory in the required time frame to meet requirements of time-sensitive analyses.

## 14.6 BIOMONITORING SAMPLING

### Preservation and Sample Volume

Aqueous samples collected for Bioassay can be collected in either glass or HDPE plastic. There is no required chemical preservation for this type of sample but the sample must be kept at  $4 \pm 2^{\circ}\text{C}$ . The required volume varies independently with each type of analysis but the minimum collected is 250mL. The samples can be held for a maximum of 36 hours from the time of collection until first use.



Sample Collection

Grab sample protocols are utilized for acute bioassay unless otherwise specified in permit requirements. Composite sampling protocols are utilized for chronic bioassays unless otherwise specified in permit requirements. (Actual sampling protocols are discussed in detail throughout this appendix) ESC field collection personnel are required to collect all bioassay samples by completely filling the sample bottle and leaving no headspace. It is important that bottles be filled completely to reduce possible aeration that may reduce the toxic properties of the sample. If a client chooses to collect the samples, a trained ESC field collection person can explain in detail the importance of reducing aeration by filling the sample bottle completely.

14.6.1 Biomonitoring Sampling Containers

All bioassay glassware are cleaned using the following EPA protocol:

- soak for 15 minutes in hot tap water with detergent and scrub
- rinse thoroughly with hot tap water
- rinse thoroughly with dilute nitric acid (10%)
- rinse thoroughly with deionized water
- rinse thoroughly with pesticide grade acetone

**TABLE 14.6: PRESERVATION, HOLDING TIME AND SAMPLE CONTAINERS**

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
<b>AIR METHODS</b>									
Volatiles in Ambient Air	Air	EPA TO-15	NA	Various	Canister	None	Ambient	14	Days
Volatiles in Ambient Air	Air	EPA TO-15	NA	Various	Tedlar	None	Ambient	5	Days
Volatiles in Ambient Air	Air	EPA Method 18	NA	Various	Canister	None	Ambient	14	Days
Volatiles in Ambient Air	Air	EPA Method 18	NA	Various	Tedlar	None	Ambient	5	Days
Ohio VAP EPA Method 8260B	Air	NA	EPA 8260B	Various	Canister	None	Ambient	14	Days
Ohio VAP EPA Method 8260B	Air	NA	EPA 8260B	Various	Tedlar	None	Ambient	5	Days
Methane, Ethane, Ethene, Propane	Air	RSK-175	NA	Various	Canister	None	Ambient	14	Days
Methane, Ethane, Ethene, Propane	Air	RSK-175	NA	Various	Tedlar	None	Ambient	5	Days
Fixed Gases - C <sub>2</sub> , CO <sub>2</sub> , CO, and CH <sub>4</sub>	Air	ASTM D1946/D5314	NA	Various	Canister	None	Ambient	14	Days
Fixed Gases - C <sub>2</sub> , CO <sub>2</sub> , CO, and CH <sub>4</sub>	Air	ASTM D1946/D5314	NA	Various	Tedlar	None	Ambient	5	Days

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Arizona State Specific VOCs in Vapor - 8260B	Air	NA	EPA 8260B	Various	Canister	None	Ambient	30	Days
Arizona State Specific VOCs in Vapor - 8260B	Air	NA	EPA 8260B	Various	Tedlar	None	Ambient	72	Hours
Arizona State Specific VOCs in Vapor - 8015B	Air	NA	EPA 8015B	Various	Canister	None	Ambient	30	Days
Arizona State Specific VOCs in Vapor - 8015B	Air	NA	EPA 8015B	Various	Tedlar	None	Ambient	72	Hours
<b>AQUATIC TOXICITY &amp; RELATED</b>									
C.dubia - Acute	NPW	2002	NA	1L/1Gal	HDPE	None	0 - 6°C	36	Hours
Minnow - Acute	NPW	2000	NA	1L/1Gal	HDPE	None	0 - 6°C	36	Hours
Toxicity C.dubia - Chronic	NPW	1002	NA	1L/1Gal	HDPE	None	0 - 6°C	36	Hours
Toxicity Minnow - Chronic	NPW	1000	NA	1L/1Gal	HDPE	None	0 - 6°C	36	Hours
<b>BACTERIA</b>									
Chlorophyll A/Pheophytin A	NPW	SM10200H	NA	1L	Amber Glass	None	0 - 6°C	72	Hours
Coliform, Total	NPW	SM9222B	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	8	Hours
E. Coli	NPW	SM9223B, Colilert	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	8	Hours
Enterococci	NPW	ASTM D6503-99, Enterolert	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	8	Hours
Fecal Coliform	NPW	SM9222D	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	6	Hours
Fecal Coliform	NPW	SM9221C/E	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	6	Hours
Heterotropic Plate Count	NPW	9215B	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	6	Hours
Salmonella	NPW	SM9260D	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	8	Hours
Cryptosporidium	PW	1622, 1623	NA	10L	LDPE	None	<20°C	96	Hours
E. Coli	PW	SM9223B	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	30	Hours
Fecal Coliform (MPN)	PW	9221E	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	30	Hours
Fecal Coliform	PW	SM9222D	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	30	Hours
Enterococci	PW	ASTM D6503-99	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	30	Hours
Heterotropic Plate Count	PW	9215B	NA	110ml	Micro	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	6	Hours
Coliform, Total	PW	9222B, 9223B	NA	110ml	Plastic	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	30	Hours
Coliform, Total	SS	SM9221B, 9222	NA	Sterile 125mL	Plastic	None	0 - 6°C	24	Hours
Fecal Coliform (MPN)	SS	9221E	NA	Sterile 125mL	Plastic	None	0 - 6°C	24	Hours
Fecal Coliform (Sludge)	SS	9222D	NA	Sterile 125mL	Plastic	None	0 - 6°C	24	Hours

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Enterococci	SS	ASTM D6503-99	9230	Sterile 125mL	Plastic	None	0 - 6°C	6	Hours
Salmonella	SS	SM9260D	NA	110ml	Micro	None	0 - 6°C	6	Hours
Heterotropic Plate Count	SS	SM9215B	NA	110ml	Micro	None	0 - 6°C	6	Hours
S.O.U.R.	SS	SM 2710B	NA	1L	HDPE	None	0 - 6°C	2	Hours
<b>INORGANIC CLASSIC</b>									
Acidity	NPW	SM2310B, ASTM D1067	NA	250ml	HDPE	None	0 - 6°C	14	Days
Alkalinity	NPW	SM2320B	NA	500ml	HDPE	None	0 - 6°C	14	Days
Alkalinity	NPW	310.2	NA	500ml	HDPE	None	0 - 6°C	14	Days
Ammonia Nitrogen	NPW	350.1, SM4500NH <sub>3</sub> G	NA	500ml	HDPE	H <sub>2</sub> SO <sub>4</sub> +Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	28	Days
Ammonia, distilled/titration (4500)	NPW	SM4500NH <sub>3</sub> C	NA	500ml	HDPE	H <sub>2</sub> SO <sub>4</sub> +Na <sub>2</sub> S <sub>2</sub> O <sub>4</sub>	0 - 6°C	28	Days
Asbestos	NPW	100.1	NA	1L	Glass	None	0 - 6°C	48	Hours
BOD/CBOD (Total & Soluble)	NPW	SM5210B	NA	1L	HDPE	None	0 - 6°C	48	Hours
Bromide	NPW	300.0, SM4110B	9056	125ml	HDPE	None	0 - 6°C	28	Days
Carbon Dioxide	NPW	SM4500CO <sub>2</sub> D	NA	1L	HDPE	None	0 - 6°C	15	Min
Chemical Oxygen Demand (COD)	NPW	410.4, SM5220D	NA	250ml	HDPE	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Chemical Oxygen Demand (COD), Soluble	NPW	410.4, SM5220D	NA	250ml	HDPE	None	0 - 6°C	28	Days
Chloride	NPW	300.0, SM4110B	9056	125ml	HDPE	None	0 - 6°C	28	Days
Chlorine, residual	NPW	SM4500Cl-G	NA	250ml	HDPE	None	0 - 6°C	15	Min
Color	NPW	SM2120B	NA	250ml	HDPE	None	0 - 6°C	48	Hours
CTAS Surfactants	NPW	SM5540D	NA	1L	HDPE	None	0 - 6°C	48	Hours
Cyanide - Total	NPW	335.4, SM4500CNE	9012	250ml	Amber HDPE	NaOH	0 - 6°C	14	Days
Cyanide - Total	NPW	Kelada-01	NA	250ml	Amber HDPE	NaOH	0 - 6°C	14	Days
Cyanide, Amenable	NPW	SM4500CNG	9012	250ml	Amber HDPE	NaOH	0 - 6°C	14	Days
Cyanide, Free	NPW	SM4500CNE	NA	250ml	Amber HDPE	NaOH	0 - 6°C	14	Days
Cyanide, Weak Acid Dissoc.	NPW	SM4500CN-I	NA	250ml	Amber HDPE	NaOH	0 - 6°C	14	Days
Dissolved Organic Carbon (DOC)	NPW	SM5310B	9060	250ml	Amber Glass	None	0 - 6°C	28	Days

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Ferrous Iron	NPW	SM3500FeB	NA	250ml	Amber Glass	HCl	0 - 6°C	15	Min
Fluoride	NPW	300.0, SM4110B	9056	125ml	HDPE	None	0 - 6°C	28	Days
Hardness	NPW	200.7, SM2340B	NA	250ml	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
Hardness	NPW	130.1	NA	500ml	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
Hardness	NPW	SM2340C	NA	500ml	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
Iodide	NPW	345.1	NA	250ml	HDPE	None	0 - 6°C	Immed	
Kjeldahl Nitrogen, TKN	NPW	351.2, SM4500Norg B/C	NA	250ml	HDPE	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Methylene Blue Active Subst. (MBAS)	NPW	SM5540C	NA	250ml	HDPE	None	0 - 6°C	48	Hours
Nitrate	NPW	300.0, SM4110B	9056	125ml	HDPE	None	0 - 6°C	48	Hours
Nitrate + Nitrite	NPW	353.2, SM4500NO <sub>3</sub> F	NA	250ml	HDPE	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Nitrite	NPW	300.0, SM4110B	9056	125ml	HDPE	None	0 - 6°C	48	Hours
Oil & Grease (Hexane Extr)	NPW	1664A, SM5520B	9070	1L	Glass	HCl	0 - 6°C	28	Days
Oil & Grease, Free	NPW	1664A	9070	1L	Amber Glass	None	0 - 6°C	28	Days
Organic Nitrogen	NPW	351.2 - 350.1	NA	500ml	HDPE	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Oxygen, dissolved (DO)	NPW	SM4500O C, SM4500O G	NA	125ml	HDPE	None	0 - 6°C	15	Min
pH	NPW	SM4500H B	9040	125ml	HDPE	None	0 - 6°C	15	Min
Phenols (Total) by 4AAP	NPW	420.1, 420.4	9066	250ml	Amber Glass	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Phosphate, Ortho	NPW	365.1, SM4500P-E	NA	250ml	HDPE	None	0 - 6°C	48	Hours
Phosphorus, Total	NPW	365.1, SM4500P-B.5	NA	250ml	HDPE	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Residue, Filterable (TDS)	NPW	SM2540C	NA	250ml	HDPE	None	0 - 6°C	7	days
Residue, non-Filterable (TSS)	NPW	SM2540D	NA	1L	HDPE	None	0 - 6°C	7	Days
Residue, Settleable (SS)	NPW	SM2540F	NA	1L	HDPE	None	0 - 6°C	48	Hours
Residue, Total (TS)	NPW	SM2540B	NA	250ml	HDPE	None	0 - 6°C	7	Days
Specific Conductance (Conductivity)	NPW	120.1, SM2510B	9050	250ml	HDPE	None	0 - 6°C	28	Days
Sulfate	NPW	300.0, SM4110B	9056	125ml	HDPE	None	0 - 6°C	28	Days
Sulfide	NPW	NA	9030, 9034	500ml	HDPE	NaOH+ZnAc	0 - 6°C	7	Days

Parameter	Matrix <sub>1</sub>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Sulfide	NPW	SM4500S <sup>2</sup> D	NA	500ml	HDPE	NaOH+ZnAc	0 - 6°C	7	Days
Sulfide, Dissolved	NPW	SM4500S <sup>2</sup> D	NA	125ml	Amber Glass	NaOH+ZnAc	0 - 6°C	7	Days
Sulfite	NPW	SM4500SO <sub>3</sub> B	NA	250ml	HDPE	None	0 - 6°C	15	Min
Tannins and Lignins	NPW	SM5550B	NA	250ml	HDPE	None	0 - 6°C	NA	
Temperature	NPW	SM2550B	NA	onsite		None	0 - 6°C	15	Min
Total Organic Carbon (TOC)	NPW	SM53010B	9060	250ml	Amber Glass	HCl	0 - 6°C	28	Days
Total Organic Halides (TOX)	NPW	450.1, 9020	NA	1L	Amber Glass	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Total Organic Halides (TOX)	NPW	SM5320B	NA	1L	Amber Glass	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	14	Days
Turbidity	NPW	180.1, SM2130B	NA	250ml	HDPE	None	0 - 6°C	48	Hours
Volatile Solids (VS)	NPW	160.4	NA	250ml	HDPE	None	0 - 6°C	7	Days
Volatile Susp. Solids (VSS)	NPW	SM2540E	NA	500ml	HDPE	None	0 - 6°C	7	Days
Alkalinity	PW	2320B	NA	500ml	HDPE	None	0 - 6°C	14	Days
Ammonia Nitrogen	PW	350.1, SM4500NH <sub>3</sub> G	NA	250ml	HDPE	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Ammonia, distilled/titration (4500)	PW	SM4500NH <sub>3</sub> C	NA	250ml	HDPE	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Asbestos	PW	100.1	NA	1L	Glass	None	0 - 6°C	48	Hours
Bromide	PW	300.0, SM4110B	NA	125ml	HDPE	None	0 - 6°C	28	Days
Calcium-hardness	PW	SM3500-Ca B	NA	250ml	Amber Glass	HNO <sub>3</sub>	0 - 6°C	180	Days
Carbon Dioxide	PW	SM4500CO <sub>2</sub> D	NA	1L	HDPE	None	0 - 6°C	15	Min
Chloride	PW	300.0, SM4110B	NA	125ml	HDPE	None	0 - 6°C	28	Days
Chlorine, residual	PW	SM4500Cl-G	NA	250ml	HDPE	None	0 - 6°C	15	Min
Color	PW	SM2120B	NA	250ml	HDPE	None	0 - 6°C	48	Hours
Corrosivity	PW	Calc	NA		Plastic	None	0 - 6°C	NA	
Cyanide - Total	PW	335.4, SM4500CNE	NA	250ml	HDPE Amber	NaOH	0 - 6°C	14	Days
Cyanide - Total	PW	Kelada-01	NA	250ml	HDPE Amber	NaOH	0 - 6°C	14	Days
Cyanide, Amenable	PW	SM4500CNG	NA	250ml	HDPE Amber	NaOH	0 - 6°C	14	Days
Cyanide, Free	PW	SM4500CNE	NA	250ml	HDPE Amber	NaOH	0 - 6°C	14	Days
Dissolved Organic Carbon (DOC)	PW	SM5310C	NA	250ml	Amber Glass	None	0 - 6°C	28	Days
Dissolved Solids (TDS)	PW	SM2540C	NA	250ml	HDPE	None	0 - 6°C	7	Days

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Fluoride	PW	300.0, SM4110B	NA	125ml	HDPE	None	0 - 6°C	28	Days
Hardness	PW	200.7, SM2340B	NA	250ml	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
Hardness	PW	130.1	NA	500ml	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
Hardness	PW	SM2340C	NA	500ml	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
Methylene Blue Active Subst. (MBAS)	PW	SM5540C	NA	1L	HDPE	None	0 - 6°C	48	Hours
Nitrate	PW	300.0, SM4110B	NA	125ml	HDPE	None	0 - 6°C	48	Hours
Nitrate + Nitrite	PW	353.2, SM4500NO <sub>3</sub> F	NA	250ml	HDPE	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Nitrite	PW	300.0, SM4110B	NA	125ml	HDPE	None	0 - 6°C	48	Hours
Odor	PW	SM2150B	NA	250ml	Amber Glass	None	0 - 6°C	24	Hours
Perchlorate	PW	314	NA	125ml	HDPE	None	0 - 6°C	28	Days
pH	PW	150.1, SM4500-H B	NA	125ml	HDPE	None	0 - 6°C	15	Min
Phosphate, Ortho	PW	SM4500P-E	NA	250ml	HDPE	None	0 - 6°C	48	Hours
Specific Conductance	PW	SM2510B	NA	250ml	HDPE	None	0 - 6°C	28	Days
Sulfate	PW	300.0, SM4110B	NA	125ml	HDPE	None	0 - 6°C	28	Days
Total Organic Carbon (TOC)	PW	SM5310C	NA	250ml	Amber Glass	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Total Organic Halides (TOX)	PW	SM5320B	NA	1L	Amber Glass	H <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28	Days
Turbidity	PW	180.1, SM2130B	NA	250ml	HDPE	None	0 - 6°C	48	Hours
UV Absorbance at 254 nm	PW	SM5910B	NA	250ml	Amber Glass	None	0 - 6°C	48	Hours
Asbestos	SS	PLM	NA			None	0 - 6°C	NA	
Bromide	SS	NA	9056	4 oz.	Glass	None	0 - 6°C	28	Days
Chloride	SS	NA	9056	4 oz.	Glass	None	0 - 6°C	28	Days
Corrosivity	SS	NA	9045D	4 oz.	Glass	None	0 - 6°C	15	Min
Cyanide - Total	SS	NA	9010/9012	4 oz.	Glass	None	0 - 6°C	14	Days
Cyanide, Amenable	SS	NA	9010/9012	4 oz.	Glass	None	0 - 6°C	14	Days
Cyanide, Free	SS	NA	9010/9012	4 oz.	Glass	None	0 - 6°C	14	Days
Extractable Organic Halides (EOX)	SS	NA	9023	4 oz.	Glass	None	0 - 6°C	28	Days
Fluoride	SS	NA	9056	4 oz.	Glass	None	0 - 6°C	28	Days
Kjeldahl Nitrogen, TKN	SS	351.2	NA	2 oz.	Glass	None	0 - 6°C	28	Days
Nitrate	SS	NA	9056	4 oz.	Glass	None	0 - 6°C	28	Days
Nitrite	SS	NA	9056	4 oz.	Glass	None	0 - 6°C	28	Days
Oil & Grease	SS	NA	9071	4 oz.	Glass	None	0 - 6°C	28	Days

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
pH	SS	NA	9040, 9045	2 oz.	Glass	None	0 - 6°C	15	Min
Phenols by 4AAP	SS	NA	9066	4 oz.	Glass	None	0 - 6°C	28	Days
Phosphate, Ortho	SS	SM4500P-E	NA	4 oz.	Glass	None	0 - 6°C	48	Hours
Phosphorus, Total	SS	NA	9056	4 oz.	Glass	None	0 - 6°C	28	Days
Residue, Total	SS	SM2540G	NA	4 oz.	Glass	None	0 - 6°C	14	Days
Solids, Total	SS	SM2540B	NA	4 oz.	Glass	None	0 - 6°C	14	Days
Specific Conductance	SS	NA	9050	4 oz.	Glass	None	0 - 6°C	28	Days
Sulfate	SS	NA	9056	4 oz.	Glass	None	0 - 6°C	28	Days
Sulfide	SS	NA	9030, 9034	2 oz.	Glass	none	0 - 6°C	7	Days
Total Organic Carbon (TOC)	SS	NA	9060	2 oz.	Glass	None	0 - 6°C	28	Days
Total Organic Carbon (TOC)	SS	ASTM F1647-02A mod	NA	4 oz.	Glass	None	0 - 6°C	28	Days
Total Organic Carbon (TOC)	SS	USDA LOI	NA	4 oz.	Glass	None	0 - 6°C	28	Days
<b>INORGANIC METALS</b>									
Chromium, Hexavalent - Cr <sup>+6</sup>	NPW	SM3500CrB	7196	250ml	HDPE	None	0 - 6°C	24	Hours
Chromium, Hexavalent - Cr <sup>+6</sup>	NPW	SM3500CrC	7199	250ml	HDPE	None	0 - 6°C	24	Hours
Chromium, Hexavalent - Cr <sup>+6</sup>	NPW	218.6, SM3500CrC	NA	125ml	HDPE	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0 - 6°C	28 <sup>5</sup>	Days
Mercury (Dissolved)	NPW	245.1	7470	500ml	HDPE	None	0 - 6°C	28	Days
Mercury (Total)	NPW	245.1	7470	500ml	HDPE	HNO <sub>3</sub>	0 - 6°C	28	Days
Metals (Dissolved) ICP	NPW	200.7	6010	500ml	HDPE	None	NA	180	Days
Metals (Dissolved) ICPMS	NPW	200.8	6020	500ml	HDPE	None	NA	180	Days
Metals (Total) ICP	NPW	200.7	6010	500ml	HDPE	HNO <sub>3</sub>	NA	180	Days
Metals (Total) ICPMS	NPW	200.8	6020	500ml	HDPE	HNO <sub>3</sub>	NA	180	Days
Chromium, Hexavalent - Cr <sup>+6</sup>	PW	218.7	NA	125ml	HDPE	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> /(NH <sub>4</sub> )OH	0 - 6°C	14	Days
Mercury (Dissolved)	PW	245.1	NA	500ml	HDPE	None	0 - 6°C	28	Days
Mercury (Total)	PW	245.1	NA	500ml	HDPE	HNO <sub>3</sub>	0 - 6°C	28	Days
Metals (Dissolved) ICP	PW	200.7	NA	500ml	HDPE	None	NA	180	Days

Parameter	Matrix <sub>1</sub>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Metals (Dissolved) ICPMS	PW	200.8	NA	500ml	HDPE	None	NA	180	Days
Metals (Total) ICP	PW	200.7	NA	500ml	HDPE	HNO <sub>3</sub>	NA	180	Days
Metals (Total) ICPMS	PW	200.8	NA	500ml	HDPE	HNO <sub>3</sub>	NA	180	Days
Chromium, Hexavalent - Cr <sup>+6</sup>	SS	NA	3060/7196	4 oz.	Glass	None	0 - 6°C	30	Days
Chromium, Hexavalent - Cr <sup>+6</sup>	SS	NA	3060/7199	4 oz.	Glass	None	0 - 6°C	30	Days
Mercury (Total)	SS	NA	7471	2 oz.	Glass	<6 C	0 - 6°C	28	Days
Metals (Total) ICP	SS	NA	6010	2 oz.	Glass	None	NA	180	Days
Metals (Total) ICPMS	SS	NA	6020	4 oz.	Glass	None	NA	180	Days
Sodium Adsorption Ratio (SAR)	SS	NA	6010	250mL	Glass	None	0 - 6°C	180	Days
Michigan Fine/Coarse Soil Sieve for Lead	SS	NA	NA	250mL	Glass	None	0 - 6°C	180	Days
<b>PHYSICAL</b>									
Flashpoint/ignitability (Closed Cup)	NPW	ASTM 93-07	1010	1L	Glass	None	0 - 6°C	14	Days
Flashpoint/ignitability (Open Cup)	NPW	ASTM 92-05A	NA	1L	Glass	None	0 - 6°C	14	Days
Flashpoint/ignitability (Closed Cup)	SS	ASTM 93-07	1010	4 oz.	Glass	None	0 - 6°C	14	Days
Flashpoint/ignitability (Open Cup)	SS	ASTM 92-05A	NA	4 oz.	Glass	None	0 - 6°C	NA	
Ash Content	SS	SM2540G, ASTM D2974	NA	4 oz.	Glass	None	0 - 6°C	14	Days
Cation Exchange Capacity	SS	NA	9081	4 oz.	Glass	None	0 - 6°C	180	Days
Paint Filter Test	SS	NA	9095	4 oz.	Glass	None	0 - 6°C	NA	
Permeability (Section 2.8)	SS	NA	9100	Various	Shelby Tube	None	0 - 6°C	28	Days
React. Sulf.(SW846 7.3.4.2)	SS	NA	Sec. 7.3	4 oz.	Glass	None	0 - 6°C	7	Days
Reactive CN (SW846 7.3.4.1)	SS	NA	Sec. 7.3	4 oz.	Glass	None	0 - 6°C	14	Days
Resistivity (ASTM)	SS	NA	NA	16 oz	Glass	None	0 - 6°C	28	Days
Specific Gravity	SS	NA	NA	Various	Plastic	None	0 - 6°C	14	Days



Parameter	Matrix <sub>1</sub>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
<b>LEACHING METHODS</b>									
Cal Wet (CACR Title22 Chap11 AppII)	SS	NA	NA	100g	Glass	None	0 - 6°C	14/28/180	Days
EP TOX	SS	NA	1310	100g	Glass	None	0 - 6°C	14/28/180	Days
MEP	SS	NA	1320	100g	Glass	None	0 - 6°C	14/28/180	Days
SPLP	SS	NA	1312	100g	Glass	None	0 - 6°C	14/28/180	Days
TCLP	SS	NA	1311	100g	Glass	None	0 - 6°C	14/28/180	Days
<b>ORGANIC - SEMIVOLATILES</b>									
Base/Neutral/Acid (BNA)	NPW	NA	8270	1L or 100mL	Amber Glass	None	0 - 6°C	7	Days
Base/Neutral/Acid (BNA)	NPW	625, SM6410B	NA	1L or 100mL	Amber Glass	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	7	Days
Diesel Range Organics	NPW	NA	8015	1L, 100mL, or 40mL	Amber Glass	HCl	0 - 6°C	7	Days
Dioxin	NPW	1613	NA	1L	Amber Glass	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	1	Year
EDB/DBCP	NPW	NA	8011	2 x 40 ml	Glass	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	7	Days
Formaldehyde	NPW	NA	8315	1L	Amber Glass	None	0 - 6°C	3	Days
Herbicides	NPW	1658, SM6640B	8151	1L	Amber Glass	None	0 - 6°C	7	Days
Polynuclear Aromatic Hydrocarbons (PAH)	NPW	625, SM640B	8270	1L, 100mL, or 40mL	Amber Glass	None	0 - 6°C	7	Days
Polynuclear Aromatic Hydrocarbons (PAH-SIM)	NPW	NA	8270	1L, 100mL, or 40mL	Amber Glass	None	0 - 6°C	7	Days
Polynuclear Aromatic Hydrocarbons (PAH)	NPW	610, SM6440B	8310	1L	Amber Glass	None	0 - 6°C	7	Days
Pesticides - Organophos Comp	NPW	614, 622, 1657	8141	1L	Amber Glass	None	0 - 6°C	7	Days
Pesticides & PCB's	NPW	608, SM6630B, SM6630C	8081, 8082	1L or 100mL	Amber Glass	None	0 - 6°C	7	Days
Base/Neutral/Acid (BNA)	PW	525	NA	1L	Amber Glass	HCl+Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	7	Days
Carbamates	PW	531.1	NA	2 x 60ml	Amber Glass	AcAcid+Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	7	Days
Dioxin	PW	1613	NA	1L	Amber Glass	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	7	Days
Diquat	PW	549	NA	1L	PVC Amber	H <sub>2</sub> SO <sub>4</sub> + Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	7	Days
EDB/DBCP	PW	504.1	NA	2 x 40 ml	Glass	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	28	Days
Endothall	PW	548	NA	250ml	Amber Glass	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	7	Days

Parameter	Matrix <sub>1</sub>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Glyphosate	PW	547	NA	2 x 60ml	Glass	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	7	Days
Herbicides	PW	515.1, SM6640B	NA	1L	Amber Glass	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	7	Days
Pesticides - Nitrogen/phosphorus Comp	PW	507	NA	1L	Amber Glass	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	14	Days
Pesticides - Organochlorine	PW	508	NA	1L	Amber Glass	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0 - 6°C	7	Days
Haloacetic acids - HAA's	PW	552.2	NA	500ml	Amber Glass	NH <sub>4</sub> Cl	0 - 6°C	28	Days
Base/Neutral/Acid (BNA)	SS	NA	8270	4 oz.	Glass	None	0 - 6°C	14	Days
Dioxin	SS	NA	8290	5 oz.	Glass	None	0 - 6°C	30	Days
Formaldehyde	SS	NA	8315	4 oz.	Glass	None	0 - 6°C	3	Days
Herbicides	SS	NA	8151	4 oz.	Glass	None	0 - 6°C	14	Days
Polynuclear Aromatic Hydrocarbons (PAH)	SS	NA	8270	4 oz.	Glass	None	0 - 6°C	14	Days
Polynuclear Aromatic Hydrocarbons (PAH-SIM)	SS	NA	8270	4 oz.	Glass	None	0 - 6°C	14	Days
Polynuclear Aromatic Hydrocarbons (PAH)	SS	NA	8310	4 oz.	Glass	None	0 - 6°C	14	Days
Pesticides - Organophos Comp	SS	NA	8141	4 oz.	Glass	None	0 - 6°C	14	Days
Pesticides & PCB's	SS	NA	8081, 8082	4 oz.	Glass	None	0 - 6°C	14	Days
Total Chlorine in Oil	SS	ASTM D808-00	NA	125ml	HDPE	None	0 - 6°C	24	Hours
<b>ORGANIC - VOLATILES</b>									
Meetic - Methanol and Ethanol	NPW	NA	EPA 8015 Mod	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Methane, Ethane, Ethene, Propane	NPW	RSK-175	NA	40ml	Amber Glass	HCl	0 - 6°C	14	Days
BTEX (water)	NPW	602, SM6200C	8021	2 x 40 ml	Amber Glass	HCl	0 - 6°C	14	Days
BTEX (water)	NPW	602, SM6200C	8021	2 x 40 ml	Amber Glass	None	0 - 6°C	7	Days
Gasoline Range Organics (GRO)	NPW	NA	8015	2 x 40 ml	Amber Glass	HCl	0 - 6°C	14	Days
VOC's	NPW	624, SM6200B	8260	2 x 40 ml	Amber Glass	HCl	0 - 6°C	14	Days
VOC's	NPW	624, SM6200B	8260	2 x 40 ml	Amber Glass	none	0 - 6°C	7	Days
VOC's	PW	524.2	NA	2 x 40 ml	Amber Glass	Ascorbic Acid+HCl	0 - 6°C	14	Days

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Meeteac - Methanol and Ethanol	SS	NA	EPA 8015 Mod	2 oz.	Glass	None	0 - 6°C	14	Days
BTEX (soil)	SS	NA	8021	4 oz.	Glass	None	0 - 6°C	14	Days
VOC's	SS	NA	8260	2 oz.	Glass	none	0 - 6°C	14	Days
VOC's	SS	NA	8260	40ml	Amber Glass	MeOH	0 - 6°C	14	Days
VOC's	SS	NA	8260	40ml	Amber Glass	NaHSO <sub>4</sub> or TSP(MO) or DI Water(FL)	0 - 6°C	14	Days
VOC's	SS	NA	8260	NA	Encore	none	0 - 6°C	48	Hours
<b>RADIOCHEMISTRY</b>									
Rad - Gross alpha	NPW	900	na	1L	Plastic	HNO <sub>3</sub>	0 - 6°C	180	Days
Rad - Gross beta	NPW	900	na	1L	Plastic	HNO <sub>3</sub>	0 - 6°C	180	Days
Rad - Radium 226	NPW	903.1	na	1L	Plastic	HNO <sub>3</sub>	0 - 6°C	180	Days
Rad - Radium 228	NPW	904	na	1L	Plastic	HNO <sub>3</sub>	0 - 6°C	180	Days
Rad - Gross alpha	PW	900	na	1L	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
Rad - Gross beta	PW	900	na	1L	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
Rad - Radium 226	PW	903.1	na	1L	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
Rad - Radium 228	PW	904	na	1L	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
Rad - Tritium	PW	906	na	1L	HDPE	None	0 - 6°C	180	Days
Strontium-90	PW	905	na	1L	HDPE	HNO <sub>3</sub>	0 - 6°C	180	Days
<b>STATE SPECIFIC PETROLEUM METHODS</b>									
Alaska DRO	NPW	NA	AK102	100ml	Amber Glass	HCl	0 - 6°C	14	Days
Alaska DRO	SS	NA	AK102	4 oz.	Glass	None	0 - 6°C	14	Days
Alaska GRO	NPW	NA	AK101	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Alaska GRO	SS	NA	AK101	60ml	Amber Glass	MeOH	0 - 6°C	28	Days
Alaska Motor Oil	NPW	NA	AK103	100ml	Glass	HCl	0 - 6°C	14	Days
Alaska Motor Oil	SS	NA	AK103	4 oz.	Glass	None	0 - 6°C	14	Days
Arizona GRO	SS	NA	AZ 8015	2 oz.	Glass	None	0 - 6°C	14 <sup>9</sup>	Days
Arizona TPH	SS	NA	AZ 8015	4 oz.	Glass	None	0 - 6°C	14	Days
California DRO	NPW	NA	8015	1L	Amber Glass	HCl	0 - 6°C	7	Days
California DRO	NPW	NA	8015	40ml	Amber Glass	HCl	0 - 6°C	7	Days
California DRO	SS	NA	8015	4 oz.	Glass	None	0 - 6°C	7	Days
Connecticut EPH	NPW	NA	8015	1L	Amber Glass	HCl	0 - 6°C	14	Days
Connecticut EPH	SS	NA	8015	4 oz.	Glass	None	0 - 6°C	14	Days

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Florida TPH	NPW	NA	FL-Pro	1L	Amber Glass	HCl	0 - 6°C	7	Days
Florida TPH	SS	NA	FL-Pro	4 oz.	Glass	None	0 - 6°C	14	Days
Indiana DRO	NPW	NA	8015	1L	Amber Glass	HCl	0 - 6°C	7	Days
Indiana DRO	SS	NA	8015	4 oz.	Glass	None	0 - 6°C	14	Days
Indiana ERO	NPW	NA	8015	1L	Amber Glass	HCl	0 - 6°C	7	Days
Indiana ERO	SS	NA	8015	4 oz.	Glass	None	0 - 6°C	7	Days
Indiana GRO	NPW	NA	8015	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Indiana GRO	SS	NA	8015	40ml	Amber Glass	MeOH	0 - 6°C	14	Days
Indiana GRO	SS	NA	8015	40ml	Amber Glass	NaHSO <sub>4</sub>	0 - 6°C	14	Days
Iowa GRO	NPW	NA	OA-1	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Iowa GRO	SS	NA	OA-1	4 oz.	Glass	None	0 - 6°C	14	Days
Iowa DRO	NPW	NA	OA-2	1L	Amber Glass	None	0 - 6°C	7	Days
Iowa DRO	SS	NA	OA-2	4 oz.	Glass	None	0 - 6°C	14	Days
Louisiana EPH	NPW	NA	MADEP EPH	1L	Amber Glass	HCl	0 - 6°C	14	Days
Louisiana EPH	SS	NA	MADEP EPH	4 oz.	Amber Glass	None	0 - 6°C	14	Days
Louisiana VPH	NPW	NA	MADEP VPH	1L	Amber Glass	HCl	0 - 6°C	14	Days
Louisiana VPH	SS	NA	MADEP VPH	40ml	Amber Glass	MeOH	0 - 6°C	28	Days
Massachusetts EPH	NPW	NA	MADEP EPH	1L	Amber Glass	HCl	0 - 6°C	14	Days
Massachusetts EPH	SS	NA	MADEP EPH	4 oz.	Amber Glass	None	0 - 6°C	14	Days
Massachusetts VPH	NPW	NA	MADEP VPH	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Massachusetts VPH	SS	NA	MADEP VPH	40ml	Amber Glass	MeOH	0 - 6°C	28	Days
Minnesota DRO	NPW	NA	WI DRO	1L	Amber Glass	HCl	0 - 6°C	7	Days
Minnesota DRO	SS	NA	WI DRO	60ml	Amber Glass	CH <sub>3</sub> Cl	0 - 6°C	47 <sup>9</sup>	Days
Minnesota GRO	NPW	NA	WI GRO	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Minnesota GRO	SS	NA	WI GRO	60ml	Amber Glass	MeOH	0 - 6°C	21 <sup>7</sup>	Days
Missouri DRO	NPW	NA	8270	1L	Amber Glass	None	0 - 6°C	7	Days
Missouri DRO	SS	NA	8270	4 oz.	Glass	None	0 - 6°C	14	Days
Missouri GRO	NPW	NA	8260	40ml	Amber Glass	TSP	0 - 6°C	14	Days
Missouri GRO	SS	NA	8260	40ml	Amber Glass	TSP	0 - 6°C	14	Days
Missouri GRO	SS	NA	8260	40ml	Amber Glass	MeOH	0 - 6°C	14	Days

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Montana EPH	NPW	NA	MT EPH	1L	Amber Glass	HCl	0 - 6°C	14	Days
Montana EPH	SS	NA	MT EPH	4 oz.	Amber Glass	None	0 - 6°C	14	Days
Montana VPH	NPW	NA	MT VPH	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Montana VPH	SS	NA	MT VPH	Encore	Amber Glass	None	0 - 6°C	7	Days
Montana VPH	SS	NA	MT VPH	40ml	Amber Glass	MeOH	0 - 6°C	28	Days
New Jersey EPH	NPW	NA	NJ EPH	1L	Amber Glass	HCl	0 - 6°C	14	Days
New Jersey EPH	SS	NA	NJ EPH	4 oz.	Amber Glass	None	0 - 6°C	14	Days
North Carolina EPH	NPW	NA	MADEP EPH	1L	Amber Glass	HCl	0 - 6°C	14	Days
North Carolina EPH	SS	NA	MADEP EPH	4 oz.	Amber Glass	None	0 - 6°C	14	Days
North Carolina VPH	NPW	NA	MADEP VPH	1L	Amber Glass	HCl	0 - 6°C	14	Days
North Carolina VPH	SS	NA	MADEP VPH	40ml	Amber Glass	MeOH	0 - 6°C	28	Days
Ohio DRO	NPW	NA	8015	1L	Amber Glass	None	0 - 6°C	7	Days
Ohio DRO	NPW	NA	8015	100ml	Amber Glass	None	0 - 6°C	7	Days
Ohio DRO	NPW	NA	8015	40ml	Amber Glass	None	0 - 6°C	7	Days
Ohio DRO	SS	NA	8015	4 oz.	Glass	None	0 - 6°C	14	Days
Ohio GRO	NPW	NA	8015	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Ohio GRO	SS	NA	8015	2 oz.	Glass	None	0 - 6°C	14	Days
Ohio GRO (VAP)	SS	NA	8015	Encore - Low Level	None	None	0 - 6°C	14 <sup>8</sup>	Days
Ohio GRO (VAP)	SS	NA	8015	Encore - High Level	None	MeOH	0 - 6°C	14	Days
Oklahoma DEQ GRO	NPW	NA	OK DEQ GRO	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Oklahoma DEQ GRO	SS	NA	OK DEQ GRO	4 oz.	Glass	None	0 - 6°C	14	Days
Oklahoma DEQ DRO	NPW	NA	OK DEQ DRO	1L	Amber Glass	HCl	0 - 6°C	7	Days
Oklahoma DEQ DRO	SS	NA	OK DEQ DRO	60ml	Amber Glass	CH <sub>3</sub> Cl	0 - 6°C	7 <sup>6</sup>	Days
Oregon TPH-Gx	NPW	NA	NWTPH-Gx	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Oregon TPH-Gx	SS	NA	NWTPH-Gx	4 oz.	Glass	None	0 - 6°C	14	Days
Oregon TPH-Dx	NPW	NA	NWTPH-Dx	1L	Amber Glass	HCl	0 - 6°C	14	Days
Oregon TPH-Dx	SS	NA	NWTPH-Dx	4 oz.	Glass	None	0 - 6°C	14	Days

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Tennessee DRO	NPW	NA	TN EPH	1L	Amber Glass	HCl	0 - 6°C	7	Days
Tennessee DRO	NPW	NA	TN EPH	100 ml	Amber Glass	HCl	0 - 6°C	7	Days
Tennessee DRO	SS	NA	TN EPH	4 oz.	Glass	None	0 - 6°C	14	Days
Tennessee GRO	NPW	NA	TN GRO	40ml	Amber Glass	HCl	0 - 6°C	7	Days
Tennessee GRO	SS	NA	TN GRO	2 oz.	Glass	None	0 - 6°C	14	Days
Texas TPH	NPW	NA	TX1005/ TX1006	60ml	Amber Glass	HCl	0 - 6°C	14	Days
Texas TPH	SS	NA	TX1005/ TX1006	4 oz.	Glass	None	0 - 6°C	14	Days
Washington TPH-Gx	NPW	NA	NWTPH- Gx	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Washington TPH-Gx	SS	NA	NWTPH- Gx	4 oz.	Glass	None	0 - 6°C	14	Days
Washington TPH-Dx	NPW	NA	NWTPH- Dx	1L	Amber Glass	HCl	0 - 6°C	14	Days
Washington TPH-Dx	SS	NA	NWTPH- Dx	4 oz.	Glass	None	0 - 6°C	14	Days
Wisconsin DRO	NPW	NA	WI DRO	1L	Amber Glass	HCl	0 - 6°C	7	Days
Wisconsin DRO	NPW	NA	WI DRO	100 ml	Amber Glass	HCl	0 - 6°C	7	Days
Wisconsin DRO	SS	NA	WI DRO	60ml	Amber Glass	CH <sub>3</sub> Cl	0 - 6°C	47 <sup>9</sup>	Days
Wisconsin GRO	NPW	NA	WI GRO	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Wisconsin GRO	SS	NA	WI GRO	60ml	Amber Glass	MeOH	0 - 6°C	21 <sup>7</sup>	Days
Wyoming DRO	NPW	NA	8015	40ml	Amber Glass	HCl	0 - 6°C	7	Days
Wyoming DRO	NPW	NA	8015	1L	Amber Glass	HCl	0 - 6°C	7	Days
Wyoming DRO	SS	NA	8015	4 oz.	Glass	None	0 - 6°C	14	Days
Wyoming GRO	NPW	NA	8015	40ml	Amber Glass	HCl	0 - 6°C	14	Days
Wyoming GRO	SS	NA	8015	2 oz.	Glass	None	0 - 6°C	14	Days
<b>INDUSTRIAL HYGIENE (IH) METHODS</b>									
Particulates not otherwise regulated	Air	NIOSH 0500	NA	NA	2 piece 37mm PVC Pre-weighed filter	None	NA	NA	NA
Respirable Dust	Air	NIOSH 0600	NA	NA	3 piece 37mm PVC Pre-weighed filter	None	NA	NA	NA
Metals	Air	NA	EPA 6010B	NA	0.8-µm MCE or 5.0-µm PVC cassette	None	NA	NA	NA

Parameter	Matrix <sup>1</sup>	EPA Approved Method <sup>2</sup>	SW846 <sup>3</sup>	Rec. Volume	Bottle Type	Pres.	Temp	Holding Time	Holding Time Units
Metals	Air	NIOSH 7300	NA	NA	0.8-µm MCE or 5.0-µm PVC cassette	None	NA	NA	NA
Metals	Air	OSHA ID-125G	NA	NA	0.8-µm MCE or 5.0-µm PVC cassette	None	NA	NA	NA

Footnotes:

- 1) Matrix - NPW=Nonpotable Water, PW= Potable Water, SS=Solids
- 2) EPA Approved Method - Where applicable EPA methods are listed. Compounds/programs not regulated by EPA will have methods appropriate to their regulatory oversight.
- 3) SW846 Method - Where one exists, the appropriate Solid Waste method will be listed
- 4) Preservative Key
  - (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> = Ammonium Sulfate
  - AcAcid = Acetic Acid
  - CH<sub>3</sub>Cl = Methylene Chloride
  - H<sub>2</sub>SO<sub>4</sub> = Sulfuric Acid
  - HCl= Hydrochloric Acid
  - HNO<sub>3</sub> = Nitric Acid
  - MeOH = Methanol
  - Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> = Sodium Thiosulfate
  - NaHSO<sub>4</sub> = Sodium Bisulfate
  - NH<sub>4</sub>Cl = Ammonium Chloride
  - TSP = Trisodium Phosphate
  - ZnAc = Zinc Acetate
- 5) Must be field filtered to achieve the extended holding time.
- 6) Must be received by lab within 7 days of sampling for solvent addition.
- 7) Must be received by lab within 4 days of sampling for solvent addition.
- 8) Must be received by lab within 48 hours of sampling for freezing.
- 9) Must be received by lab within 72 hours of sampling for solvent addition.

## 14.7 SAMPLE CONTAINER PACKING PROCEDURES

ESC routinely sends sample containers to clients. Standard operating procedure determines the containers needed for the requested analyses. A sample request form is completed to document what is needed, the destination, the date prepared and the initials of the preparer. Containers are prepared, with appropriate preservatives, labels, and custody seals, and organized for the client's convenience in a cooler. The cooler also contains a temperature blank, chain of custody, a return address label, and applicable instructions. The cooler is bound with packaging tape (and a custody seal if requested) and shipped UPS.

## 15.0 *SAMPLE DISPATCH*

Samples collected during field investigations or in response to a hazardous materials incident are classified by the project manager, prior to shipping, as either environmental or hazardous material samples. The shipment of samples, designated as environmental samples, is not regulated by the U.S. Department of Transportation.

Samples collected from certain process streams, drums, bulk storage tanks, soil, sediment, or water samples from suspected areas of high contamination may need to be shipped as hazardous. These regulations are promulgated by the US-DOT and described in the Code of Federal Regulations (49 CFR 171 through 177). The guidance for complying with US-DOT regulations in shipping environmental laboratory samples is given in the "National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples."

### 15.1 SHIPMENT OF ENVIRONMENTAL SAMPLES

Shipping receipts are maintained at the ESC laboratory. The shipment of preserved sample containers or bottles of preservatives (i.e., NaOH pellets, HCl, etc.) which are designated as hazardous under the US-DOT, Hazardous Materials Table, 49 CFR 171.101, must be transported pursuant to the appropriate US-DOT regulations. Samples packaged for shipment by ESC shall be segregated by sample type, preservation requirements, and potential contaminant level. During events in which large numbers of samples are collected, samples are segregated by analyses required. If multiple sites are sampled, or if specific and separate areas of interest are identified, samples are further segregated for packaging prior to shipment.

Environmental samples are packed prior to shipment using the following procedures:

1. Select a cooler (clean and strong). Line the cooler with a large heavy-duty plastic bag.
2. Allow sufficient headspace (except VOC's or others with zero headspace requirements) to compensate for any pressure and temperature changes.
3. Be sure the lids on all bottles are tight.
4. Place all bottles in appropriately sized polyethylene bags.
5. Place VOC vials in foam material transport sleeves.
6. Place foam padding in the bottom of the cooler and then place the bottles in the cooler with sufficient space to allow for the addition of more foam between the bottles.
7. Put ice on top of and/or between the samples.
8. Place chain of custody in a clean dry bag and into the cooler. Close the cooler and securely tape the cooler shut. The chain of custody seals should be affixed to the top and sides of the cooler so that the cooler cannot be opened without breaking the seal.



9. The shipping containers must be marked "THIS END UP". The name and address of the shipper shall be placed on the outside of the container. Labels used in the shipment of hazardous materials are not permitted to be on the outside of the container used to transport environmental samples and shall not be used.

## **16.0 INVESTIGATION WASTE**

### **16.1 GENERAL**

Field surveys conducted by ESC may generate waste materials. Some of these waste materials may be hazardous requiring proper disposal in accordance with EPA regulations.

#### 16.1.1 Types of Investigation Derived Wastes (IDW)

Materials which may be included in the IDW category are:

- Personnel protective equipment (PPE)
- Disposable sampling equipment (DE)
- Soil cuttings
- Groundwater obtained through well purging
- Spent cleaning and decontamination fluids
- Spent calibration standards

#### 16.1.2 Managing Non-hazardous IDW

Disposal of non-hazardous IDW should be addressed prior to initiating work at a site. Facility personnel should be consulted and wastes handled in an appropriate manner as directed by the client.

For development and purge water generated in the State of Florida, specific disposal requirements apply. The water is contained on-site in temporary storage until it is characterized. Appropriate disposal and/or treatment methods are then determined. Possible disposal options are:

- Direct discharge on-site to infiltrate the same or a more contaminated source
- Transportation to an off-site facility

In no case shall the water be discharged into any surface water unless permitted.

### 16.1.3 Management of Hazardous IDW

Disposal of hazardous or suspected hazardous IDW (as defined in 40 CFR 261.30-261.33 or displaying the characteristics of ignitability, corrosivity, reactivity, or TC toxicity) must be specified in the sampling plan. Hazardous IDW must be disposed in compliance with USEPA regulations. If appropriate, these wastes may be taken to a facility waste treatment system. These wastes may also be disposed of in the source area from which they originated if state regulations permit.

If on-site disposal is not feasible, appropriate analyses must be conducted to determine if the waste is hazardous. If so, they must be properly contained and labeled. They may be stored on the site for a maximum of 90 days before they must be manifested and shipped to a permitted treatment or disposal facility. Weak acids and bases may be neutralized in lieu of disposal as hazardous wastes. Neutralized wastewaters may be flushed into a sanitary sewer.

If possible, arrangements for proper containment, labeling, transportation, and disposal/treatment of IDW should be anticipated beforehand.

Investigation derived wastes should be kept to a minimum. Most of the routine studies conducted by ESC should not produce any IDW that are hazardous. Many of the above PPE and DE wastes can be deposited in municipal dumpsters if care is taken to keep them segregated from hazardous waste contaminated materials. Disposable equipment can often be cleaned to render it nonhazardous, as can some PPE, such as splash suits. The volume of spent solvent waste produced during equipment decontamination can be reduced or eliminated by applying only the minimum amount of solvent necessary.

## 17.0 SAMPLING BIBLIOGRAPHY

- 17.1 *Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual*, February 1, 1991, US EPA Region IV, Environmental Services Division.
- 17.2 *RCRA Ground-Water Monitoring Technical Enforcement Guidance Document* (GPO #5500000260-6), US EPA, September 1986.
- 17.3 *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition, Office of Solid and Emergency Response, US EPA, November 1986.
- 17.4 *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039, December 1988.

- 17.5 Florida Department of Environmental Regulation (DER) Quality Assurance Section (QAS) Guidance Documents:  
#89-01 - Equipment Material Construction, revised April 7, 1989  
#89-02 - Field QC Blanks, revised April 28, 1989  
#89-03 - Teflon<sup>®</sup> /Stainless Steel Bladder Pumps, revised May 10, 1988  
#89-04 - Field Cleaning Procedures, revised August 10, 1989
- 17.6 *DER Manual for Preparing Quality Assurance Plans*, DER-QA-001/90, revised September 30, 1992.
- 17.7 *NPDES Compliance Inspection Manual*, United States Environmental Protection Agency, Enforcement Division, Office of Water Enforcement and Permits, EN-338, 1988.
- 17.8 *Handbook for Monitoring Industrial Wastewater*, United States Environmental Protection Agency, Technology Transfer, 1973.
- 17.9 *EPA Primary Drinking Water Regulations*, 40 CFR 141.
- 17.10 *Rapid Bioassessment Protocols For Use in Streams and Rivers*, United States Environmental Protection Agency, Office of Water, EPA/841/B-99-002.
- 17.11 *Environmental Sampling and Analysis: A Practical Guide*. Lawrence H. Keith, Ph.D., 1991. Lewis Publishers.
- 17.12 *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*. Fifth Edition. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA/821/R-02/012
- 17.13 *Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms*. Fourth Edition. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA/821/R-02/013.

## 18.0 REVISIONS

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of this appendix are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0 (Appendix III)	Section 6.1 – Removed respirators from PPE Section 16 – New section for summary of revisions to previous version.

1.0 SIGNATORY APPROVALS

# WET LAB QUALITY ASSURANCE MANUAL

## APPENDIX IV TO THE ESC QUALITY ASSURANCE MANUAL

for

ESC LAB SCIENCES  
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Prepared by

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**NOTE: The QAM has been approved by the following people.**

  
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### **3.0 SCOPE AND APPLICATION**

This manual discusses specific QA requirements for general analytical protocols to ensure analytical data generated from the Wet Chemistry Laboratory, or Wet Lab, are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

### **4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES**

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling guidance and materials, and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual*.

### **5.0 PERSONNEL AND TRAINING**

#### **5.1 PERSONNEL**

Chris Unterstein, with a B.S. degree in Chemistry, is the Wet Chemistry Supervisor and is responsible for the overall production of this laboratory; including the management of the staff and scheduling. Mr. Unterstein has over 8 years of environmental laboratory experience. In his absence, Andrew Holt assumes responsibility for departmental decisions in Wet Chemistry laboratory.

Mr. Holt, with a B.S. in Plant and Soil Science, is proficient in wet chemistry analytical methods. Mr. Holt has 9 years of environmental laboratory experience.

#### **5.2 TRAINING**

- 5.2.1 All new analysts to the laboratory are trained by a Chemist or the Supervisor according to ESC protocol. ESC's training program is outlined in *SOP 030205 Technical Training and Personnel Qualifications*. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in Wet Lab analyses is demonstrated by acceptable participation in multiple proficiency testing programs (PTs) and daily Quality Control sample analyses. Documentation of analyst training is maintained on file within the department.

## **6.0 FACILITIES AND LABORATORY SAFETY**

### **6.1 FACILITIES**

The main area of the laboratory has approximately 2800 square feet with roughly 750 square feet of bench area. There is an additional 400 square feet of storage space and the lighting standard is fluorescence. The air system is a 5-ton Trane package unit and a 10-ton Trane package unit with natural gas for heating. The laboratory reagent water is provided through the US Filter deionizer system with a Millipore Milli-Q Academic A-10 system for finished water. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's waste disposal contractor. Waste handling is discussed in detail in Section 6.0 of the ESC Quality Assurance Manual. ESC's building information guides and site plan are shown in Appendix I.

### **6.2 LABORATORY SAFETY**

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods.
- ESC's laboratory safety guidelines are detailed in the *ESC Chemical Hygiene Plan*.

## **7.0 SAMPLING PROCEDURES**

### **7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING**

- Field Sampling procedure are described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Matrices for Wet Lab environmental analyses include groundwater, wastewater, drinking water, soil, and sludge. The Wet Lab also performs analyses on air filters for Industrial Hygiene monitoring of particulates.
- Sample containers, preservation methods and holding times vary depending on analyses requested. Please see the determinative procedures for specific directions.



## 8.0 EQUIPMENT

### 8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Wet Lab					
<i>This table is subject to revision without notice</i>					
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Instrument Name</i>	<i>Serial #</i>	<i>Location</i>
Analytical Balance	Mettler	AT200	Balance 1	m26291	Wet Lab
Analytical Balance	Mettler	AG204 Delta Range	Balance 2	118420883	Wet Lab
Analytical Balance	Mettler	XP205	Balance 3	1129420141	Wet Lab
Autoanalyzer	Lachat	Quikchem 8000	Lachat 2	A83000-1027	Wet Lab
Autoanalyzer	Lachat	Quikchem 8000	Lachat 3	A83000-1638	Wet Lab
Autoanalyzer	Lachat	Quikchem 8500	Lachat 4	60900000341	Wet Lab
Autoanalyzer	Lachat	Quikchem 8500	Lachat 5	60900000342	Wet Lab
Autoanalyzer	Lachat	Quikchem 8500	Lachat 6	70500000452	Wet Lab
Autoanalyzer - digester	Lachat	BD-46	DIG1	100700000-982	Wet Lab
Autoanalyzer - digester	Lachat	BD-46	DIG2	1000700000-982	Wet Lab
Autoanalyzer - digester	Lachat	BD-46	DIG1	1800-871	Wet Lab
Autoanalyzer - digester	Lachat	BD-46	DIG2	1800-872	Wet Lab
Automated titrator	Metrohm	855 titrosampler	Titrand	3256	Wet Lab
Centrifuge	Thermo	Megafuge 40	Centrifuge	41123868	Wet Lab
Class “T” weights	Troemner	Serial #7944		7944	Wet Lab
COD Reactor	HACH	45600	COD1	10800	Wet Lab
COD Reactor	HACH	45600	COD2	10090C0036	Wet Lab
Conductivity Meter	ORION	MODEL 170	ATI Orion	32470007	Wet Lab
Distillation Unit - Cyanide	Environmental Express	Distillation 1	LMD1920-106	2270	Wet Lab
Distillation Unit - Cyanide	Environmental Express	Distillation 2	LMD1920-106	2271	Wet Lab
Distillation Unit - Cyanide	Environmental Express	Distillation 3	LMD1920-106	2272	Wet Lab
Distillation Unit - Phenol	Westco Scientific	Model EASY-DIST	Dist 1	1062	Wet Lab
Distillation Unit - Phenol	Westco Scientific	Model EASY-DIST	Dist 2	1198	Wet Lab
Flash Point Tester	Koehler	Pensky-Martens K16200	Manual	R07002693B	Wet Lab

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Wet Lab</b>					
<i>This table is subject to revision without notice</i>					
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Instrument Name</i>	<i>Serial #</i>	<i>Location</i>
Flash Point Tester	Koehler	Pensky-Martens K16201	Manual	R07002510B	Wet Lab
Hot Plate	Thermolyne Fisher	Type 2200	Hot	16237	Wet Lab
Hot Plate	Thermolyne Fisher	Type 2200	Hot	16240	Wet Lab
Hot Plate	Cole Parmer	HS19 C-P	Hot Plate	50000073	Wet Lab
Ion Chromatograph	Dionex	ICS-2000	IC5	6050731	Wet Lab
Ion Chromatograph	Dionex	ICS 1500	IC6	8100010	Wet Lab
Ion Chromatograph	Dionex	ICS 1500	IC7	8100267	Wet Lab
Ion Chromatograph	Dionex	ICS 2000	IC8	8090820	Wet Lab
Ion Chromatograph	Dionex	ICS 2100	IC9	10060822	Wet Lab
Ion Chromatograph	Dionex	ICS 2100	IC10	10091285	Wet Lab
Ion Chromatograph	Dionex	ICS 2100	IC11	11012204	Wet Lab
Ion Chromatograph	Dionex	ICS 2100	IC12	12020460	Wet Lab
Ion Chromatograph	Thermo Fisher	ICS 1600	IC13	13031204	Wet Lab
Muffle Furnace	Thermolyne	(1) 30400	FURNACE	23231	Wet Lab
Autoanalyzer	OI Analytical	FS 3100	FS 3100-1	301831056 (NH3) 251833391 (CN)	Wet Lab
Autoanalyzer	OI Analytical	FS 3100	FS 3100-2	3168140781(NH3) 325833494 (CN)	Wet Lab
Autoanalyzer	OI Analytical	FS 3100	FS 3100-3	407831164 (NO2NO3) 403833925 (PHT)	Wet Lab
ORP Meter	YSI	ORP15	ORP	JC000114	Wet Lab
Oven - Drying	Blue M	Stabil-Therm	#1	NA	Wet Lab
Oven - Drying	Equatherm	D1576	#2	NA	Wet Lab
Oven - Drying	VWR	1305U	#3	4082804	Wet Lab
Oven - Drying	Equatherm	D1576	#4	10AW-3	Wet Lab
Oven - Drying	VWR	1305U	#5	4082104	Wet Lab
pH Meter	Fisher	AB15	AB15+	AB92329028	Wet Lab
pH Meter	Orion	410A	Orion	58074	Wet Lab
pH Meter	Fisher	AB15	AB15+	AB92325899	Wet Lab
pH Meter	Thermo Fisher	Orion Versa Star	Orion VS-1	V00659	Wet Lab
Refrigerated Recirculator	Polyscience	Recirculator	Recirculator1	1282	Wet Lab
Refrigerated Recirculator	Polyscience	Recirculator	Recirculator2	1608	Wet Lab
Spectrophotometer (UV/Vis)	Hach	DR 5000	DR5000-1	1381711	Wet Lab

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Wet Lab</b>					
<i>This table is subject to revision without notice</i>					
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Instrument Name</i>	<i>Serial #</i>	<i>Location</i>
Spectrophotometer (UV/Vis)	Hach	DR 5000	DR5000-2	1326829	Wet Lab
Total Organic Carbon Analyzer	Shimadzu	Model TOC-VWS	TOC2	39830572	Wet Lab
Total Organic Carbon Analyzer	Shimadzu	TOC-VCPH	TOC3	H51304435	Wet Lab
Total Organic Carbon Analyzer	OI-Analytical	Aurora 1030	TOC4	E141788082	Wet Lab
Total Organic Halogen Analyzer	Mitsubishi	TOX-100	TOX2	1035	Wet Lab
Total Organic Halogen Analyzer	Mitsubishi	AOX-200	AOX1	E7B00107	Wet Lab
Turbidimeter	Hach	2100N	Turbidimeter1	941100000903	Wet Lab

## 8.2 E EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

<i>INSTRUMENT</i>	<i>P. M. DESCRIPTION</i>	<i>FREQUENCY</i>
Analytical Balances	•Check with Class "I" weights	Daily
Analytical Balances	•Service/Calibration (semi-annual contract maintenance and calibration check)	Tolerance - $\pm 0.1\%$
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semi-annually
Refrigerators & Incubators	•Maintenance service	As needed - determined by daily temperature performance checks
Water Bath	•Check thermometer vs. NIST	Once/year
Water Bath	•Remove from service when not maintaining temperature and send off for repair or replace	As needed
Flash Point Tester	•Check thermometer vs. certified traceable	Once/year
Lachat Autoanalyzer	•Check pump tubes, change valve flares	At least 1/month
Pensky Martens	•Check fuel level, refill	As needed
Pensky Martens	•Clean cup thoroughly	Between each test and after use
TOC	•Maintain manufacturer's service contract	Renew each year
Turbidimeter - Hach 2100A	•Illumination lamp or window (alignment and/or replacement)	Erratic or poor response
pH Meters	•Reference junction & electrode replacement	As needed
pH Meters	•Probe stored in KCl	At all times when not in use
pH Meters	•Other	As described in the manufacturer's O & M manual
Ion Chromatograph	•Replace guard and analytical columns	As needed
Ion Chromatograph	•Replace the end-line filter (P/N 045987)	As needed

<i>INSTRUMENT</i>	<i>P. M. DESCRIPTION</i>	<i>FREQUENCY</i>
Ion Chromatograph	• Replace the pump piston rinse seals and piston seals	Every 6 months or as needed
Ion Chromatograph	• Replace the sampling tip and the tubing between the tip and the injection valve.	As needed
Ion Chromatograph	• Replace lines throughout the instrument	As needed
Ion Chromatograph	• Perform Preventive Maintenance using PM kit (P/N 057954)	Annual

### 8.3 S TANDARDS AND REAGENTS

Table 8.3A lists standard sources, receipt, and preparation information. Table 8.3B is designed to provide general calibration range information. These ranges may change depending on regulatory requirements, procedural changes, or project needs. Table 8.3C indicates the procedures and frequency for the standardization of laboratory solutions used for titrations.

<b>Table 8.3A: Standard sources, description and calibration information.</b>						
<i>This table is subject to revision without notice</i>						
<b>Instrument Group</b>	<b>Standard Source</b>	<b>How Received*</b>	<b>Source/Storage</b>	<b>Preparation from Source</b>	<b>Lab Stock Storage</b>	<b>Preparation Frequency</b>
Alkalinity, Acidity	Lab preparation	Acidity-matrix standard grade KHP	Room temp.	0.0500N	4°± 2°C	6 months
Ammonia-Nitrogen and Total Kjeldahl Nitrogen Primary Stock	Lab preparation	ACS grade NH4Cl	Room temp.	1,000ppm stock standard	Room temp.	Annually or sooner if check samples reveal a problem
Ammonia-Nitrogen and Total Kjeldahl Nitrogen	Lab preparation	Primary Stock	Room temp.	Working Standards	Not stored	Prepared fresh as needed
COD	Lab preparation	Acid grade KHP	Dessicator	Stock solution (10,000ppm)	4°± 2°C	When absorbance of curve changes or check samples are out of control
Cyanide (Autoanalyzer)	Lab preparation	KCN	Reagent shelf	Stock solution (1,000ppm)	4°± 2°C	6 months. Working dilutions prepared daily as needed
Fluoride Primary Stock	Inorganic Standard. NSI Lab preparation	ACS grade KF	Room temp.	100ppm stock solution	Room temp.	1 year or as needed when reference standard fails
Fluoride	Lab preparation	Primary Stock	Room temp.	Dilute standards	Not stored	Prepared fresh daily
Hardness	Lab preparation	Chelometric Std. CaCO <sub>3</sub>	Room temp.	1mg/mL as CaCO <sub>3</sub>	Room temp.	Annually or sooner if check samples reveal a problem
IC (Chloride, Nitrate, Nitrite, Bromide, Sulfate, Fluoride)	Commercial source	Varies	4°± 2°C	Working Standards as needed per analyte	4°± 2°C	6 months or sooner if check samples reveal a problem
IC (Chloride, Nitrate, Nitrite, Bromide, Sulfate, Fluoride)	Inorganic Standards	Varies	4°± 2°C	Working Standards as needed per analyte	4°± 2°C	Midpoint standard prepared weekly or sooner if necessary

<b>Table 8.3A: Standard sources, description and calibration information.</b>						
<i>This table is subject to revision without notice</i>						
<b>Instrument Group</b>	<b>Standard Source</b>	<b>How Received*</b>	<b>Source/Storage</b>	<b>Preparation from Source</b>	<b>Lab Stock Storage</b>	<b>Preparation Frequency</b>
IC (Chloride, Nitrate, Nitrite, Bromide, Sulfate, Fluoride)	NSI (2nd source)	Varies	4°± 2°C	Working Standards as needed per analyte	4°± 2°C	Prepared weekly or sooner if necessary
MBAS	Lab preparation	LAS Reference Material	4°± 2°C	1,000mg/mL working standards	4°± 2°C Wet Stored	6 months or when check standards are out of control. Prepared fresh.
Nitrite-Nitrate (autoanalyzer)	Lab preparation	ACS grade KNO3	Reagent shelf	Stock solution (1000ppm)	4°± 2°C	When absorbance of curve changes or check samples are out of control
pH Meter	Commercial Source	pH 4.0 Buffer	Room temp.	No prep required	NA	Annual/Expiration Date
pH Meter	Commercial Source	pH 7.0 Buffer	Room temp.	No prep required	NA	Annual/Expiration Date
pH Meter	Commercial Source	pH 10.0 Buffer	Room temp.	No prep required	NA	Annual/Expiration Date
Phenols (autoanalyzer)	Lab preparation	ACS Certified Phenol	Reagent shelf	Stock solution (1000ppm)	4°± 2°C	Every month. Working solutions prepared daily as needed.
Phosphate	(H <sub>2</sub> O) - Prepared in Lab Total Phos. (soils) RICCA, ERA	KH <sub>2</sub> PO <sub>4</sub>	Reagent shelf	Stock solution (50ppm as P)	Room temp.	When absorbance of curve changes or check samples are out of control. Working solutions prepared daily as needed.
Specific Conductivity Meter	NSI-Primary	ACS Certified KCl	Room temp.	Working Standard (0.01M)	Room temp.	As needed
Specific Conductivity Meter	ERA-2nd Source	ACS Certified KCl	Room temp.	Working Standard (0.01M)	Room temp.	As needed
Sulfate	Inorganic Standards, NSF Prepared in Lab	Anhydrous Na <sub>2</sub> SO <sub>4</sub>	Reagent shelf	Stock solution (100ppm)	Room temp.	When visible microbiological growth or check samples are out of control
Turbidimeter	Commercial Source Hach	Hach	Room temp.	No prep required	NA	Checked daily against Formazin Standards
pH Meter	Commercial Source	pH 1.0 Buffer	Room temp.	No prep required	NA	Annual/Expiration Date
pH Meter	Commercial Source	pH 13.0 Buffer	Room temp.	No prep required	NA	Annual/Expiration

<b>Table 8.3B: WORKING STANDARD CALIBRATION</b>	
<b>Analysis</b>	<b>Calibration Standard</b>
Alkalinity, Acidity- Titrimetric	Primary standard grade Na <sub>2</sub> CO <sub>3</sub> .
Alkalinity - Methyl orange Autoanalyzer	Primary standard grade Na <sub>2</sub> CO <sub>3</sub> ; 0, 10, 25, 50,100, 250, 375, 500 mg/L
Bromide IC	Range -1.0, 5.0, 10, 50, 100, mg/L
Chloride IC	Range -1.0, 5.0, 10, 50, 100, mg/L1
Conductivity	Standard KCl solution: 1413
Cyanides	Blank, 0.0025 – 0.40ppm. Distill one standard as check with each batch.
COD	KHP (Potassium hydrogen phthalate) standards 20 – 1000 mg/L
Chromium – Hexavalent (Colorimetric)	Blank, 0.0101, 0.0202, 0.0505, 0.1010, 0.2525, 0.5050, 1.010 mg/L

<b>Table 8.3B: WORKING STANDARD CALIBRATION</b>	
<b>Analysis</b>	<b>Calibration Standard</b>
Chromium – Hexavalent (IC)	Blank, 0.5, 1.0, 2.0, 10, 20, 50, 100 ug/L
Fluoride – IC	Range –0.10, 0.50, 1.0, 5.0, 10.0, mg/L
Hardness	CaCO <sub>3</sub> , chelometric standard.
Hardness (Colorimetric)	Range – 30, 50, 60, 100, 150, 200, 300 mg/L
MBAS	LAS reference material: 0.0, 0.1, 0.5, 1.0, 1.5, 2.0 mg/L
Nitrogen-Ammonia – Autoanalyzer	Calibration standards: 0, 0.10, 0.50, 1.0, 2.0, 5.0, 10, 20 mg/L
Nitrogen-Nitrate, Nitrite – Autoanalyzer	Blank, 0.1, 0.50, 1.00 5.0, 7.0, 10.0 mg/L
Nitrogen-Nitrate – IC	Range –0.10, 0.50, 1.0, 5.0, 10.0, mg/L
Nitrogen-Nitrite – IC	Range –0.10, 0.50, 1.0, 5.0, 10.0, mg/L
Orthophosphate, Total Phosphate	Blank, 0.025, 0.10, 0.25, 0.50, 0.75, 1.0mg/L diluted from standard KH <sub>2</sub> PO <sub>4</sub>
Perchlorate	Range – 0.5, 1.0, 3.0, 5.0, 10, 20, 25 mg/L
pH	Buffers 1.0, 4.0, 7.0, 10, 13
Phosphate, Total	Range – 0.0, 0.1, 0.5, 1.0, 2.5, 5.0 mg/L
Phosphate – IC	Range –0.10, 0.50, 1.0, 5.0, 10.0, 15.0, 20.0 mg/L
Phenols (chloroform ext.)	Blank 0.04, 0.05, 0.10, 0.50, 1.0, 2.0mg/L Distill one standard with each batch
Solids	Gravimetric balance calibrated charts, checked with Class “I” weights in range of sample tare weights.
Sulfate – IC	Range –1.0, 5.0, 10, 50, 100, 150, 200 mg/L
Sulfide (Colorimetric)	Range –0.0, 0.05, 0.1, 0.5, 1.0, 1.5, 2.0 mg/L
Sulfite	Titration
TKN	Range – 0.0, 0.1, 0.5, 1.0, 2.5, 5.0, 10, 20 mg/L
Turbidity	Range –0, 20, 200, 1000, 4000NTU
TOC	Range –0, 1.0, 2.5, 5.0, 7.5, 10, 20, 50, 75, 100 mg/L
TOX	Cell checks at 1, 20, 40 ug

<b>Table 8.3C: STANDARDIZATION OF TITRATION SOLUTIONS</b>		
<b>Solution Primary</b>	<b>Standard</b>	<b>Frequency</b>
0.0200 N NaOH	0.050 N KHP	Daily as needed
0.0200 N H <sub>2</sub> SO <sub>4</sub>	Freshly prepared and standardized NaOH (from KHP standard)	6 months or with each new batch
0.0141 N Hg (NO <sub>3</sub> ) <sub>2</sub>	Standard NaCl solution 500 ug Cl/ml	Daily as used
0.0100 M EDTA	Standard CaCO <sub>3</sub> solution 1 mg CaCO <sub>3</sub> /liter	Daily as used

## 8.4 I INSTRUMENT CALIBRATION

### Total Organic Carbon Analyzer (TOC) in GW/WW – SOP Number 340356A

The TOC standard curve is prepared using a minimum of five standards. Linear regression is used for quantitation with the correlation coefficient being at least 0.995. The calibration range is 1.0mg/L to 100mg/L. During the analytical sequence, the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recover within 10% of the expected value for each analyte.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within ±15% of the expected concentration.

**Total Organic Carbon Analyzer (TOC) in DW – SOP Number 340356B**

The TOC standard curve is prepared using a minimum of five standards. Linear regression is used for quantitation with the correlation coefficient being at least 0.995. The calibration range is 0.5mg/L to 5.0mg/L. During the analytical sequence, the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recover within 10% of the expected value for each analyte.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within  $\pm 15\%$  of the expected concentration. Dissolved organic carbon can be analyzed using this procedure by filtering the unpreserved sample using a 0.45um filter, then performing the analysis on the filtrate using the same process as the TOC procedure.

**Total Organic Carbon Analyzer (TOC) in Soil (Walkley Black) – SOP Number 340368**

The Walkley Black standard curve is prepared using a minimum of six standards. Linear regression is used for quantitation with the correlation coefficient being at least 0.995. The calibration range is 0.1mg/L to 5.0mg/L. During the analytical sequence, the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recover within 10% of the expected value for each analyte.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within  $\pm 50\%$  of the expected concentration. This method is used to determine Fractional Organic Carbon (FOC) as required by the state of Indiana.

**Total Organic Halogen Analyzer (TOX) – SOP Number 340360**

The cell performance of the TOX analyzer is verified at the beginning of each analytical sequence in the low, mid and high ranges. The verifications must recover within 3% of the expected target value. The instrument performs a linear regression using the values determined with the required correlation coefficient being at least 0.995. During the analytical sequence, the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recover within 10% of the expected value for each analyte.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within  $\pm 15\%$  of the expected concentration.

### **Anions by Ion Chromatography – SOP 340319**

Least Squares Linear Regression is the primary method of quantitation; where a minimum of five standards is used and the correlation coefficient must be at least 0.995 for each analyte of interest. The calibration range varies depending upon the analyte(s) to be determined. During the analytical sequence, the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recover within 10% of the expected value for each analyte, except during the analysis of groundwater and soil using EPA Method 9056 that must recover within 5%.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within  $\pm 10\%$  for water samples and  $\pm 15\%$  of the expected concentration for soil samples.

### **Hexavalent Chromium by Ion Chromatography – SOP 340372 & 340372A**

These procedures are utilized to analyze for hexavalent chromium (Cr<sup>6+</sup>) by ion chromatography using a variety of published methods and the relevant SOP addresses both the common and method specific requirements for each published method. The Cr<sup>6+</sup> standard curve is prepared using a minimum of five or six standards at various levels depending on the expected concentration of the field samples, the analytical method requested and the matrix. Linear regression is used for quantitation with the correlation coefficient being at least 0.995. During the analytical sequence, the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The recovery of the CCV must be within 10% of the expected value for the analyte using SM 3500Cr C and EPA Method 7199. The CCV for EPA 218.6 must recover within  $\pm 5\%$  and the mid-level CCV for EPA 218.7 must recover within  $\pm 15\%$ .

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within  $\pm 10\%$  of the expected concentration.

Aqueous and solid Hexavalent Chromium samples can also be analyzed by colorimetry using EPA 7196A and SM 3500Cr B – SOP 340318B & 350318C. Specific requirements for those methods are contained within the specified SOPs. Soil samples are prepared for both the IC and colorimetric method using alkaline digestion found in EPA 3060A and discussed in both soil SOPs 350318C and 340372A.



### **Gravimetric Analyses – Various SOPs**

Gravimetric analyses are performed using several different published methods, including TDS, TSS, TVDS, TS, TVS, VSS, Settleable Solids, Total Particulates, and Respirable Particulates. Calibration for these methods require use of Class I weights and a properly performing and verified balance. Where possible, laboratory control standards are analyzed in conjunction with field sample analysis to verify that the analytical process is performing accurately. Sample duplicate analyses also provide verification that the analytical process is performing as required.

### **Auto-Analyzer (Lachat) – Various SOPs**

The Autoanalyzer calibration curve is prepared using a minimum of five standards. For most analyses, linear regression is used for quantitation with the correlation coefficient being at least 0.995. The calibration range varies depending upon the analyte to be determined. During the analytical sequence, the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. Routinely, the CCV must recover within 10% of the expected value for each analyte, but is dependent on the analyte of concern, the matrix of the sample and the determinative method.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within  $\pm 15\%$  of the expected value, except for cyanide, ammonia, total phosphorus, NO<sub>2</sub>NO<sub>3</sub> and TKN where  $\pm 10\%$  applies.

### **Perchlorate in Drinking Water – ESC SOP 340370**

The Ion Chromatograph calibration curve is prepared using a minimum of five standards. The instrument performs a linear regression using the values determined with the required correlation coefficient being at least 0.995. During the analytical sequence, the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recover within 15% of the expected value for each analyte.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within  $\pm 10\%$  of the expected concentration.

### 8.5 A ACCEPTANCE/REJECTION OF CALIBRATION

All new standard curves are immediately checked with a laboratory control standard from a separate source than that used for calibration. All curves are visually reviewed to ensure that acceptable correlation represents linearity. Calibration curves may be rejected for nonlinearity, abnormal sensitivity, or poor response of the laboratory control standard. Specific criteria for each instrument are outlined in Table 8.5.

Continuing calibration is performed following every tenth sample. If a check standard does not perform within established criteria then the instrument is evaluated to determine the problem. Once the problem is corrected, all samples between the last “in control” sample and the out of control check are re-analyzed.

**TABLE 8.5: INSTRUMENT CALIBRATION**

Instrument (Analysis)	Calibration Type	Number of Standards	Type of Curve	Acceptance/Rejection Criteria	Frequency
pH Meter*	Initial	5 (buffers) 1 reference buffer	Log.	Third pH of a different value buffer must read within 0.05 units of true value	Daily as used
	Continuing	1 buffer (may be any certified buffer)		Buffer solution must read within 0.05 units of true value	Every 10th sample; Field**
Conductivity Meter*	Initial	1	1 point	Calculation of cell constant between 0.95 - 1.05	Daily as used
	Continuing	1		Must be within 5% of true value	Every 10th sample; Field**
Turbidimeter *	Initial	5	Linear	Formazin-confirmed Gelex standards in appropriate range. Check with second standard must be within 5%	Daily as used
	Continuing	1 reference of different value, 1 (high-level)		Must be within 5% of true value	Every 10th sample; Field**
UV/VIS Spec.	Initial	At least 5 standards calibration standards	Linear	Calibration Curve must have a correlation of 0.995 or better	Daily as used
	Continuing	2 laboratory control standard 1 mid-level reference std.		Must be within $\pm 15\%$ of the calibration curve. Must be within 90 – 110%	Daily as used Every 10th sample
Total Organic Halogen Analyzer	Initial	3 calibration standards	Linear	Calibration Curve must have a correlation of 0.995 or better	Daily as used
	Continuing	1 laboratory control standard 1 mid-level reference std.		Laboratory control standard must agree within $\pm 15\%$ of calibration curve Must be within 90 – 110%	Daily as used Every 10th sample
Total	Initial	5 calibration	Linear	Calibration Curve must have a	Every 6

Instrument (Analysis)	Calibration Type	Number of Standards	Type of Curve	Acceptance/Rejection Criteria	Frequency
Organic Carbon Analyzer	Continuing	standards 2 laboratory control standard 1 mid-level reference std.		correlation of 0.995 or better Laboratory control standard must agree within $\pm 15\%$ of calibration curve Must be within 90 – 110%	months or as needed Daily as used Every 10th sample

Note: ESC defines a "laboratory control standard" as a standard of a different concentration and source than those stock standards used for calibration.

\*This equipment is also calibrated and used in the field.

\*\*Field equipment must be checked every 4 hours and at the end of the day.

## 9.0 LABORATORY PRACTICES

### 9.1 R REAGENT GRADE WATER

Reagent grade water is obtained from either a Barnstead NANOpure Diamond system or the Millipore Milli-Q Academic A-10 system.

### 9.2 G GLASSWARE WASHING AND STERILIZATION PROCEDURES

#### General

Routine laboratory glassware is washed in a non-phosphate detergent and warm tap water. Before washing all labeling and large deposits of grease are removed with acetone. Glassware is then rinsed with: tap water, "No Chromix" solution, tap water, and deionized (DI) water. Glassware is stored in designated drawers or on shelves, inverted when possible. All glassware is rinsed with the required solvent, prior to use. DI water is then used as a precaution against airborne contamination

#### Phosphate Glassware

Glassware involved in phosphate analysis is marked and segregated. All labels and markings are removed from the glassware prior to washing. The glassware is then washed using hot water and a non-phosphorus detergent. It is then rinsed thoroughly in hot water followed by a rinse in DI water. It is rinsed in 1:1 HCl followed by a final rinse of DI water. If the phosphate glassware has not been used recently, it is the responsibility of the analyst to rinse the glassware with warm 1+9 hydrochloric acid prior to use.

**Nutrients and Minerals Glassware**

All labels and markings are removed from the glassware prior to washing. The glassware is then washed using hot water and detergent. It is then rinsed thoroughly in hot water followed by a rinse in DI water. It is rinsed in 1:1 HCl followed by a final rinse of DI water.

Immediately prior to use, the ammonia glassware is rinsed in DI water. Routine blanks are run on ammonia glassware to ensure that the detergent is contaminant free.

**Non-Metals (CN, COD) Glassware**

All labels and markings are removed prior to washing. The glassware is soaked in hot soapy water followed by a thorough rinse with hot tap water. A final rinse of DI water is then performed.

**10.0 ANALYTICAL PROCEDURES**

10.1 A list of laboratory SOPs associated with the Wet Lab can be found in the following table:

**TABLE 10.1: WET LAB DEPARTMENT SOPs**

*This table is subject to revision without notice*

SOP #	Title
340300	Acidity (SM 2310B)
340301	Alkalinity (Titrimetric)
340302	Alkalinity - Lachat
340305	Chlorine, Total Residual DPD- 330.5 SM4500-CL-G
340307	Cyanide- All Forms (Colorimetric Automated UV) - Lachat
340307	Cyanide- OI Method
340309	Chemical Oxygen Demand
340310	Color by Visual Comparison (SM2120B, EPA 110.2)
340313	Density (Specific Gravity)
340317	Total Hardness (mg/l as CaCO3) - (Titrimetric)
340317	Total Hardness by Lachat Method 130.1
340318	Hexavalent Chromium (Colorimetric) Soil 3060A/7196A
340318	Hexavalent Chromium (Colorimetric) Water 7196A
340319	Ion Chromatography - Anions by 300.0, SM 4110B and 9056/9056A
340319	Ion Chromatography - Anions OH VAP
340325	MBAS (Methylene Blue Active Substances)
340327	Ammonia, Phenolate (OI)
340327	Ammonia, Phenolate (Lachat)
340328	Organic Nitrogen
340331	Threshold Odor Test
340333	Nitrate/Nitrite (Lachat Autoanalyzer)
340333	Nitrate/Nitrite (OI Autoanalyzer)
340334	Paint Filter Test

SOP #	Title
340335	pH/Corrosivity
340336	Phenol - 4AAP (Lachat Autoanalyzer)
340338	Total Phos GW/WW (365.4) Colorimetric
340338	Total Phos.( 361.2, 4500P-B/F) Colorimetric
340338	Orthophosphate (365.2,4500P-E) Colorimetric
340339	Reactivity
340340	Reactive Cyanide/Sulfide Distillation
340342	Specific Conductance (120.1, 2510B)
340344	Sulfide (Colorimetric Methylene Blue) (376.2)
340344	Sulfide Acid-soluble,and acid-insoluble Method 9034
340345	Sulfite
340346	Settleable Solids
340347	Total Dissolved Solids
340348	Total Suspended Solids (Non-Filterable Residue)
340349	Total Solids/Percent Moisture
340350	Total Volatile Solids
340352	Total Kjeldahl Nitrogen
340354	Turbidity
340356	Total Organic Carbon In Soils (loss of weight on ignit.
340356	TOC for Drinking Water only
340356	Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC) using Shimadzu 5000A for GW and WW
340357	Ignitability
340359	UV254
340360	TOX (total organic halides)
340361	Ferrous Iron, SM-3500-Fe-B
340362	Heat of Combustion
340365	Particles Not Otherwise Regulated, Total (PNOR) NIOSH 0500
340366	Oxidation Reduction Potential
340367	Extractable Organic Halides
340368	TOC in Soil (Walkley-Black)
340369	Carbon Dioxide by Calculation
340370	Perchlorate in DW
340371	Chlorine in Oil (ASTM D808-00)
340372	Hexavalent Chromium in Soil by IC (3060A/7199)
340372	Hexavalent Chromium in Water by IC (218.7/SM 3500Cr)
340373	Organic Matter (FOM) and Fractional Organic Carbon (FOC)
340374	Total Volatile Dissolved Solids (TVDS)
340375	Hexavalent Chromium in Air by IC
340376	Total Organic Halides in Oil (EPA 9076)
340377	Manual Nitrocellulose Analysis
340378	Volatile Suspended Solids
340379	Guanidine Nitrate by IC
340381	Ash in Petroleum Products (ASTM D482-07)

## 11.0 QUALITY CONTROL CHECKS

**NOTE:** For specific guidance on each determinative method, including required quality control and specific state requirements/modifications, refer to the relevant laboratory standard operating procedure(s).

- 11.1 ESC participates in proficiency testing (PTs) in support of various laboratory accreditations/recognitions. Environmental samples are purchased from Phenova. The WS, WP and solid matrix studies are completed every 6 months.
- 11.2 Initial Demonstrations of Capability (IDOCs) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability (CDOCs) must be updated at least annually. The associated data is filed within the department and available for review.
- 11.3 Where appropriate, Matrix Spike and Matrix Spike Duplicates are performed on each batch of samples analyzed, depending on analytical method requested.
- 11.4 A Laboratory Control Sample (LCS) is analyzed once per batch of samples. Where appropriate, an LCS Duplicate may also be analyzed.
- 11.5 Where appropriate, a method preparation blank is performed per batch of samples processed. If one-half the reporting limit [RL] is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary, based on the following criteria:
  - The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch or
  - The blank contamination is greater than 1/10 of the specified regulatory limit.The concentrations of common laboratory contaminants shall not exceed the reporting limit. Any samples associated with a blank that fail these criteria shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in non-detected results for the failing analytes.

## 12.0 DATA REDUCTION, VALIDATION AND REPORTING

### 12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in ESC SOP #030201, *Data Handling and Reporting*. A secondary review of the data package using the ESC SOP #030227, *Data Review*. The reviewer verifies that the analysis has been performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required qualifiers on test reports, etc.)

**TABLE 12.1: Data Reduction Formulas**

PARAMETER FORMULA	
Acidity, Alkalinity	$\frac{\text{mL titrant} \times \text{normality titrant} \times 50,000}{\text{mL sample}}$
COD, Sulfate	Concentration from curve x dilution factor
Orthophosphate, Hexavalent Chromium	Calculated by computer software as provided by HACH Corp.
Nitrogen-Nitrate, Phenols, Nitrogen-Ammonia, Total Phosphate, Nitrogen-Total Kjeldahl**	Calculated by computer software as provided by Lachat Corp.
Anions, Hexavalent Chromium	Calculated by computer software as provided by Dionex
Conductivity*, pH, Turbidity,	Directly read from instrument
Cyanide, Total and Amenable	$\frac{\mu\text{g from standard curve} \times \text{mL total volume absorbing solution}}{\text{mL volume sample} \times \text{mL volume of absorbing solution colored}}$ <i>Calculated by software as provided by Lachat Corp.</i>
Solids, Total and Total Dissolved	$\frac{((\text{mg wt of dried residue} + \text{dish}) - \text{mg wt of dish}) \times 1000}{\text{mL sample}}$
Solids, Total Suspended	$\frac{((\text{mg wt of dried residue} + \text{filter}) - \text{mg wt of filter}) \times 1000}{\text{mL sample}}$

## 12.2 V ALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.3 by method for current QC targets, controls and current reporting limits.

## 12.3 R EPORTING

Reporting procedures are documented in *SOP 030201 Data Handling and Reporting*.

*Inorganic Control Limits:* Inorganic QC targets are statutory. The laboratory calculated limits verify the validity of the regulatory limits. The Wet Lab QC targets for all inorganic analyses are within the range of  $\pm 5$  to 15% for accuracy, depending on determinative method requirements, and, where applicable,  $\leq 20$  RPD for precision, unless laboratory-generated data indicate that tighter control limits can be routinely maintained. When using a certified reference material for QC sample analysis, the acceptance limits used in the laboratory will conform to the provider's certified ranges for accuracy and precision.

**Table 12.3A: QC Targets for Wet Lab Accuracy (LCS), Precision and RLs**

This table is subject to revision without notice

Analyte An	alysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppm)
Acidity	SM 2310B	w	85 - 115	<20	10
Acidity	SM 2310B	s	85 - 115	<20	10
Alkalinity	SM 2320B	w	85 - 115	<20	20

**Table 12.3A: QC Targets for Wet Lab Accuracy (LCS), Precision and RLs**

This table is subject to revision without notice

Analyte An	alysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppm)
Ammonia	350.1, SM 4500-NH3-B	w	90 - 110	<20	0.25
Ammonia	350.1 (mod.)	s	Certified Values	<20	5.0
Ash	ASTM D482-07	s	90 - 110	<20	n/a
Bromide	300.0/9056/9056A/SM 4110B	w	90 - 110	<20	1.0
Bromide	9056/9056A	s	Certified Values	<20	10
Chloride	300.0/9056/SM 4110B	w	90 - 110	<20	1.0
Chloride	9056A	w	90 - 110	<15	1.0
Chloride	300.0/9056	s	Certified Values	<20	10
Color	SM 2120B	w	n/a	<20	1 CU
Conductivity	120.1/9050A, 2510B	w	85 - 115	<20	n/a
Cyanide	335.4, 335.2 (CLP-M), 9012B, SM 4500-CN-E	w	90 - 110	<20	0.005
Cyanide	335.2 (CLP-M), 9012B	s	Certified Values	<20	0.25
Ferrous Iron	3500FE B	w	85 - 115	<20	15
Fluoride	300.0/9056/9056A/SM 4110B	w	90 - 110	<20	100
Fluoride	9056A	s	Certified Values	<20	1.0
Hardness	130.1	w	85 - 115	<20	30
Hardness	130.2/SM 2340C	w	85 - 115	<20	5.0
Hexavalent Chromium	SM3500 Cr B/7196A	w	85 - 115	<20	10
Hexavalent Chromium	7196A	s	Certified Values	<20	2.0
Hexavalent Chromium	7199	w	90 - 110	<20	0.50
Hexavalent Chromium	218.7	w	85 - 115	<15	0.02
Hexavalent Chromium	7199	s	80 - 120	<20	1.0
Ignitability	1010A	w/s	±3 degrees C	<20	n/a
Methylene Blue Active Substances	5540C SM20 <sup>th</sup>	w	85 - 115	<20	0.10
Nitrate-Nitrite	300.0/9056/9056A/SM 4110B	w	90 - 110	<20	1.0
Nitrate-Nitrite	9056A	w	90 - 110	<15	1.0
Nitrate-Nitrite	300.0/9056	s	Certified Values	<20	10
Nitrate	300.0/9056/SM 4110B	w	90 - 110	<20	0.1
Nitrate	9056A	w	90 - 110	<15	0.1
Nitrate	300.0/9056	s	Certified Values	<20	1.0
Nitrite	300.0/9056/SM 4110B	w	90 - 110	<20	0.1



**Table 12.3A: QC Targets for Wet Lab Accuracy (LCS), Precision and RLs**

This table is subject to revision without notice

Analyte An	alysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppm)
Nitrite	9056A	w	90 - 110	<15	0.1
Nitrite	300.0/9056	s	Certified Values	<20	1.0
pH	SM 4500-H, 9040C	w	n/a	<1	n/a
pH	9045D	s	n/a	<1	n/a
Phosphate (ortho)	SM 4500-P E	w	85 - 115	<20	25
Phosphate (ortho)	SM 4500-P E	s	85 - 115	<20	250
Phosphorous/Total	365.1, SM 4500-P	w	90 - 110	<20	3.0
Phosphorous/Total	365.4	w	90 - 110	<20	100
Phosphorous/Total	9056	s	Certified Values	<20	1.0
Residual Chlorine	SM 4500Cl G	w	90 - 110	<20	0.1
Residue, Total (TS)	SM 2540-B, SM2540-G	w	85 - 115	<20	10
Residue, Total (TS)	SM2540-G	s	85 - 115	<20	100
Residue, Filterable (TDS)	SM 2540-C	w	95 - 105	<20	10
Residue Non-Filterable (TSS)	SM 2540-D	w	95 - 105	<20	2.5
Residue, Total Volatile (TVS)	SM 2540-E	w	80 - 120	<20	1.0 (% of TS)
Residue, Total Volatile (TVS)	160.4/SM 2540-E,	s	80 - 120	<20	1.0 (% of TS)
Sulfate	300.0/9056/SM 4110B	w	90 - 110	<20	5.0
Sulfate	9056A	w	90 - 110	<15	5.0
Sulfate	300.0/9056	s	Certified Values	<20	50
Sulfide	SM 4500S2 D	w	85 - 115	<20	20
Sulfite	SM 4500SO3 B	w	85 - 115	<20	3.0
Total Kjeldahl Nitrogen	351.2	w	90 - 110	<20	0.25
Total Kjeldahl Nitrogen	SM 4500NOrg C	s	Certified Values	<20	20
Total Organic Carbon	415.1, SM 5310B,9060A	w	85 - 115	<20	1.0
Total Organic Carbon	SM 5310C	w	85 - 115	<20	0.5
Total Organic Carbon	USDA LOI, ASTM F1647-02A	s	50 - 150	<20	10
Total Organic Carbon	Walkley-Black,	s	50 - 150	<20	100
Dissolved Organic Carbon	415.1, SM 5310B,9060A	w	85 - 115	<20	1.0
Dissolved Organic Carbon	SM 5310C	w	85 - 115	<20	0.5
Total Organic Halogens	9020A, SM 5320B	w	85 - 115	<20	0.1
EOX	9023	s	85 - 115	<20	20000

**Table 12.3A: QC Targets for Wet Lab Accuracy (LCS), Precision and RLs**

This table is subject to revision without notice

Analyte An	alysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppm)
Total Phenol	420.2	w	90 - 110	<20	0.04
Total Phenol	9066	w	90 - 110	<20	0.04
Total Phenol	9066	s	90 - 110	<20	0.67
Turbidity	180.1, SM 2130B	w	90 - 110	<20	0.1 NTU

**Table 12.3B: QC Targets for IH Accuracy (LCS), Precision and RLs**

This table is subject to revision without notice

Analyte An	alysis Method	Matrix	Duplicate Precision (% RPD)	RL
Total Dust	NIOSH 0500	Filters	<5.0	0.010mg/filter
Respirable Dust	NIOSH 0600	Filters	<5.0	0.010mg/filter

### 13.0 CORRECTIVE ACTIONS

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CARs are kept on file by the Regulatory Affairs Department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

#### 13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these control limits are exceeded, corrective action must be taken. Calculated sample spike control limits are also used.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP.

##### 13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory, the method criteria takes precedence.

### 13.2.2 Calibration Verification Criteria Are Not Met: Inorganic Analysis

Rejection Criteria - See Table 8.5.

Corrective Action - If a standard curve linearity is not acceptable and/or the absorbance for specific standard(s) is not analogous to historic data, the instrument settings, etc. are examined to ensure that nothing has been altered, clogged, etc. Check the standard curve for linearity and re-analyze the standards once. If the failure persists, the working standards are made fresh, intermediate dilutions are re-checked and the instrument is re-calibrated. If a problem persists, the Supervisor or Regulatory Affairs Department is notified for further action.

If the initial reference check sample is out of control, the instrument is re-calibrated and the check sample is re-analyzed. If the problem continues the check sample is re-prepared. If the problem still exists then the standards and reagent blank are re-prepared. If the problem persists, the Supervisor or Regulatory Affairs Department is notified for further action.

### 13.2.3 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

Rejection Criteria - Blank reading is more than twice the background absorbance or more than 1/2 RL.

Corrective Action - Blanks are re-analyzed and the response is assessed. Standard curves and samples are evaluated for any obvious contamination that may be isolated or uniform throughout the run. If necessary, reagents are re-prepared. Field sample analyses are not started until the problem is identified and solved. If samples have already been partially prepared or analyzed, the Supervisor or Regulatory Affairs Department is consulted to determine if data needs to be rejected or if samples need to be re-prepped.

### 13.2.4 Out Of Control Laboratory Control Standards (LCS)

Rejection Criteria - If the performance of associated laboratory control sample(s) is outside of control limits either method defined or calculated as the mean of at least 20 data points  $\pm$  3 times the standard deviation of those points. (Listed in Section 12).

Corrective Action - Instrument settings are checked, LCS standard is re-analyzed. If the LCS is still out of control, re-calibration is performed, and samples affected since the last "in control" reference standard are re-analyzed. The Supervisor or Regulatory Affairs Department is consulted for further action.

### 13.2.5 Out Of Control Matrix Spike Samples

Rejection Criteria - If either the MS or MSD sample is outside the established control limits.

Corrective Action - Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance, however, is based on

method blank and LCS performance, not on MS/MSD recoveries. Specific methods, customers, and programs may require further corrective action in some cases.

#### 13.2.6 Out Of Control Duplicate Samples

Rejection Criteria - Lab-generated or method required maximum RPD limit (as listed under precision in Section 12)

Corrective Action - Instrument and samples are checked to see if precision variance is likely (i.e., high suspended solids content, high viscosity, etc.). They are re-analyzed in duplicate and samples just preceding and following the duplicated sample are re-analyzed. If problem still exists, the Supervisor or Regulatory Affairs Department is notified to review the analytical techniques.

#### 13.2.7 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

Corrective Action - Instrument settings are checked, calibration verification standard is reanalyzed. If the standard is still out of control, re-calibration is performed, and samples affected since the last “in control” reference standard are re-analyzed. The Supervisor or Regulatory Affairs Department is consulted for further action.

### 14.0 RECORD KEEPING

Record keeping is outlined in SOP #030230, *Standards Logger*, SOP #030227, *Data Review* and SOP #030201, *Data Handling and Reporting*

All calibration data and graphs generated for wet chemistry are kept digitally with the following information: date prepared, calibration concentrations, correlation, and analyst initials. The analyst reviews the calibration and evaluates it against acceptance criteria before placing it in the calibration notebook. Data on initial and continuing reference standards, as well as matrix spikes and duplicates, are entered in the QC box generated on each analysis page. If a test allows the use of a previously established calibration curve then the calibration check standard is reviewed against acceptance criteria and if acceptable, analysis can proceed. In this situation the calibration date is referenced so that the curve can be easily reviewed, if necessary.

## 15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 13.0 and SOP #010104, *Internal Audits*.

## 16.0 REVISIONS

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of this appendix are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0 (Appendix IV)	Section 5.1 – Removed language about supervisor and backup reviewing and approving all data for Wet Chem Section 8.4 – Revised calibration range for TOC in DW and LCS limits for perchlorate in DW Section 8.5 – Removed language about comparing new curves to previous curves and within 10% criteria Section 13.2.5 – Reworded MS/MSD criteria and revised corrective action to just qualify unless method, customer, or program states to do something else. Section 16 – New section for summary of revisions to previous version

1.0 SIGNATORY APPROVALS

# Metals Department QUALITY ASSURANCE MANUAL

## APPENDIX V TO THE ESC QUALITY ASSURANCE MANUAL

for

ESC LAB SCIENCES  
12065 LEBANON ROAD  
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(615) 758-5858

Prepared by

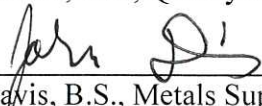
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**NOTE: The QAM has been approved by the following people.**

  
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### **3.0 SCOPE AND APPLICATION**

This appendix discusses specific QA requirements for general analytical protocols to ensure that data generated from the Metals Laboratory is scientifically valid and is of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

### **4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES**

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling guidance and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual*.

### **5.0 PERSONNEL AND TRAINING**

#### **5.1 PERSONNEL**

John Davis, with a B.S. degree in Biology, is the Department Supervisor and is responsible for the overall production of these laboratories; including the management of the staff and scheduling. Mr. Davis has 12 years of environmental laboratory experience. In his absence, Rodney Street assumes responsibility for departmental decisions in the Metals Department.

Mr. Rodney Street, with a B.S. degree in Medical Technology, is the Primary Analyst for the Metals Lab. He is proficient in inorganic analytical methods and has 34 years of environmental laboratory experience.

#### **5.2 TRAINING**

Senior Analysts or the Supervisor trains all new analysts to the laboratory according to ESC protocol. ESC's training program is outlined in *SOP 030205 Technical Training and Personnel Qualifications*. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in metals analysis and preparation is also demonstrated by acceptable participation in multiple proficiency testing programs (PTs) and using daily Quality Control sample analyses. Documentation of analyst training is maintained on file within the department.



## 6.0 FACILITIES AND LABORATORY SAFETY

### 6.1 FACILITIES

The main area of the analysis laboratory has approximately 1200 square feet with roughly 90 square feet of bench area. The main area of the metals prep laboratory has approximately 1200 square feet with 232 square feet of bench area. The main area of the Mercury/TCLP laboratory has approximately 1272 square feet with 136 square feet of bench area. The lighting standard in all three labs is fluorescence. The air system is a 15-ton make-up unit plus 15-ton HVAC with electric heat. The laboratory reagent water is provided through the US Filter deionizer system. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's waste disposal company. ESC's building information guides and site plan are shown in Appendix I.

### 6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods.
- ESC's laboratory safety guidelines are detailed in *the ESC Chemical Hygiene Plan*.

## 7.0 SAMPLING PROCEDURES

### 7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Matrices for metals analysis are as follows: groundwater, wastewater, drinking water, soil, sludge, paint chips, wipes, filters, and leachates.
- Sample containers, preservation methods and holding times:
  - Glass containers are acceptable for all elements except Boron and Silicon. Plastic must be used for Boron and Silicon.
  - Water samples that are analyzed for dissolved metals must be filtered using a 0.45 $\mu$ m pore membrane. Water samples for total metals are not filtered. All water samples are acidified with 1+1 nitric acid to a pH<2. Filtered water samples (dissolved metals) are preserved immediately after filtration. All other water samples are preserved immediately after sampling. Water samples are not refrigerated prior to analysis.
  - Paint chips, dust wipes and filters do not require preservation.
  - Soil samples for mercury are stored not frozen but  $\leq 6^{\circ}\text{C}$ . All other soil samples for metals analysis do not require temperature preservation.
  - Hold times for all metals, except Mercury, are 180 days. Mercury has a hold time of 28 days.

## 8.0 EQUIPMENT

### Instrument Software

- Agilent ICPMS 7700 and 7900 - Mass Hunter - Used for calibration, calculation, QC review, diagnostics, and data storage
- Perkin Elmer ICP Optima DV - PE - ICP Winlab - Used for calibration, calculation, qc review, diagnostics, data storage
- Thermo 7400 ICP - Qtegra - Used for calibration, calculation, QC, review, diagnostics, data storage
- Leeman Hydra II AA – Envoy - Used for calibration, calculation, QC review, diagnostics, data storage
- Perkin Elmer Fims 100- Winlab- Used for calibration, calculation, QC review, diagnostics, data storage

**NOTE:** All purchased software that is used in conjunction with software specific instruments is guaranteed by the supplier to function as required. The supplier of the software performs all troubleshooting or software upgrades and revisions.

## 8.1 EQUIPMENT LIST

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Metals Analysis and Preparation</b>						
<i>This table is subject to revision without notice</i>						
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Name</i>	<i>#</i>	<i>Serial number</i>	<i>Location</i>
Balance- Top Loading	Trobal	AGN100		1	701001026	Metals Prep Lab
Balance - Top Loading	Mettler Toledo	PB3002-5		1	1119070828	Metals Prep Lab
Balance - Top Loading	Mettler Toledo	PB3002-5		1	71242213216	Mercury Lab
Balance - Top Loading	Mettler Toledo	PB3002-5		1	1121462199	Metals Prep Lab
Hot Block	Env. Express	SC154	C	1	3994CEC1880	Metals Prep Lab
ICPMS with autosampler	Agilent	7700	ICPMS7	1	JP12482187	Metals Lab
ICPMS with autosampler	Agilent	7900	ICPMS8	1	JP14080166	Metals Lab
ICPMS with autosampler	Agilent	7900	ICPMS9	1	JP14400452	Metals Lab
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 5300DV ASX-510	ICP5	1	077N5041802	Metals Lab
ICP Simultaneous with autosampler	Thermo	7400	ICP12	1	IC74DC141801	Metals Lab
ICP Simultaneous with autosampler	Thermo	7400	ICP13	1	IC74DC143804	Metals Lab
ICP Simultaneous with autosampler	Thermo	7400	ICP14	1	IC74DC151103	Metals Lab
Hot Block	CPI	Mod Block	HGA	1	004412	Mercury Lab
Hot Block	CPI	Mod Block	HGB	1	604443	Mercury Lab

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Metals Analysis and Preparation</b>						
<i>This table is subject to revision without notice</i>						
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Name</i>	<i>#</i>	<i>Serial number</i>	<i>Location</i>
Hot Block	CPI	Mod Block	MPA	1	4430	Metals Prep Lab
Hot Block	CPI	Mod Block	MPB	1	4434	Metals Prep Lab
Mercury Auto Analyzer	Perkin Elmer	(1) FIMS 100	III	1	110156051101	Mercury Lab
Mercury Auto Analyzer	Perkin Elmer	FIMS 100	IV	1	101S11061403	Mercury Lab
Mercury Auto Analyzer	Leeman	Hydra II AA	HG5	1	Install #65043	Mercury Lab
Microwave	CEM	MARS Xpress	NA	1	MD-2861	Metals Prep Lab
Microwave	CEM	MARS Xpress	NA	1	MD-9972	Metals Prep Lab
Microwave	CEM	MARS Xpress	NA	1	MD-9640	Metals Prep Lab
Microwave	CEM	MARS Xpress	NA	1	MD-4692	Metals Prep Lab
Microwave	CEM	MARS 6	NA	1	MJ2771	Metals Prep
Prep Station	Env. Express	Automated prep station	Autoblock 3	1	AB1002-0708-001	Metals Prep Lab
TCLP Extraction Unit	Env. Express	6 Position	NA	1	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	5	4803-12-542	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	5	1918-12-415	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	5	1918-12-414	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	5	5152-12-548	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	2	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	2	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	2	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	2	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	2	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	10 Position	NA	1	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	Teflon Vessels	NA	12	NA	TCLP Lab
TCLP Zero Headspace Extractor	Env. Express	Vessels	NA	41	NA	TCLP Lab
Centrifuge	Thermo	Sovall ST40	NA	1	41179863	Metals Prep
Turbidimeter	HACH	2100N		1	05090C020685	Metals Prep Lab
Water Purification - Nanopure	Barnstead	D11951		1	1372051120948	Metals Prep Lab
PH Meter	Orion Versastar	VSTAR50	NA	1	V04967	TCLP Lab
Balance	Mettler Toledo			1	B246522879	TCLP Lab
Auto pipettors 1000µl to 20 µl	Oxford	Varies	NA		NA	Metals Lab
Auto pipettors	Eppendorf, Oxford	Varies	NA		NA	Metals Prep Lab
MAX/MIN Thermometer	Fischer Scientific	MAX/MIN	TCLP #1		122376671	TCLP Lab

## 8.2 E EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
ICP	•Maintain manufacturer's service contract	Renew annually
ICP and ICPMS	•Pump tubing, torch alignment, o-ring, injector tip and torch	Check daily and adjust/change as needed
ICPMS	•Sampler and Skimmer cones	Clean or replace when needed
ICP and ICPMS	•Pump rollers	Clean and lubricate when needed
ICP and ICPMS	•Nebulizer	As needed
Mercury Analyzer	•Calibrate and check sensitivity with previous data	Daily with use
Mercury Analyzer	•Response factor problems, check tubing for leaks, particularly in pump head, and check cell for fogging	As needed
Mercury Analyzer	•Replace desiccant in tube	With each use
Mercury Analyzer	•Check rotometer for airflow, if inadequate, replace flex tubing in pump lead	As needed
TCLP Apparatus (ZHE)	•Change O-rings	As needed
Thermometer	•All working thermometers are compared to a NIST thermometer.	Semi-annually
pH Meter	•Calibrated according to manufacturer's instructions. •The slope is documented and acceptable range 95-105%	Daily
Analytical Balance	•Analytical balances are checked and calibrated by a certified technician semi-annually. •Calibration is checked daily with class S weights. Must be within 0.1% S class weights calibrated annually	Semi-annually
		Daily
TCLP Tumblers	•Visually timed and confirmed to be 30±2 rpm.	Monthly
Microwaves	•Checked and calibrated by a certified technician	Semi-annually, calibrated weekly by staff
Microwaves	Check cap membranes for leaks	As needed

### 8.3 S TANDARDS AND REAGENTS

All reagents and standards must meet the requirements listed in the analytical methods.

**Table 8.3A: Stock Standard sources, receipt, and preparation information.**  
*(subject to revision as needed)*

<b>STOCK STANDARD SOURCES</b>					
*ICP metals used – Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, Zn, S					
*ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn, B, U, Th, Na, Ca, Mg, K, Al, Ti, Sr					
<i>Instrument Group/Standard</i>	<i>Standard Source*</i>	<i>How Received*</i>	<i>Source/Storage</i>	<i>Lab Stock Storage</i>	<i>Receipt Frequency</i>
ICP/CCVLL	Env. Express	2ppm-Al, Fe 20ppm-Ca, K, Na, Mg, S 1ppm- B, Si, Zn, Sn, Ti 0.04ppm-Be 0.1ppm-Pb, Mo, Cd, Ba 0.2ppm-Cr, Co, Ag, Mn, Sr, 0.4ppm-Tl, Sb, Ni, Cu, As, Se, V 0.3ppm- Li	Room temp.	2% HNO3 w/ Tr HF	Annual/Expiration Date
ICP (single element standards)	Env. Express or High Purity	1000ppm	Room temp.		Annual/Expiration Date
ICP/ICV	High Purity	500ppm – Al, Ca, Fe, Mg, Na, K, S 50ppm – All others	Room temp.	5% HNO3 w/ Tr HF	As needed
ICP/Calibration Standard and CCV	Env. Express	500ppm- Ca, K, Na 100ppm- Fe, Mg, Al 50ppm- S 30ppm- Zn 20ppm- Sb, As, B, Cu, Ni, Se, Si, Tl, Sn 10ppm- Cr, Co, Mn, Ag, Sr, Ti, V 5ppm- Ba, Pb, Mo, Cd 2ppm Be	Room temp.	5% HNO3 w/ Tr HF	As needed
ICP/LCS water/soil	Ultra Scientific	1000ppm – Ca, Mg, K, Na 100ppm – all others except Li (spiked separately)	Room temp.	5% HNO3	As needed
ICP/LCS soil (only for IH)	ERA	Varies with Lot #	Room temp.	none	As needed
ICP/ICSA	Env. Express	5000ppm – Al, Ca, Mg, 2000ppm – Fe	Room temp.	10% HNO3	As needed
ICP/ICSB	Env. Express	100ppm – B, Cd, Pb, Ag, Ni, Si, Zn, 50ppm – all others except Sr, Li	Room temp.	4% HNO3 w/ Tr HF	As needed
ICP/Yttrium	Env. Express	10,000 ppm	Room temp.	4% HNO3	As needed
ICP/Indium	Env. Express	1,000ppm	Room temp.	2%HNO3	As needed
ICPMS/ICV	Inorganic Ventures	1000ppm-Ca, Mg, K, Na, Al, Fe 10ppm- all others	Room temp.	5% HNO3 w/ Tr HF	As needed

**Table 8.3A: Stock Standard sources, receipt, and preparation information.**

*(subject to revision as needed)*

**STOCK STANDARD SOURCES**

\*ICP metals used – Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, Zn, S

\*ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn, B, U, Th, Na, Ca, Mg, K, Al, Ti, Sr

<i>Instrument Group/Standard</i>	<i>Standard Source*</i>	<i>How Received*</i>	<i>Source/ Storage</i>	<i>Lab Stock Storage</i>	<i>Receipt Frequency</i>
ICPMS/ Calibration Standard and CCV	Env. Express	100 ppm- Al, Fe 1000ppm-Mg, K, Ca, , Na 10ppm- All others	Room temp.	2% HNO3 w/ Tr HF	As needed
ICPMS/LCS water/soil	Inorganic Ventures	1000ppm – Ca, Mg, K, Na, Al, Fe 10ppm – all others	Room temp.	5% HNO3	As needed
ICPMS/LCS soil (for IH only)	ERA	Varies with Lot #	Room temp.	none	As needed
ICPMS/ICSA	Inorganic Ventures	10000ppm – Cl 2000ppm – C 1000ppm – Al, Ca, Fe, Mg, P, K, Na, S 20ppm – Mo, Ti	Room temp.	1.4% HNO3	As needed
ICPMS/ICSB	Inorganic Ventures	2ppm – Sb, As, Be, Cd, Cr, Co, Cu, Pb, Ni, Se, Ag, Tl, Sn, Zn, B, Ba, Cr, Mn, Sr, Th, V, U	Room temp.	5%HNO3 w/ Tr HF	As needed
Hg/ICV and LCS	Inorganic Ventures	1000ppm – Hg	Room temp.	2% HNO3	As needed
Hg/Calibration Standard and CCV	Env. Express	1000ppm – Hg	Room temp.	2% HNO3	As needed

\*Equivalent Providers may be utilized.

**Table 8.3B: Working standard concentration, storage and preparation information.**

*(subject to revision as needed)*

**WORKING STANDARD PREPARATION**

\*ICP metals used – Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, Zn, S

\*ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn, B, U, Th, Na, Ca, Mg, K, Al, Ti, Sr

<i>Instrument Group/Standard</i>	<i>How Prepared</i>	<i>Final Concentration</i>	<i>Source/ Storage</i>	<i>Expiration</i>
ICP/ICV	2mL Custom Stock ICV A and B, adjusted to 100mL with 10% HNO3	10ppm – Al, Ca, Fe, K, Mg, Na 1ppm – All others	Room temp.	1 month
ICP/Calibration Standard	12.5mL Stock Cal. Std. 5mL Stock Cal. Std. 1mL Stock Cal. Std. 5mL Stock LL Std.All adjusted to 100 mL with 10%HNO3	Std 4 – 0.05/1.25/2.5/3.75/5/7.5/12.5/25/125ppm Std 3 0.2/.5/1/1.5/2/3/5/10/50ppm Std 2 – .04/.1/.2/.3/.4/.6/2/10ppm Std 1 – 0.002/.005/.01/.015/.02/.03/.01/.5/1ppm	Room temp.	1 month

**Table 8.3B: Working standard concentration, storage and preparation information.**  
 (subject to revision as needed)

<b>WORKING STANDARD PREPARATION</b>				
*ICP metals used – Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, Zn, S				
*ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn, B, U, Th, Na, Ca, Mg, K, Al, Ti, Sr				
<b>Instrument Group/Standard</b>	<b>How Prepared</b>	<b>Final Concentration</b>	<b>Source/Storage</b>	<b>Expiration</b>
ICP/CCV	50mL Custom Stock CCV adjusted to 1000mL with 10% HNO <sub>3</sub>	50ppm- Ca, K, Na 10ppm- Fe, Mg, Al 5ppm- S 3ppm- Zn 2ppm- Sb, As, B, Cu, Ni, Se, Si, Tl, Sn 1ppm- Cr, Co, Mn, Ag, Sr, Ti, V 0.5ppm- Ba, Pb, Mo, Cd 0.2ppm Be	Room temp.	1 month
ICP/ICSA	100mL Custom Stock ICSA adjusted to 1000mL with 10% HNO <sub>3</sub>	500ppm – Al, Ca, Mg, 200ppm – Fe	Room temp.	1 month
ICP/ICSAB	100mL Custom Stock ICSA, 10mL Stock ICSAB adjusted to 1000mL with 10% HNO <sub>3</sub>	500ppm – Al, Ca, Mg, 200ppm – Fe 1ppm – B, Cd, Pb, Ag, Ni, Si, Zn, 0.5ppm – all others except Sr, Li, S, K, Na	Room temp.	1 month
ICP/Yttrium	5mL Stock Yttrium adjusted to 10L with 10% HNO <sub>3</sub>	5 ppm	Room temp.	1 month
ICP/Indium	30mL stock Indium adjusted to 1L with 10% HNO <sub>3</sub>	30ppm	Room temp.	1 month
ICPMS/ICV	0.5mL Stock ICV A and B, adjusted to 50mL with 2% HNO <sub>3</sub> /0.5%HCl	10ppm Ca, Mg, K, Na, Fe, Al 0.1ppm for all other elements	Room temp.	1 month
ICPMS/ Calibration Standard	1mL Stock Cal Std adjusted to 50mL with 2%HNO <sub>3</sub> /0.5%HCl. Serial Dilutions are done each calibration from 0.2ppm Std.	Cal 6 – 20ppm, 2ppm, 0.2ppm Cal 5 – 10ppm, 1ppm, 0.1ppm Cal 4 – 5ppm, 0.5ppm, 0.05ppm Cal 3 – 1ppm, 0.1ppm, 0.01ppm Cal 2 – 0.2ppm, 0.02ppm, 0.002ppm Cal 1 – 0.1ppm, 0.01ppm, 0.001ppm	Room temp.	1 month
ICPMS/CCV	0.5mL Stock CCV adjusted to 50mL with 2% HNO <sub>3</sub> /0.5%HCl.	10ppm- Ca, Mg, Na, K 1ppm-Fe, Al 0.1ppm- all other elements	Room temp.	1 month
ICPMS/ICSA	5mL Stock ICSA adjusted to 50mL with 2% HNO <sub>3</sub> /0.5%HCl.	1000ppm – Cl 200ppm – C 100ppm – Al, Ca, Fe, Mg, P, K, Na, S 2ppm – Mo, Ti	Room temp.	1 month
ICPMS/ICSAB	5mL Stock ICSA, .5mL Stock A and B ICSAB adjusted to 50mL with 2% HNO <sub>3</sub> /0.5%HCl	1000ppm – Cl 200ppm – C 100ppm – Al, Ca, Fe, Mg, P, K, Na, S 2ppm – Mo, Ti 0.02ppm – all other elements	Room temp.	1 Month
Hg/ICV	25µL of 3ppm Intermediate	0.0025ppm – Hg	Room temp.	1 Month

**Table 8.3B: Working standard concentration, storage and preparation information.**  
*(subject to revision as needed)*

<b>WORKING STANDARD PREPARATION</b>				
*ICP metals used – Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, Zn, S				
*ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn, B, U, Th, Na, Ca, Mg, K, Al, Ti, Sr				
<b>Instrument Group/Standard</b>	<b>How Prepared</b>	<b>Final Concentration</b>	<b>Source/Storage</b>	<b>Expiration</b>
Hg/Calibration Standard	Soils and waters: Std 6 - 1000µL of 300ppb Intermediate Std 5 - 500µL of 300ppb Intermediate Std 4 - 200µL of 300ppb Intermediate Std 3 - 100µL of 300ppb Intermediate Std 2 - 40µL of 300ppb Intermediate Std 1 - 20µL of 300ppb Intermediate	Std 6 – 0.01ppm Std 5 – 0.005ppm Std 4 – 0.002ppm Std 3 – 0.001ppm Std 2 – 0.0004ppm Std 1 – 0.0002ppm	Room temp.	4 days
Hg/CCV	2.5ppb CCV - 25µL of 3ppm Intermediate	0.0025ppm	Room temp.	1 Month
Hg/LCS Waters	30µL of 3ppm Intermediate	0.003ppm – Hg	Room temp.	1 Month
Hg/LCS Soils	50uL of 3ppm Intermediate	0.005ppm- Hg	Room temp.	1 Month

## 8.4 I INSTRUMENT CALIBRATION

### Mercury Analyzer - SOP Numbers 340384A & 340384B

Calibration of the mercury analyzer is achieved using 5 standards. Acceptable calibration is achieved when the correlation coefficient  $\geq 0.995$ . All results are calculated using software based on the peak area of the sample. A second source ICV is analyzed initially and must recover within  $\pm 10\%$  for Methods 7470A/7471A/7471B and within  $\pm 5\%$  for method 245.1. A primary source CCV is analyzed after every tenth sample and at the conclusion of the analytical sequence. The CCV must recovery within  $\pm 10\%$  for all analyses. Spike analyses are performed on 5% of the samples analyzed using EPA Method 7470A/7471A/7471B and on 10% of the samples analyzed using EPA Method 245.1.

### Inductively Coupled Plasma (ICP and ICPMS) - SOP Numbers 340386 & 340390

The PE ICP Optima 5300DV, Thermo 7400 ICP and Agilent ICPMS 7700, 7900 and PE ELAN 9000 are calibrated using at least 3 standards. A new calibration curve is analyzed daily. All calculations are performed by software using computerized linear regression. The linear regression correlation coefficient for the each analyte in the calibration curve lines must be 0.995 or better for all methods, except for EPA 6010C and 6020A which must have a correlation coefficient of 0.998 or better, A second source ICV is run initially and a primary source CCV is run after every tenth sample. For method 200.7, the ICV must recover within 5% of the true value and for all other methods, the ICV must recover within 10%. The CCV for all methods must recover within 10% of the true value. Duplicate and spike analyses are performed on 5% of the samples for EPA Methods 6010B, 6010C, 6020, 6020A and on 10% of the samples analyzed using EPA Methods 200.7 & 200.8.



**TABLE 8.4: CALIBRATION STANDARD CONCENTRATIONS**  
*This table is subject to revision without notice*

Analyte	ICP (mg/L)	ICP/MS (mg/L)	CVAA (ug/L)
Aluminum	0.10 - 500	0.01 - 2.0	
Antimony	0.02 - 5.0	0.001 - 0.2	
Arsenic	0.01 - 5.0	0.001 - 0.2	
Barium	0.005 - 10	0.001 - 0.2	
Beryllium	0.002 - 2.0	0.001 - 0.2	
Boron	0.05 - 5.0	0.001 - 0.2	
Cadmium	0.002 - 2.0	0.001 - 0.2	
Calcium	0.5 - 500	1.0 - 20.0	
Chromium	0.01 - 2.5	0.001 - 0.2	
Cobalt	0.01 - 2.5	0.001 - 0.2	
Copper	0.01 - 5.0	0.001 - 0.2	
Iron	0.10- 200	0.01 - 2.0	
Lead	0.005 - 2.0	0.001 - 0.2	
Lithium	0.015 - 3.75	-----	
Magnesium	0.5 - 500	1.0 - 20.0	
Manganese	0.010 - 2.5	0.001 - 0.2	
Molybdenum	0.005 - 2.0	0.001 - 0.2	
Nickel	0.01 - 5.0	0.001 - 0.2	
Potassium	0.50 - 100	1.0 - 20.0	
Selenium	0.01 - 5.0	0.001 - 0.2	
Silicon	0.05 - 5.0	-----	
Silver	0.005 - 2.5	0.001 - 0.2	
Sodium	0.50 - 500	1.0 - 20.0	
Strontium	0.01 - 2.5	0.001 - 0.2	
Sulfur	0.5 - 100	-----	
Thallium	0.01 - 5.0	0.001 - 0.2	
Thorium	-----	0.001 - 0.2	
Tin	0.05 - 5.0	0.001 - 0.2	
Titanium	0.05 - 2.5	0.001 - 0.2	
Uranium	-----	0.001 - 0.2	
Vanadium	0.02 - 2.5	0.001 - 0.2	
Zinc	0.05 - 7.5	0.001 - 0.2	
Mercury			Blank, 0.2 - 0.010

### 8.5 A ACCEPTANCE/REJECTION OF CALIBRATION

The initial calibration curve is compared with previous curves for the same analyte. All new standard curves are immediately checked with a secondary source or laboratory control standard prepared from a separate source than those used for calibration. All curves are visually reviewed to ensure that acceptable correlation represents linearity. Calibration curves may be rejected for nonlinearity, abnormal sensitivity, or poor response of the laboratory control standard. Specific criteria for each instrument are outlined in Table 8.5.

Continuing calibration verification is performed after every tenth sample. If a check standard does not perform within established criteria then the instrument is evaluated to determine the problem. Once the problem is corrected, all samples between the last in control sample and the first out of control check are re-analyzed.

TABLE 8.5 INSTRUMENT CALIBRATION & QC				
Instrument (Analysis)	Calibration Type	Number of Standards	Acceptance/Rejection Criteria	Frequency
ICP & ICPMS	Linear/ Initial	3 - 5	6010B, 6020, 200.7 200.8: Must have a correlation coefficient of at least 0.995. 6010C, 6020A: Must have a correlation coefficient of at least 0.998	Daily
ICP & ICPMS	Initial	Secondary source (ICV)	6010B, 6010C, 6020, 6020A, 200.8: ICV must be within +/-10%; 200.7: ICV must be within +/-5%	After initial calibration
ICP & ICPMS	Initial	1 Initial Calibration Blank	< ½ RL, concentrations of common laboratory contaminants shall not exceed the RL	After initial calibration
ICP, ICPMS, Mercury	Continuing	1 mid-level ref. std. (CCV)	Must be within ±10%	Every 10 <sup>th</sup> sample
ICP & ICPMS, Mercury	Continuing	1 Continuing Calibration Blank	< RL, concentrations of common laboratory contaminants must not exceed the RL	Every 10 <sup>th</sup> sample
ICP & ICPMS	Continuing	1 ICSA 1 ICSAB	Must be within ±20% for ICP and ICPMS	After initial calibration, at end and every 8 hours of run time.
ICP, ICPMS, Mercury	Continuing	1 Method Blank	< RL (<1/2 RL for DOD).	1 per batch
ICP, ICPMS, Mercury	Continuing	1 Laboratory Control Standard	200.8, 200.7, 245.1: LCS must be within 15%. 6010B, 6010C, 6020, 6020A, 7470A, 7471A/B must be within 20%	1 per batch
ICP & ICPMS	Continuing	1 Matrix Spike (MS), 1 Matrix Spike Duplicate (MSD)	6010B, 6010C, 6020, 6020A: Spike must be within ±25%, 200.8, 200.7 must be within 30%. MS and MSD must have an RPD ≤20%	1 of each per batch

TABLE 8.5 INSTRUMENT CALIBRATION & QC				
Instrument (Analysis)	Calibration Type	Number of Standards	Acceptance/Rejection Criteria	Frequency
Mercury	Linear/ Initial	3 - 5	Must have a correlation coefficient of at least 0.995	Daily
Mercury	Initial	Secondary source (ICV)	7470A, 7471: ICV must be within $\pm 10\%$ 245.1: ICV must be within $\pm 5\%$	After initial calibration
Mercury	Continuing	1 Matrix Spike (MS), 1 Matrix Spike Duplicate (MSD)	7470A, 7471A/B: Spike must be within $\pm 25\%$ , 245.1 must be within 30%. MS and MSD must have an RPD $\leq 20\%$	1 of each per batch

## 9.0 LABORATORY PRACTICES

### 9.1 REAGENT GRADE WATER

Reagent grade water is obtained from an ELGA Purelab Ultra system.

### 9.2 GLASSWARE WASHING AND STERILIZATION PROCEDURES

Much of the glassware used in metals preparation is disposable; however non-disposable glassware involved in metals preparation is washed with soap and water, rinsed in 1+1 nitric acid, and rinsed in DI water. Through digestion blanks, it has been determined that chromic acid washing is unnecessary. Glassware with visible gummy deposits remaining after washing is disposed of properly. All metals glassware is given another DI water rinse immediately prior to use. Metals glassware is segregated from all other glassware.

## 10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOPs associated with the metals laboratory can be found in the following table.

**TABLE 10.1: METALS DEPARTMENT SOPS**

*This table is subject to revision without notice*

SOP #	Title
<b>TCLP SOPs</b>	
340358	TCLP
340704	SPLP
340363	EP TOX
340364	MEP
340705	California Waste Extraction Test
<b>Mercury SOPs</b>	
340384A	Mercury in Liquid Waste (Cold-Vapor Technique) 7470A/245.1
340384B	Mercury in Solid Waste (Cold-Vapor Technique) 7471A
<b>Metals Prep SOPs</b>	
340389	Acid Digestion of Aqueous Samples and Extracts Method 3005A/3010A/3015/3030C
340380	Digestion of Metals and Trace Elements in DW and Wastes Method 200.2
340388	Acid Digestion of Sediments, Sludge, Soils and Oils Method 3050B/3051

SOP #	Title
340701	Metals Digestion of personal cassettes Method 7300, 3051
340702	Metals Digestion for Sediments, Soils, and Sludge NIOSH 7300, Method 3051 for ELLAP Paint chips and ELLAP soils
340703	Metals Digestion of Hi-Vol filters and Environmental Lead Wipes 3050B and 3051
340391	Silver (Photographic Waste) Method 7760 and 272.1
340354A	Turbidity-Metals Drinking Water Screen Only (EPA Method 180.1)
340392	Sodium Absorption Ratio
340707	Fine, Coarse Soil Sieve Preparation for Lead Analysis for Michigan DEQ
<b>Metals Analysis SOPs</b>	
340386	Metals by ICP Method 6010, 200.7
340390	Metals by ICP-MS Method 6020, 200.8

## 11.0 QUALITY CONTROL CHECKS

**NOTE:** For specific guidance on each determinative method, including required quality control and specific state requirements/modifications, refer to the relevant laboratory standard operating procedure(s).

- 11.1 ESC participates in proficiency testing (PTs) in support of various laboratory accreditations/recognitions. Environmental samples are purchased from Phenova. The WS, WP and solid matrix studies are completed every 6 months. For industrial hygiene and environmental lead accreditation, PTs are administered by AIHA. IHPAT samples for metals analysis by NIOSH 7300/7301/7303, including lead in air, is completed every quarter. Soil, wipes and paint PTs are also completed quarterly in conjunction with the AIHA Environmental Lead Laboratory Accreditation Program (ELLAP). All proficiency testing samples are received and analyzed by method according to the vendor's instructions and according to the applicable analytical SOP.
- 11.2 Initial Demonstrations of Capability (IDOCs) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability (CDOCs) must be updated at least annually. The associated data is filed within the department and available for review.
- 11.3 Matrix Spike and Matrix Spike Duplicates are performed on 5–10% of samples analyzed depending on analytical method requested. For methods 6010, 6020, 7470A and 7471A duplicates, matrix spikes and matrix spike duplicates are performed on 5% of samples. For methods 200.7, 200.8 and 245.1, the same QC is performed on 10% of samples. The RPD must not exceed 20%.
- 11.4 A laboratory control sample (LCS) is analyzed one per batch of samples. The acceptance criteria for all water samples is  $\pm 15\%$  for 245.1, 200.7, and 200.8. All other methods have an acceptance criteria of  $\pm 20\%$ . For Industrial Hygiene samples, the LCS is analyzed in duplicate per batch and utilizes a blank matrix that is as similar to the field samples as possible.

11.5 A method preparation blank is performed per batch of samples processed. If the reporting limit [RL] is exceeded, the laboratory evaluates whether reprocessing of the samples is necessary, based on the following criteria:

- The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch or
- The blank contamination is greater than 1/10 of the specified regulatory limit.

The concentrations of common laboratory contaminants must not exceed the reporting limit. Any samples associated with a blank that fail these criteria are re-processed in a subsequent preparation batch, except when the sample analysis resulted in non-detected results for the failing analytes.

## 12.0 DATA REDUCTION, VALIDATION, AND REPORTING

### 12.1 DATA REDUCTION

The analyst performs the data calculation and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in ESC SOP #030201, *Data Handling and Reporting*. A secondary review of the data package is performed according to ESC SOP #030227, *Data Review*. The reviewer verifies that the analysis has been performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required flags on test reports, etc.)

### 12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.1 for current QC targets and controls and current reporting limits.

### 12.3 REPORTING

Reporting procedures are documented in SOP #030201, *Data Handling and Reporting*.

Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs (subject to revision without notice)							
Class An	alyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppm)
(ICP-AES)	Aluminum	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.10
(ICP-AES)	Aluminum	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.10
(ICP-AES)	Aluminum	3050B/3051A	6010B/C	Solid	80 - 120	<20	10
(ICP-MS)	Aluminum	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.10
(ICP-MS)	Aluminum	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.1

<b>Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs</b>							
<i>(subject to revision without notice)</i>							
<b>Class An</b>	<b>alyte</b>	<b>Prep Method</b>	<b>Analysis Method</b>	<b>Matrix</b>	<b>Accuracy Range (%)</b>	<b>Precision (RPD)</b>	<b>RL (ppm)</b>
(ICP-MS)	Aluminum	3050B/3051A	6020/A	Solid	80 - 120	<20	10
(ICP-AES)	Antimony	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.02
(ICP-AES)	Antimony	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.02
(ICP-AES)	Antimony	3050B/3051A	6010B/C	Solid	80 - 120	<20	2.0
(ICP-MS)	Antimony	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Antimony	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-MS)	Antimony	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Arsenic	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-AES)	Arsenic	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-AES)	Arsenic	3050B/3051A	6010B/C	Solid	80 - 120	<20	2.0
(ICP-MS)	Arsenic	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Arsenic	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-MS)	Arsenic	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Barium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.005
(ICP-AES)	Barium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.005
(ICP-AES)	Barium	3050B/3051A	6010B/C	Solid	80 - 120	<20	0.50
(ICP-MS)	Barium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.002
(ICP-MS)	Barium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.005
(ICP-MS)	Barium	3050B/3051A	6020/A	Solid	80 - 120	<20	0.20
(ICP-AES)	Beryllium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.002
(ICP-AES)	Beryllium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-AES)	Beryllium	3050B/3051A	6010B/C	Solid	80 - 120	<20	0.20
(ICP-MS)	Beryllium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Beryllium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-MS)	Beryllium	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Boron	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.20
(ICP-AES)	Boron	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.20
(ICP-AES)	Boron	3050B/3051A	6010B/C	Solid	80 - 120	<20	10
(ICP-MS)	Boron	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.02
(ICP-MS)	Boron	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.02
(ICP-MS)	Boron	3050B/3051A	6020/A	Solid	80 - 120	<20	1.0
(ICP-AES)	Cadmium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.002
(ICP-AES)	Cadmium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-AES)	Cadmium	3050B/3051A	6010B/C	Solid	80 - 120	<20	0.50
(ICP-MS)	Cadmium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Cadmium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.001
(ICP-MS)	Cadmium	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Calcium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	1.0

<b>Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs</b>							
<i>(subject to revision without notice)</i>							
<b>Class An</b>	<b>alyte</b>	<b>Prep Method</b>	<b>Analysis Method</b>	<b>Matrix</b>	<b>Accuracy Range (%)</b>	<b>Precision (RPD)</b>	<b>RL (ppm)</b>
(ICP-AES)	Calcium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	1.0
(ICP-AES)	Calcium	3050B/3051A	6010B/C	Solid	80 - 120	<20	100
(ICP-MS)	Calcium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1.0
(ICP-MS)	Calcium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	1.0
(ICP-MS)	Calcium	3050B/3051A	6020/A	Solid	80 - 120	<20	50
(ICP-AES)	Chromium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-AES)	Chromium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-AES)	Chromium	3050B/3051A	6010B/C	Solid	80 - 120	<20	1.0
(ICP-MS)	Chromium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Chromium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-MS)	Chromium	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Cobalt	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-AES)	Cobalt	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-AES)	Cobalt	3050B/3051A	6010B/C	Solid	80 - 120	<20	1.0
(ICP-MS)	Cobalt	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Cobalt	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-MS)	Cobalt	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Copper	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-AES)	Copper	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-AES)	Copper	3050B/3051A	6010B/C	Solid	80 - 120	<20	2.0
(ICP-MS)	Copper	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.002
(ICP-MS)	Copper	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.005
(ICP-MS)	Copper	3050B/3051A	6020/A	Solid	80 - 120	<20	0.20
(ICP-AES)	Iron	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.10
(ICP-AES)	Iron	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.10
(ICP-AES)	Iron	3050B/3051A	6010B/C	Solid	80 - 120	<20	10
(ICP-MS)	Iron	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.10
(ICP-MS)	Iron	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.10
(ICP-MS)	Iron	3050B/3051A	6020/A	Solid	80 - 120	<20	250
(ICP-AES)	Lead	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.005
(ICP-AES)	Lead	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.005
(ICP-AES)	Lead	3050B/3051A	6010B/C	Solid	80 - 120	<20	0.50
(ICP-MS)	Lead	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Lead	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-MS)	Lead	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Lithium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.015
(ICP-AES)	Lithium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.015
(ICP-AES)	Lithium	3050B/3051A	6010B/C	Solid	80 - 120	<20	5.0

<b>Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs</b>							
<i>(subject to revision without notice)</i>							
<b>Class An</b>	<b>alyte</b>	<b>Prep Method</b>	<b>Analysis Method</b>	<b>Matrix</b>	<b>Accuracy Range (%)</b>	<b>Precision (RPD)</b>	<b>RL (ppm)</b>
(ICP-AES)	Magnesium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	1.0
(ICP-AES)	Magnesium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	1.0
(ICP-AES)	Magnesium	3050B/3051A	6010B/C	Solid	80 - 120	<20	100
(ICP-MS)	Magnesium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1.0
(ICP-MS)	Magnesium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	1.0
(ICP-MS)	Magnesium	3050B/3051A	6020/A	Solid	80 - 120	<20	50
(ICP-AES)	Manganese	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-AES)	Manganese	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-AES)	Manganese	3050B/3051A	6010B/C	Solid	80 - 120	<20	1.0
(ICP-MS)	Manganese	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.002
(ICP-MS)	Manganese	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.005
(ICP-MS)	Manganese	3050B/3051A	6020/A	Solid	80 - 120	<20	0.20
(CVAA)	Mercury	7471A/B	7471A/B	Solid	80 - 120	<20	0.02
(CVAA)	Mercury	7470A	7470A	Liquid/Aqueous	80 - 120	<20	0.0002
(CVAA)	Mercury	245.1 /7470A	245.1	Liquid/Aqueous	85 - 115	<20	0.0002
(ICP-AES)	Molybdenum	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.005
(ICP-AES)	Molybdenum	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.005
(ICP-AES)	Molybdenum	3050B/3051A	6010B/C	Solid	80 - 120	<20	0.50
(ICP-MS)	Molybdenum	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.002
(ICP-MS)	Molybdenum	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.005
(ICP-MS)	Molybdenum	3050B/3051A	6020/A	Solid	80 - 120	<20	0.20
(ICP-AES)	Nickel	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-AES)	Nickel	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-AES)	Nickel	3050B/3051A	6010B/C	Solid	80 - 120	<20	2.0
(ICP-MS)	Nickel	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Nickel	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-MS)	Nickel	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Potassium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	1.0
(ICP-AES)	Potassium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	1.0
(ICP-AES)	Potassium	3050B/3051A	6010B/C	Solid	80 - 120	<20	100
(ICP-MS)	Potassium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1.0
(ICP-MS)	Potassium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	1.0
(ICP-MS)	Potassium	3050B/3051A	6020/A	Solid	80 - 120	<20	50
(ICP-AES)	Selenium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-AES)	Selenium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-AES)	Selenium	3050B/3051A	6010B/C	Solid	80 - 120	<20	2.0
(ICP-MS)	Selenium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Selenium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002



<b>Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs</b>							
<i>(subject to revision without notice)</i>							
<b>Class An</b>	<b>alyte</b>	<b>Prep Method</b>	<b>Analysis Method</b>	<b>Matrix</b>	<b>Accuracy Range (%)</b>	<b>Precision (RPD)</b>	<b>RL (ppm)</b>
(ICP-MS)	Selenium	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Silicon	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.20
(ICP-AES)	Silicon	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.20
(ICP-AES)	Silicon	3050B/3051A	6010B/C	Solid	80 - 120	<20	20
(ICP-AES)	Silver	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.005
(ICP-AES)	Silver	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.005
(ICP-AES)	Silver	3050B/3051A	6010B/C	Solid	80 - 120	<20	1.0
(ICP-MS)	Silver	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Silver	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-MS)	Silver	3050B/3051A	6020/A	Solid	80 - 120	<20	0.20
(ICP-AES)	Sodium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	1.0
(ICP-AES)	Sodium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	1.0
(ICP-AES)	Sodium	3050B/3051A	6010B/C	Solid	80 - 120	<20	100
(ICP-MS)	Sodium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1.0
(ICP-MS)	Sodium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	1.0
(ICP-MS)	Sodium	3050B/3051A	6020/A	Solid	80 - 120	<20	50
(ICP-AES)	Strontium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-AES)	Strontium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-AES)	Strontium	3050B/3051A	6010B/C	Solid	80 - 120	<20	1.0
(ICP-MS)	Strontium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-MS)	Strontium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-MS)	Strontium	3050B/3051A	6020/A	Solid	80 - 120	<20	0.50
(ICP-AES)	Sulfur	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	1.0
(ICP-AES)	Sulfur	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	1.0
(ICP-AES)	Sulfur	3050B/3051A	6010B/C	Solid	80 - 120	<20	100
(ICP-AES)	Thallium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-AES)	Thallium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-AES)	Thallium	3050B/3051A	6010B/C	Solid	80 - 120	<20	2.0
(ICP-MS)	Thallium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Thallium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-MS)	Thallium	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Tin	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.05
(ICP-AES)	Tin	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.05
(ICP-AES)	Tin	3050B/3051A	6010B/C	Solid	80 - 120	<20	5.0
(ICP-MS)	Tin	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.001
(ICP-MS)	Tin	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.002
(ICP-MS)	Tin	3050B/3051A	6020/A	Solid	80 - 120	<20	0.10
(ICP-AES)	Titanium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.05

<b>Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs</b> (subject to revision without notice)							
Class An	alyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppm)
(ICP-AES)	Titanium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.05
(ICP-AES)	Titanium	3050B/3051A	6010B/C	Solid	80 - 120	<20	5.0
(ICP-MS)	Titanium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-MS)	Titanium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-MS)	Titanium	3050B/3051A	6020/A	Solid	80 - 120	<20	0.50
(ICP-AES)	Vanadium	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.02
(ICP-AES)	Vanadium	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.02
(ICP-AES)	Vanadium	3050B/3051A	6010B/C	Solid	80 - 120	<20	2.0
(ICP-MS)	Vanadium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.002
(ICP-MS)	Vanadium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.005
(ICP-MS)	Vanadium	3050B/3051A	6020/A	Solid	80 - 120	<20	0.20
(ICP-MS)	Uranium	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-MS)	Uranium	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.01
(ICP-MS)	Uranium	3050B/3051A	6020/A	Solid	80 - 120	<20	0.05
(ICP-AES)	Zinc	200.2 NPDES	200.7	Liquid/Aqueous	85 - 115	<20	0.05
(ICP-AES)	Zinc	3015/3010	6010B/C	Liquid/Aqueous	80 - 120	<20	0.05
(ICP-AES)	Zinc	3050B/3051A	6010B/C	Solid	80 - 120	<20	5.0
(ICP-MS)	Zinc	200.2 NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.01
(ICP-MS)	Zinc	3015/3010	6020/A	Liquid/Aqueous	80 - 120	<20	0.025
(ICP-MS)	Zinc	3050B/3051A	6020/A	Solid	80 - 120	<20	1.0

<b>Table 12.3B: QC Targets for IH Metals Accuracy (LCS), Precision and RLs</b> (subject to revision without notice)							
Class An	alyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (% RPD)	RL
(ICP-AES)	Lead	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Lead	3050B (mod.)	6010B/C	Paint Chips	80-120	<20	50. mg/kg
(ICP-AES)	Lead	3050B (mod.)	6010B/C	Wipes	80-120	<20	2.0 ug/sample
(ICP-AES)	Arsenic	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Barium	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Beryllium	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Cadmium	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Chromium	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Cobalt	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Copper	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample

<b>Table 12.3B: QC Targets for IH Metals Accuracy (LCS), Precision and RLs</b>							
<i>(subject to revision without notice)</i>							
<b>Class An</b>	<b>alyte</b>	<b>Prep Method</b>	<b>Analysis Method</b>	<b>Matrix</b>	<b>Accuracy Range (%)</b>	<b>Precision (% RPD)</b>	<b>RL</b>
(ICP-AES)	Iron	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	5.0 ug/sample
(ICP-AES)	Manganese	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Molybdenum	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Nickel	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Selenium	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Silver	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Thallium	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Tin	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Titanium	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Vanadium	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Zinc	3050B (mod.)	NIOSH 7300/7301/7303	Filters	85-115	<20	2.5 ug/sample

### 13.0 CORRECTIVE ACTIONS

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CARs are kept on file by the Regulatory Affairs Department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

#### 13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these control limits are exceeded, corrective action must be taken. Calculated sample spike control limits are also used.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP

##### 13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory, the method criteria takes precedence.

### 13.2.2 Calibration Verification Criteria Are Not Met: Inorganic Analysis

Rejection Criteria - See Table 8.5.

Corrective Action - If a standard curve linearity is not acceptable and/or the absorbance for specific standard(s) is not analogous to historic data, the instrument settings, nebulizer, etc. are examined to ensure that nothing has been altered, clogged, etc. The working standards are made fresh, intermediate dilutions are re-checked and the instrument is re-calibrated. If a problem persists, the Department Supervisor is notified for further action.

If the initial reference check sample is out of control, the instrument is re-calibrated and the check sample is rerun. If the problem continues the check sample is re-prepared. If the problem still exists then the standards and reagent blank are re-prepared. If the problem persists, the Department Supervisor is notified for further action.

### 13.2.3 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

Rejection Criteria - Blank reading is more than the RL for Method Blanks and/or Instrument Blanks. ( $\frac{1}{2}$  the RL for Method Blanks and/or instrument blanks for DoD work and also may be required for some customers and programs.)

Corrective Action - Standard curves and samples are evaluated for any obvious contamination that may be isolated or uniform throughout the sequence. If necessary, reagents, QC samples and field samples are re-prepared and re-analyzed. Re-analyses are not initiated until the cause of the contamination is identified and resolved. If samples have already been partially prepared or analyzed, the Department Supervisor is consulted to determine if data needs to be rejected or if samples need to be re-prepped.

### 13.2.4 Out Of Control Laboratory Control Standards (LCS)

Rejection Criteria - If the performance is outside of lab-generated control (Listed in Table 12.3).

Corrective Action - Instrument settings are checked. The LCS standard is re-analyzed. If the LCS is still out of control, re-calibration is performed, and samples affected since the last in control reference standard are re-analyzed. If the LCS fails again after re-calibration, the entire workgroup must be re-prepped. The Department Supervisor is consulted for further action.

### 13.2.5 Out Of Control Matrix Spike Samples

Rejection Criteria - If either the MS or MSD sample is outside the established control limits.

Corrective Action - Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance, however, is based on method blank and LCS performance, not on MS/MSD recoveries. Specific methods, customers, and programs may require further corrective action in some cases.

### 13.2.8 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

Corrective Action - Instrument settings are checked, calibration verification standard is rerun. If the standard is still out of control, re-calibration is performed, and samples affected since the last in control reference standard are rerun. The Department Supervisor is consulted for further action.

13.3 Responsibility - It is the Department Supervisor's responsibility to evaluate the validity of the corrective action response and submit it to the QA department for processing. In addition, the Supervisor is responsible for appointing the appropriate person within the department to be responsible for correcting the nonconformance. When a corrective action warrants a cessation of analysis, the following personnel are responsible for executing the "stop work" order:

- Laboratory Manager
- QA Department
- Department Supervisor
- Technical Service Representative

## 14.0 RECORD KEEPING

Record keeping is outlined in SOP #030230, *Standards Logger*, SOP #030227, *Data Review* and SOP #030201, *Data Handling and Reporting*

## 15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 12.0 and SOP #010104, *Internal Audits*.

## 16.0 REVISIONS

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of this appendix are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0 (Appendix V)	Section 5.1 – Removed language about supervisor and backup reviewing and approving all data for metals Section 7.1 – Clarified the use of plastic containers and the storage of soil samples for Hg Section 8.0 – Updated instrument software Section 8.1 – Updated equipment list Section 8.4 – Revised Hg ICAL criteria to $r \geq 0.995$ Table 8.4 – Updated calibration range of some elements Section 8.5 – Clarified CCV frequency in 2 <sup>nd</sup> paragraph Section 9.1 – Clarified what system is used to generate the reagent water Section 11.4 – Clarified that $\pm 15\%$ LCS criteria is for 245.1, 200.7, and 200.8. All other methods

Document	Revision
	are $\pm 20\%$ . Also removed language about seeing CoA for soil true values which is no longer applicable. Table 12.3A – Updated RLs Section 13.2.3 – Revised blank criteria to at the RL and clarified $\frac{1}{2}$ RL is needed for DoD and other customers/programs Section 13.2.5 – Reworded MS/MSD criteria and revised corrective action to just qualify unless method, customer, or program states to do something else. Section 16 – New section for summary of revisions to previous version

1.0 SIGNATORY APPROVALS

# VOLATILES QUALITY ASSURANCE MANUAL

## APPENDIX VI TO THE ESC QUALITY ASSURANCE MANUAL


for

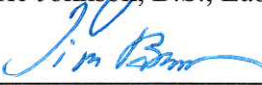
ESC LAB SCIENCES  
12065 LEBANON ROAD  
MT. JULIET, TENNESSEE 37122  
(615) 758-5858


Prepared by

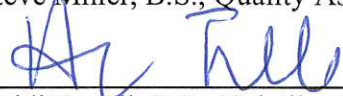
ESC LAB SCIENCES  
12065 LEBANON ROAD  
MT. JULIET, TENNESSEE 37122  
(615) 758-5858

**NOTE: The QAM has been approved by the following people.**

  
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Jim Brownfield, B.S., Compliance Director 615-773-9681

  
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### **3.0 SCOPE AND APPLICATION**

This appendix discusses specific QA requirements for general analytical protocols to ensure analytical data generated from the Volatiles (VOC) laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

### **4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES**

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling guidance and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual*.

### **5.0 PERSONNEL AND TRAINING**

#### **5.1 PERSONNEL**

Heidi Ferrell, with a B.S. degree in Chemistry, is the Department Supervisor and is responsible for the overall production of the department; including the management of the staff and scheduling. Ms. Ferrell has 9 years of environmental laboratory experience. In her absence, Brett Andersen assumes responsibility for departmental decisions in the Volatiles Lab.

Brett Andersen, with a B.S in Microbiology and M.S. in Plant Microbiology and Pathology, is the Primary Analyst for the Volatiles Lab. He is proficient in volatile organic analytical methods and has 9 years of environmental laboratory experience.

#### **5.2 TRAINING**

- 5.2.1 All new analysts to the laboratory are trained by a Primary Analyst or Supervisor according to ESC protocol. ESC's training program is outlined in *SOP 030205 Technical Training and Personnel Qualifications*. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in VOC Laboratory is also demonstrated by acceptable participation in the Phenova proficiency testing program (PTs) and using daily Quality Control sample analyses. Documentation of analyst training is maintained on file within the department.

## **6.0 FACILITIES AND LABORATORY SAFETY**

### **6.1 FACILITIES**

The main area of the instrumentation laboratory in Building #2 has approximately 7000 square feet with 700 square feet of bench area and 300 square feet of preparatory area. The lighting standard is fluorescence. The air handling systems are (1) 60-ton units with gas heating and (1) 25-ton unit. The physical and air-handling separations, between this laboratory and other ESC sections, prevent potential cross-contamination between solvent vapor generation and incompatible analytical processes. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's waste disposal carrier. Waste handling is discussed in detail in Section 6.0 of the ESC Quality Assurance Manual. ESC's building information guides and site plan are shown in Appendix I.

### **6.2 LABORATORY SAFETY**

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods.

ESC's laboratory safety guidelines are detailed in the *ESC Chemical Hygiene Plan*.

## **7.0 SAMPLING PROCEDURES**

### **7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING**

- Field Sampling procedures are described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Matrices for VOC environmental analyses include groundwater, wastewater, drinking water, soil, and sludge.
- Sample containers, preservation methods and holding times vary depending on analyses requested. Please see determinative procedures for specific directions.
- Plastic containers or lids may NOT be used for the storage of samples due to sample contamination from the phthalate esters and other hydrocarbons in the plastic.
- Environmental sample containers should be filled carefully to prevent any portion of the sample from coming into contact with the sampler's gloves causing possible contamination.
- Containers for VOC samples should be selected carefully to minimize headspace that could lead to a low bias in the analytical results. Headspace is monitored during sample login and is documented on the Sample Receipt Corrective Action form when observed.

## 8.0 EQUIPMENT

### 8.1 EQUIPMENT LIST

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Volatiles Analysis</b>						
<i>This table is subject to revision without notice</i>						
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Instrument Name</i>	<i>#</i>	<i>Serial #</i>	<i>Location</i>
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	1	3333A31215	Volatiles
Gas Chromatograph	Agilent	6890	VOCGC	2	CN10609095	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	3	2950A26786	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	4	3336A50614	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	5	3027A29678	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	6	2950A27895	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	7	3313A37610	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	13	2921A23548	Volatiles
Gas Chromatograph	Agilent	6890	VOCGC	10	US00022519	Volatiles
Gas Chromatograph	Agilent	6890	VOCGC	12	US00000410	Volatiles
Gas Chromatograph	Agilent	6890	VOCGC	14	CN10408054	Volatiles
Gas Chromatograph	Agilent	6890	VOCGC	15	US10232130	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5975 MSD	VOCMS	2	GCCN10641044 MSUS63234371	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973 MSD	VOCMS	6	GCCN10343037 MSUS44647141	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	4	GCUS00003465 MSUS82311257	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	7	GCUS00040221 MS05040022	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	8	GCUS00040221 MS03940725	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	13	GCCN103390006 MSUS91911078	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	14	GCUS00009794 MSUS63810153	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	16	GCUS00006479 MSUS82321899	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	18	GC CN10517046 MSUS03340424	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	19	GCCN10611062 MSUS60542638	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5975MSD	VOCMS	20	GCCN621S4367 MSUS469A4832	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5975MSD	VOCMS	21	GCCN621S4368 MSUS469A4833	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	22	GCCN10728074 MSUS71236615	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5975MSD	VOCMS	23	GCCN10728068 MSUS71236616	Volatiles

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Volatiles Analysis</b>						
<i>This table is subject to revision without notice</i>						
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Instrument Name</i>	<i>#</i>	<i>Serial #</i>	<i>Location</i>
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	24	GCCN10151020 MSUS10223406	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	25	GCCN99205324 MSUS98003634	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	26	GCCN10301152 MSUS10313616	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	27	GCCN10301155 MSUS10313619	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	28	GCUS000034135 MSUS94240103	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	29	GCUS00033898 MSUS94240096	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	30	GCUS10208101 MSUS10442360	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	31	GCUS14453011 MSUS54441572	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	32	GCCN13113015 MSUS92013978	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	33	GCCN11351165 MSUS52440724	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	34	GCCN13231014 MSUS50680012	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	35	GCCN108490077 MSUS83131017	Volatiles
Centurion Autosampler	(14) PTS/EST	Centurion				Volatiles
Autosampler	(24) Varian	Archon				Volatiles
Autosampler	(2) CDS	7400				Volatiles
Purge and Trap	(17) OI Analytical	Eclipse				Volatiles
Purge and Trap	(15) PTS/EST	Encon				Volatiles
Purge and Trap	(7)PTS/EST	Evolution				Volatiles

<b>8.2 E EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION</b>		
<b>INSTRUMENT</b>	<b>P. M. DESCRIPTION</b>	<b>FREQUENCY</b>
Analytical Balances	•Check with Class "I" weights	Daily; tolerance $\pm 0.1\%$
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semiannually
Refrigerators & Incubators	•Maintenance service	As needed - determined by daily temperature performance checks
Gas Chromatograph Detectors: FID	Change Quartz jet; clean; replace flame tip	As needed - when deterioration is noticeable
Gas Chromatograph Detectors: PID	Change or clean lamp	As needed - when deterioration is noticeable
Gas Chromatograph/Mass Spectrometer	•Autotune Report	Inspected daily
Gas Chromatograph/Mass Spectrometer	•Clean ion source	As needed to maintain high mass resolution

<b>8.2 E EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION</b>		
INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
Gas Chromatograph/Mass Spectrometer & Gas Chromatographs	•Replace septum and liner	As needed to maintain injection port inert
Gas Chromatograph/Mass Spectrometer	•Replace vacuum pump oil	Every 6 months
Gas Chromatograph/Mass Spectrometer & Gas Chromatographs	•Replace column	When separation begins to degrade
Archon/ Centurion Autosampler	•Monitor the Daily QC, including internal standards for changes or failure.	Daily with use

### 8.3 S STANDARDS AND REAGENTS

<b>Table 8.3A: Standard stock sources, description and calibration information.</b>					
<i>This table is subject to revision without notice</i>					
Method V	Vendor*	Description	Calibration	Storage Req.	Expiration
8260	Ultra	Gases Mix	Primary	-30°C to 4°C	1 week
	NSI	Custom VOC Mix 1	Primary	< -10°C	6 months
	NSI	Mix 2	Primary	2°C to 8°C	6 months
	Absolute Stds	n-Hexane/n-Heptane	Primary		6 months
	NSI	iso-octane	Primary	2°C to 8°C	6 months
	Ultra	Custom Std (1,2-MNP)	Primary	15°C to 30°C	6 months
	Restek	Acrolein	Primary	<0°C	3 months
	SPEX	Custom (AZ analytes)	Primary	<0°C	6 months
	Restek	TX TPH Mix (GRO)	Primary	<10°C	6 months
	SPEX	Custom (AZ analytes)	Secondary	<0°C	6 months
	NSI	Custom VOC Mix 2	Secondary	2°C to 8°C	6 months
	Ultra	Custom Std (1,2-MNP)	Secondary	15°C to 30°C	6 months
	Restek	Custom VOA LCS Mix 1	Secondary	<0°C	6 months
	Absolute Stds	n-Hexane/n-Heptane	Secondary	4°C	6 months
	Restek	Acrolein	Secondary	<0°C	3 months
Ultra	Petroleum Products Solution (GRO)	Secondary	15°C to 30°C	6 months	
8015 (GRO)	Restek	Certified BTEX in Unleaded Gas Composite Standard	Secondary	<0°C	6 months
	NSI	Gas Composite	Primary	2°C to 8°C	6 months
8021	Restek	WISC PVOC/GRO Mix	Secondary	<0°C	6 months
	NSI	PVOC/GRO Mix WI	Primary	4° ± 2°C	6 months
VPH	NSI	Primary VPH Dilution Std	Primary	15°C to 30°C	6 months
	NSI	Custom VPH LCS MIX	Secondary	4° ± 2°C	6 months

\*Equivalent Providers may be utilized.

<b>TABLE 8.3B: Working Standard Concentrations</b>			
<i>This table is subject to revision without notice</i>			
<b>ORGANIC COMPOUNDS</b>	<b>Method #</b>	<b>GC/MS</b>	<b>GC</b>
VOCs by GC/MS	524.2, 624, SM6200B 20 <sup>th</sup> , 8260B	GW/WW , 0.5, 1, 2, 5, 10, 25, 40, 75, 100, 200µg/L DW 0.25, 0.5, 1, 2, 5, 10, 25, 50, 100, 150µg/L GRO 0.4, 1, 2, 4, 5, 7, 10, 20ug/mL	
BTEX/GRO, 8015MOD, WI GRO, LA TPH G, OHIO GRO, WI PVOC, BTEX/OA1	BTEX 8021 GRO 8015, BTEX OA1 or state specific GRO		BTEX 0.5, 1, 5,10, 25,50,100,150,200, 250ug/L (m,p-Xylene is doubled) GRO 0.055, 0.11, 0.55, 1.1. 2.75, 5.5, 11mg/L
MADEP VPH	MADEP VPH		Aromatic C9-C10: 0.001, 0.002, 0.01, 0.02, 0.05, 0.1, 0.2, 0.4, 1.0, 2.0mg/L Aliphatic C5-C8: 0.006, 0.012, 0.06, 0.12, 0.3, 0.6, 1.2, 2.4, 6.0, 12.0mg/L Aliphatic C9-C12: 0.007, 0.014, 0.07, 0.14, 0.36, 0.7, 1.4, 2.8, 7.0, 14.0mg/L

## 8.4 I INSTRUMENT CALIBRATION

### 602 - BTEX - SOP Number 330351

The gas chromatograph is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of three concentration levels for each compound of interest. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors are <10 % RSD over the working range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990 (0.995 for USACE Projects). An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should recover within ±20% of the expected concentration for each analyte.

During the analytical sequence, the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recovery within 15% of the expected concentration for each analyte.

At daily instrument startup and in lieu of performing an entire initial calibration, the working calibration curve or response factors are verified on each working day by the analysis of a Quality Control Check Standard. The responses must meet the criteria found

in Table 2 of the 602 Method. If the responses do not meet these criteria, the analysis must be repeated. If the standard still does not meet the criteria, a new calibration curve is prepared.

**8021B - BTEX - SOP Number 330351**

The gas chromatograph is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of five concentration levels for each compound of interest.

The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors are <20 % RSD over the working range, the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios (Area/Ref. Area) vs (Amt./Ref Amt). If the response factors of the initial calibration are <20 % RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990 (0.995 for USACE Projects). An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should recover within  $\pm 20\%$  of the expected concentration for each analyte.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies from the predicted response by more than  $\pm 20\%$ , the analysis must be repeated using fresh standard. If the standard still does not meet the acceptance criteria, a new initial calibration curve must be generated.

**8015B/C/D & State Methods - Gasoline Range Organics - SOP Number 330351**

Certain state accreditation/registration programs may have specific requirements for calibration and analysis that must be met. Those requirements supersede the general guidance provided in this section and are addressed in the relevant determinative SOP. For EPA Method 8015 for routine GRO analyses, the gas chromatograph is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of five concentration levels for each analyte of interest. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are <20 % RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.)

for quantitation providing that the correlation coefficient is at least 0.990 (0.995 for USACE DOD Projects). An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should meet criteria of  $\pm 20\%$  of the expected concentration for each analyte.

The working calibration curve or response factors are verified on each working day by the analysis of one or more calibration standards. If the response of any analyte varies from the predicted response by more than 20% RSD, the analysis must be repeated using a new calibration standard. If the standard still does not meet the criteria, a new calibration curve is prepared.

**8260B/C, 624, SM6200B, 524.2 - Gas Chromatography/Mass Spectrometry (GC/MS):  
Volatile Organics - SOP Numbers 330363 & 330364**

Detector mass calibration is performed daily using the autotune function of the GC/MS analytical system and BFB (Bromofluorobenzene). Following verification of the appropriate masses, the instrument sensitivity is verified by injecting a tuning solution containing bromofluorobenzene (BFB). The BFB spectra must meet the following ion abundance criteria:

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 to 9% of mass 95
173	Less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

Successful tuning must occur every 12 hours for method 524.2, 8260B/C & SM6200B and every 24 hours for method 624.

Following successful tuning, the GC/MS is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of three standards for method 624, 524.2 and five standards for method 8260B/C and SM6200B. The calibration standards are tabulated according to peak height or area against concentration and the concentrations and responses of the internal standard analytes. The results are used to determine a response factor for each analyte in each standard injected. A calibration curve is constructed and is determined to be acceptable if each target analyte is found to be constant over the working range as defined as:

- $\leq 15\%$  RSD for methods 8260B
- $\leq 20\%$  RSD for method 524.2, 8260C, SM6200B
- $\leq 35\%$  RSD for method 624



Per the analytical method, specific target analytes are defined as calibration check compounds (CCCs) or system performance check compounds (SPCCs). The calibration checks compounds (CCCs) for method 8260B must be  $\leq 30\%$  RSD. When these conditions are met, linearity through the origin can be assumed and the average RF can be used in place of a calibration curve.

Linear regression can be used for any target compound exceeding the RSD criteria but less than 40% (poor performers  $< 50\%$ ), if the correlation coefficient is 0.990 or better. For USACE/DOD projects the correlation coefficient must meet 0.995 or better. The same is true for the CCCs in EPA 8260B as long as the RSD does not exceed 30%.

<b>8260B SPCCs:</b>	
<b>Analyte</b>	<b>Minimum Average Response Factor</b>
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

<b>8260B CCCs:</b>	
1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl Chloride

The initial calibration range must represent the typical environmental sample and include the RL as the lowest calibration point. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range.

A second source calibration verification standard is analyzed after each calibration. The second source should recover within 30% for all CCC compounds and within 40% for other analytes of interest, with the exception of analytes known to perform poorly (i.e. low purging efficiency, etc.) that will meet historical LCS accuracy limits. For 524.2 the second source calibration verification standard must be within  $\pm 30\%$ . Following successful calibration, the analysis of field and QC samples may begin. Sample analysis may be performed only during the timeframe of a valid tuning cycle (12 hours for 8260B, 524.2 & SM6200B and 24 hours for 624). Following the expiration of the tuning clock, the instrument must be re-tuned and either recalibrated or the existing calibration must be re-verified.

For 8260B/C, 524.2 & SM6200B analyses, daily calibration verification includes successful demonstration of BFB sensitivity and the injection of a mid-level CCV standard containing all the target analytes of interest and all required system monitoring compounds, where applicable. The BFB tune must meet the ion abundance criteria (see table above). For 8260B, each SPCC in the calibration verification standard must meet the minimum response factors listed above. The CCC must achieve the criteria of  $\pm 20\%$  RSD. For 524.2 & SM6200B, each target analyte must achieve a drift or difference

of +/-30% of the expected concentration. For V8260C each target analyte must achieve a drift or difference of +/-20% of the expected concentration.

Each internal standard in the CCV must recover between -50% to + 100%, when compared to the same internal standard compound in the mid-point standard of the initial calibration curve. Additionally, if the retention time of an internal standard changes by more than 30 seconds from the retention time of the same internal standard in the mid-level standard of the most recent initial calibration, the system must be evaluated, corrected, and possibly re-calibrated.

Daily calibration is accomplished for method 624 by a BFB tuning and analysis of a QC check standard. The BFB tune must meet EPA ion abundance criteria. The QC check standard must meet the criteria found in table 5 of the method.

Poor performing compounds for 8260B/524.2/SM6200B/624:

Propene	2-Chloroethylvinyl Ether
Dichlorodifluoromethane	Acrolein
Carbon Disulfide	Vinyl acetate
Bromomethane	trans-1,4-dichloro-2-butene
Chloroethane	Alcohols (Ethanol, TBA, TAA, ETBA, Butanol)
1,3-Butadiene	Iodomethane.
2,2-Dibromo-3-chloropropane	Naphthalene
1- Methyl-naphthalene	2-Butanone
2- Methyl-naphthalene	2-Hexanone
Acetone	4-Methyl-2-pentanone
Pentachloroethane	Cyclohexanone
Tert-butyl Formate	

## 8.5 A ACCEPTANCE/REJECTION OF CALIBRATION

### Organic Chemistry

The initial calibration curve is compared with previous curves for the same analyte. All new standard curves are immediately checked with a secondary source or laboratory control standard prepared from a separate source than those used for calibration. All curves are visually reviewed to ensure that acceptable correlation represents linearity. Calibration curves may be rejected for nonlinearity, abnormal sensitivity, or poor response of the laboratory control standard.

Continuing calibration verification is performed on each day that initial calibration is not performed and following every tenth sample for GC analyses and once per 12 hour shift for GCMS analyses. If a check standard does not perform within established criteria, the instrument is evaluated to determine the cause. Once the issue is corrected, all samples between the last in control sample and the first out of control check is re-analyzed.

**TABLE 8.5: INSTRUMENT CALIBRATION**

Instrument (Analysis)	Calibration Type	Minimum Number of Standards	Calibration Model	Acceptance/ Rejection Criteria	Frequency
GC (VOC)	Initial	3 –600 series	Avg. RF	Must be ≤10% RSD for 601/602,	As needed
		5 –All others	Avg. RF	≤20%RSD for 8021B, and ≤20% difference for 8015B	
	Second Source	1 Second Source	External	+/- 20% of true value	With each calibration
	Daily / Cont.	1/10	External	Must be within 20% of the initial calibration curve	Beginning, every 10 and ending
			Internal	Must be within 20% of the initial calibration curve	Every 12 hours
GC/MS VOC 8260/SM 6200B	Initial	5 –8000 series & SM 6200B	Avg. RF	8260B - Must be ≤15 %RSD for all target analytes and ≤30% for CCCs. 8260C - Must be ≤20 %RSD for all target analytes and ≤30% for CCCs. 6200B - Must be ≤20 %RSD for all target analytes. If Linear regression is used, an MRL check must pass +/-30%.	As needed
	Second Source	1 Second Source		8260B - Should recover within 20% for all CCC compounds and within 40% for other analytes of interest, with the exception of analytes known to perform poorly. 6200B – Should recover within 30% for all compounds, with the exception of analytes known to perform poorly	With each calibration
	Daily / Cont.	Tune & CCV every 12 hours		8260B/C - Must pass established method tuning criteria; 8260B - CCV must be ≤20% difference for CCC compounds, RF criteria for SPCC compounds must meet method criteria. Targets must meet ESC %drift criteria. 8260C/6200B – All targets meet designated minimum response factor, and all compounds ≤20% difference and 30% difference respectively, however for EPA 8260C, 20% of target compounds can fail. If any failures, an MRL check is analyzed.	Every 12 hours

Instrument (Analysis)	Calibration Type	Minimum Number of Standards	Calibration Model	Acceptance/ Rejection Criteria	Frequency
GC/MS VOC 624	Initial	3 –600 series	Avg. RF	624 - Must be ≤35 %RSD for all target analytes and ≤30% for CCCs	As needed
	Second Source	1 Second Source		Should recover within 20% for all CCC compounds and within 40% for other analytes of interest, with the exception of analytes known to perform poorly	With each calibration
	Daily / Cont.	Tune & CCV every 12 hours		Must pass established method tuning criteria; 624 - CCV must be ≤20% difference for CCC, RF for SPCC compounds must meet method criteria. Targets must meet ESC %drift criteria.	Every 12 hours

## 9.0 LABORATORY PRACTICES

### 9.1 REAGENT GRADE WATER

Reagent grade water is obtained from an ELGA Purelab Ultra system.

### 9.2 GLASSWARE WASHING PROCEDURE

All VOA sampling vials are purchased specifically for volatiles analysis and only used once. They are stored in a contaminant-free environment in the original carton with screw cap lids tightly fastened. All glassware used for volatiles analysis (volumetric flasks, syringes, etc.) is segregated from other laboratory glassware. Standard cleaning procedures involve rinsing three times with methanol. When a highly contaminated sample is purged, a blank is analyzed before another sample can be purged to ensure cleanliness of the analytical system. If the blank proves to be contaminant free, the system is then ready for further field sample analyses.

## 10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOPs associated with the volatiles laboratory can be found in the following table:

**TABLE 10.1: VOLATILE DEPARTMENT SOPS**

*This table is subject to revision without notice*

SOP #	Title/Description
330351	BTEX and Gasoline Range Organics by Gas Chromatography (8015B)
330351A	8015 TN GRO Using Component Standards
330354	MA Volatile Petroleum Hydrocarbons
330357	Volatile Organic Compounds (GRO by GCMS)
330363OH	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry 8260A/B (Ohio VAP)

SOP #	Title/Description
330363	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry
330364	DW Volatile Organic Compounds by GC/MS (524.2)
330365	VOC Screen using RAE Systems PID ppbRAE
330375	GRO Analysis in Air (based on EPA 8015)
330751	5035 Closed System Purge and Trap and Extraction for VOC's in Soil and Waste
330751OH	5035 Closed System Purge and Trap and Extraction for VOC's in Soil for Ohio VAP
330752OH	5030B Purge and Trap for OH VAP Samples
330752	5030B Purge and Trap for Aqueous Samples

## 11.0 QUALITY CONTROL CHECKS

**NOTE:** For specific guidance on each determinative method, including required quality control and specific state requirements/modifications, refer to the relevant laboratory standard operating procedure(s).

- 11.1 ESC participates in proficiency testing (PTs) in support of various laboratory accreditations/recognitions. Environmental samples are purchased from Phenova. The WS, WP and solid matrix studies are completed every 6 months. PT samples are received and analyzed by method according to the vendor's instructions and according to ESC SOP.
- 11.2 Initial Demonstrations of Capability (IDOCs) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability (CDOCs) must be updated at least annually. The associated data is filed within the department and available for review.
- 11.3 Matrix Spike and Matrix Spike Duplicates are performed on each batch of samples analyzed depending on analytical method requested.
- 11.4 A Laboratory Control Sample (LCS) and LCS Duplicate (LCSD) are analyzed one per batch of samples.
- 11.5 A method preparation blank is performed per batch of samples processed. If the acceptance criteria as listed in the determinative SOP is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary, based on the following criteria:
  - The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch or
  - The blank contamination is greater than 1/10 of the specified regulatory limit. The concentrations of common laboratory contaminants shall not exceed the reporting limit.

Any samples associated with a blank that fail these criteria shall be reprocessed in a subsequent preparation batch, except where the sample analysis resulted in non-detected results for the failing analytes.

## 12.0 DATA REDUCTION, VALIDATION AND REPORTING

### 12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in SOP #030201, *Data Handling and Reporting*. A secondary review of the data package is performed according to ESC SOP #030227, *Data Review*. The reviewer verifies that the analysis has been performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required flags on test reports, etc.)

**TABLE 12.1 Data Reduction Formulas**

PARAMETER	FORMULA
GC	$\frac{\text{response of sample analyte } \{area\} \times \text{final extract volume } \{mL\} \times \text{dilution}}{\text{response factor } \{area/(mg/L)\} \times \text{initial extract volume-mass } \{mL \text{ or } g\}}$ <p style="text-align: center;"><i>Calculations performed by HP Enviroquant Software</i></p>
GC/MS	$\frac{\text{response of analyte } \{area\} \times \text{extract volume } \{mL\} \times \text{dilution} \times \text{int. std amt. } \{area\}}{\text{response factor } \{area/(mg/mL)\} \times \text{initial volume-mass } \{mL \text{ or } g\} \times \text{int. std cal. } \{area\}}$ <p style="text-align: center;"><i>Calculations performed by HP Enviroquant Software</i></p>

### 12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.3 by method for current QC targets and controls and current reporting limits.

**Marginal Exceedance** – When a large number of analytes exist in the LCS, it is statistically possible for a few analytes to be outside established control limits while the analytical system remains in control. These excursions must be random in nature and, if not, a review of the control limits or analytical process is necessary.

Upper and lower marginal exceedance (ME) limits are established as the mean of at least 20 data points  $\pm$  four times their standard deviations. The number of allowable marginal exceedances per event is based on the number of analytes spiked in the LCS.

Allowable Marginal Exceedance per Event	
Analytes in LCS:	ME Allowable
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

**Organic Control Limits** - The organic QC targets are statutory in nature; warning and control limits for organic analyses are initially established for groups of compounds based on preliminary method validation data. When additional data becomes available, the QC targets are reviewed. All QC targets are routinely re-evaluated at least annually (and updated, if necessary) against laboratory historical data to insure that the limits continue to reflect realistic, method achievable goals.

### 12.3 REPORTING

Reporting procedures are documented in SOP #030201, *Data Handling and Reporting*.

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RLs This table is subject to revision without notice							
Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	1,1,1,2-TETRACHLOROETHANE	8260B/C, 624, 6200B	GW, WW	78.5-125	20.0	0.001	mg/L
Volatiles	1,1,1-TRICHLOROETHANE	8260B/C, 624, 6200B	GW, WW	71.1-129	20.0	0.001	mg/L
Volatiles	1,1,2,2-TETRACHLOROETHANE	8260B/C, 624, 6200B	GW, WW	79.3-123	20.0	0.001	mg/L
Volatiles	1,1,2-TRICHLOROETHANE	8260B/C, 624, 6200B	GW, WW	81.6-120	20.0	0.001	mg/L
Volatiles	1,1,2-TRICHLORO-TRIFLUOROETHANE	8260B/C, 624, 6200B	GW, WW	62.0-141	20.0	0.001	mg/L
Volatiles	1,1-DICHLOROETHANE	8260B/C, 624, 6200B	GW, WW	71.7-127	20.0	0.001	mg/L
Volatiles	1,1-DICHLOROETHENE	8260B/C, 624, 6200B	GW, WW	59.9-137	20.0	0.001	mg/L
Volatiles	1,1-DICHLOROPROPENE	8260B/C, 624, 6200B	GW, WW	72.5-127	20.0	0.001	mg/L
Volatiles	1,2,3-TRICHLOROBENZENE	8260B/C, 624, 6200B	GW, WW	75.7-134	20.0	0.001	mg/L
Volatiles	1,2,3-TRICHLOROPROPANE	8260B/C, 624, 6200B	GW, WW	74.9-124	20.0	0.0025	mg/L
Volatiles	1,2,3-TRIMETHYLBENZENE	8260B/C, 624, 6200B	GW, WW	79.9-118	20.0	0.001	mg/L
Volatiles	1,2,4-TRICHLOROBENZENE	8260B/C, 624, 6200B	GW, WW	76.1-136	20.0	0.001	mg/L
Volatiles	1,2,4-TRIMETHYLBENZENE	8260B/C, 624, 6200B	GW, WW	79.0-122	20.0	0.001	mg/L
Volatiles	1,2-DIBROMO-3-CHLOROPROPANE	8260B/C, 624, 6200B	GW, WW	64.8-131	20.0	0.005	mg/L
Volatiles	1,2-DIBROMOETHANE	8260B/C, 624, 6200B	GW, WW	79.8-122	20.0	0.001	mg/L
Volatiles	1,2-DICHLOROBENZENE	8260B/C, 624, 6200B	GW, WW	84.7-118	20.0	0.001	mg/L
Volatiles	1,2-DICHLOROETHANE	8260B/C, 624, 6200B	GW, WW	65.3-126	20.0	0.001	mg/L

**Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RLs**  
*This table is subject to revision without notice*

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	1,2-DICHLOROPROPANE	8260B/C, 624, 6200B	GW, WW	77.4-125	20.0	0.001	mg/L
Volatiles	1,3,5-TRIMETHYLBENZENE	8260B/C, 624, 6200B	GW, WW	81.0-123	20.0	0.001	mg/L
Volatiles	1,3-BUTADIENE	8260B/C, 624, 6200B	GW, WW	36.2-142	20.0	0.002	mg/L
Volatiles	1,3-DICHLOROBENZENE	8260B/C, 624, 6200B	GW, WW	77.6-127	20.0	0.001	mg/L
Volatiles	1,3-DICHLOROPROPANE	8260B/C, 624, 6200B	GW, WW	80.6-115	20.0	0.001	mg/L
Volatiles	1,4-DICHLOROBENZENE	8260B/C, 624, 6200B	GW, WW	82.2-114	20.0	0.001	mg/L
Volatiles	1-METHYLNAPHTHALENE	8260B/C, 624, 6200B	GW, WW	48.8-157	20.0	0.01	mg/L
Volatiles	2,2-DICHLOROPROPANE	8260B/C, 624, 6200B	GW, WW	61.3-134	20.0	0.001	mg/L
Volatiles	2-BUTANONE (MEK)	8260B/C, 624, 6200B	GW, WW	46.4-155	20.0	0.01	mg/L
Volatiles	2-CHLOROETHYL VINYL ETHER	8260B/C, 624, 6200B	GW, WW	23.4-162	23.5	0.05	mg/L
Volatiles	2-CHLOROTOLUENE	8260B/C, 624, 6200B	GW, WW	76.4-125	20.0	0.001	mg/L
Volatiles	2-HEXANONE	8260B/C, 624, 6200B	GW, WW	59.4-151	20.0	0.01	mg/L
Volatiles	2-METHYLNAPHTHALENE	8260B/C, 624, 6200B	GW, WW	55.6-154	20.0	0.01	mg/L
Volatiles	4-CHLOROTOLUENE	8260B/C, 624, 6200B	GW, WW	81.5-121	20.0	0.001	mg/L
Volatiles	4-ETHYLTOLUENE	8260B/C, 624, 6200B	GW, WW	69.5-137	20.0	0.001	mg/L
Volatiles	4-METHYL-2-PENTANONE (MIBK)	8260B/C, 624, 6200B	GW, WW	63.3-138	20.0	0.01	mg/L
Volatiles	ACETONE	8260B/C, 624, 6200B	GW, WW	28.7-175	20.9	0.01	mg/L
Volatiles	ACROLEIN	8260B/C, 624, 6200B	GW, WW	40.4-172	20.0	0.05	mg/L
Volatiles	ACRYLONITRILE	8260B/C, 624, 6200B	GW, WW	58.2-145	20.0	0.01	mg/L
Volatiles	BENZENE	8260B/C, 624, 6200B	GW, WW	73.0-122	20.0	0.001	mg/L
Volatiles	BROMOBENZENE	8260B/C, 624, 6200B	GW, WW	81.5-115	20.0	0.001	mg/L
Volatiles	BROMOCHLOROMETHANE	8260B/C, 624, 6200B	GW, WW	78.9-123	20.0	0.001	mg/L
Volatiles	BROMODICHLOROMETHANE	8260B/C, 624, 6200B	GW, WW	75.5-121	20.0	0.001	mg/L
Volatiles	BROMOFORM	8260B/C, 624, 6200B	GW, WW	71.5-131	20.0	0.001	mg/L
Volatiles	BROMOMETHANE	8260B/C, 624, 6200B	GW, WW	22.4-187	20.0	0.005	mg/L
Volatiles	CARBON DISULFIDE	8260B/C, 624, 6200B	GW, WW	53.0-134	20.0	0.001	mg/L
Volatiles	CARBON TETRACHLORIDE	8260B/C, 624, 6200B	GW, WW	70.9-129	20.0	0.001	mg/L
Volatiles	CHLOROBENZENE	8260B/C, 624, 6200B	GW, WW	79.7-122	20.0	0.001	mg/L
Volatiles	CHLORODIBROMOMETHANE	8260B/C, 624, 6200B	GW, WW	78.2-124	20.0	0.001	mg/L
Volatiles	CHLOROETHANE	8260B/C, 624, 6200B	GW, WW	41.2-153	20.0	0.005	mg/L
Volatiles	CHLOROFORM	8260B/C, 624, 6200B	GW, WW	73.2-125	20.0	0.005	mg/L
Volatiles	CHLOROMETHANE	8260B/C, 624, 6200B	GW, WW	55.8-134	20.0	0.025	mg/L
Volatiles	CIS-1,2-DICHLOROETHENE	8260B/C, 624, 6200B	GW, WW	77.3-122	20.0	0.001	mg/L
Volatiles	CIS-1,3-DICHLOROPROPENE	8260B/C, 624, 6200B	GW, WW	77.7-124	20.0	0.001	mg/L
Volatiles	DIBROMOMETHANE	8260B/C, 624, 6200B	GW, WW	78.8-119	20.0	0.001	mg/L



**Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RLs**  
*This table is subject to revision without notice*

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	DICHLORODIFLUOROMETHANE	8260B/C, 624, 6200B	GW, WW	56.0-134	20.0	0.005	mg/L
Volatiles	DICHLOROFLUOROMETHANE	8260B/C, 624, 6200B	GW, WW	53.5-145	20.0	0.005	mg/L
Volatiles	DICYCLOPENTADIENE	8260B/C, 624, 6200B	GW, WW	73.4-126	20.0	0.001	mg/L
Volatiles	DI-ISOPROPYL ETHER	8260B/C, 624, 6200B	GW, WW	65.1-135	20.0	0.001	mg/L
Volatiles	ETHYL ETHER	8260B/C, 624, 6200B	GW, WW	56.6-136	20.0	0.001	mg/L
Volatiles	ETHYLBENZENE	8260B/C, 624, 6200B	GW, WW	80.9-121	20.0	0.001	mg/L
Volatiles	HEXACHLORO-1,3-BUTADIENE	8260B/C, 624, 6200B	GW, WW	73.7-133	20.0	0.001	mg/L
Volatiles	IODOMETHANE	8260B/C, 624, 6200B	GW, WW	64.6-137	20.0	0.01	mg/L
Volatiles	ISOPROPYLBENZENE	8260B/C, 624, 6200B	GW, WW	81.6-124	20.0	0.001	mg/L
Volatiles	M&P-XYLENE	8260B/C, 624, 6200B	GW, WW	78.5-122	20.0	0.002	mg/L
Volatiles	METHYL TERT-BUTYL ETHER	8260B/C, 624, 6200B	GW, WW	70.1-125	20.0	0.001	mg/L
Volatiles	METHYLENE CHLORIDE	8260B/C, 624, 6200B	GW, WW	69.5-120	20.0	0.005	mg/L
Volatiles	NAPHTHALENE	8260B/C, 624, 6200B	GW, WW	69.7-134	20.0	0.005	mg/L
Volatiles	N-BUTYLBENZENE	8260B/C, 624, 6200B	GW, WW	75.9-134	20.0	0.001	mg/L
Volatiles	N-HEXANE	8260B/C, 624, 6200B	GW, WW	59.5-132	20.0	0.01	mg/L
Volatiles	N-PROPYLBENZENE	8260B/C, 624, 6200B	GW, WW	81.9-122	20.0	0.001	mg/L
Volatiles	O-XYLENE	8260B/C, 624, 6200B	GW, WW	79.1-123	20.0	0.001	mg/L
Volatiles	P-ISOPROPYLTOLUENE	8260B/C, 624, 6200B	GW, WW	77.6-129	20.0	0.001	mg/L
Volatiles	PROPENE	8260B/C, 624, 6200B	GW, WW	10.0-200	20.0	0.0025	mg/L
Volatiles	SEC-BUTYLBENZENE	8260B/C, 624, 6200B	GW, WW	80.6-126	20.0	0.001	mg/L
Volatiles	STYRENE	8260B/C, 624, 6200B	GW, WW	79.9-124	20.0	0.001	mg/L
Volatiles	TERT-BUTYLBENZENE	8260B/C, 624, 6200B	GW, WW	79.3-127	20.0	0.001	mg/L
Volatiles	TETRACHLOROETHENE	8260B/C, 624, 6200B	GW, WW	73.5-130	20.0	0.001	mg/L
Volatiles	TETRAHYDROFURAN	8260B/C, 624, 6200B	GW, WW	54.0-134	20.0	0.005	mg/L
Volatiles	TOLUENE	8260B/C, 624, 6200B	GW, WW	77.9-116	20.0	0.005	mg/L
Volatiles	TPH (GC/MS) LOW FRACTION	8260B/C, 624, 6200B	GW, WW	62.3-131	20.0	0.50	mg/L
Volatiles	TRANS-1,2-DICHLOROETHENE	8260B/C, 624, 6200B	GW, WW	72.6-125	20.0	0.001	mg/L
Volatiles	TRANS-1,3-DICHLOROPROPENE	8260B/C, 624, 6200B	GW, WW	73.5-127	20.0	0.001	mg/L
Volatiles	TRANS-1,4-DICHLORO-2-BUTENE	8260B/C, 624, 6200B	GW, WW	58.3-129	20.0	0.0025	mg/L
Volatiles	TRICHLOROETHENE	8260B/C, 624, 6200B	GW, WW	79.5-121	20.0	0.001	mg/L
Volatiles	TRICHLOROFLUOROMETHANE	8260B/C, 624, 6200B	GW, WW	49.1-157	20.0	0.005	mg/L
Volatiles	VINYL ACETATE	8260B/C, 624, 6200B	GW, WW	41.7-159	20.0	0.01	mg/L

**Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RLs**  
*This table is subject to revision without notice*

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	VINYL CHLORIDE	8260B/C, 624, 6200B	GW, WW	61.5-134	20.0	0.001	mg/L
Volatiles	XYLENES, TOTAL	8260B/C, 624, 6200B	GW, WW	79.2-122	20.0	0.002	mg/L
Volatiles	1,1,1,2-TETRACHLOROETHANE	8260B/C	Solid	76.7-127	20.0	0.001	mg/kg
Volatiles	1,1,1-TRICHLOROETHANE	8260B/C	Solid	69.9-127	20.0	0.001	mg/kg
Volatiles	1,1,2,2-TETRACHLOROETHANE	8260B/C	Solid	78.8-124	20.0	0.001	mg/kg
Volatiles	1,1,2-TRICHLOROETHANE	8260B/C	Solid	81.9-119	20.0	0.001	mg/kg
Volatiles	1,1,2-RICHLOROTRIFLUOROETHANE	8260B/C	Solid	62.6-138	20.0	0.001	mg/kg
Volatiles	1,1-DICHLOROETHANE	8260B/C	Solid	71.7-125	20.0	0.001	mg/kg
Volatiles	1,1-DICHLOROETHENE	8260B/C	Solid	60.6-133	20.0	0.001	mg/kg
Volatiles	1,1-DICHLOROPROPENE	8260B/C	Solid	71.2-126	20.0	0.001	mg/kg
Volatiles	1,2,3-TRICHLOROBENZENE	8260B/C	Solid	72.5-137	20.0	0.001	mg/kg
Volatiles	1,2,3-TRICHLOROPROPANE	8260B/C	Solid	74.0-124	20.0	0.0025	mg/kg
Volatiles	1,2,3-TRIMETHYLBENZENE	8260B/C	Solid	79.4-118	20.0	0.001	mg/kg
Volatiles	1,2,4-TRICHLOROBENZENE	8260B/C	Solid	74.0-137	20.0	0.001	mg/kg
Volatiles	1,2,4-TRIMETHYLBENZENE	8260B/C	Solid	77.1-124	20.0	0.001	mg/kg
Volatiles	1,2-DIBROMO-3-CHLOROPROPANE	8260B/C	Solid	64.9-131	20.0	0.005	mg/kg
Volatiles	1,2-DIBROMOETHANE	8260B/C	Solid	78.7-123	20.0	0.001	mg/kg
Volatiles	1,2-DICHLOROBENZENE	8260B/C	Solid	83.6-119	20.0	0.001	mg/kg
Volatiles	1,2-DICHLOROETHANE	8260B/C	Solid	67.2-121	20.0	0.001	mg/kg
Volatiles	1,2-DICHLOROPROPANE	8260B/C	Solid	76.9-123	20.0	0.001	mg/kg
Volatiles	1,3,5-TRIMETHYLBENZENE	8260B/C	Solid	79.0-125	20.0	0.001	mg/kg
Volatiles	1,3-BUTADIENE	8260B/C	Solid	35.1-134	20.0	0.002	mg/kg
Volatiles	1,3-DICHLOROBENZENE	8260B/C	Solid	75.9-129	20.0	0.001	mg/kg
Volatiles	1,3-DICHLOROPROPANE	8260B/C	Solid	80.3-114	20.0	0.001	mg/kg
Volatiles	1,4-DICHLOROBENZENE	8260B/C	Solid	81.0-115	20.0	0.001	mg/kg
Volatiles	1-METHYLNAPHTHALENE	8260B/C	Solid	60.4-138	24.7	0.01	mg/kg
Volatiles	2,2-DICHLOROPROPANE	8260B/C	Solid	61.9-132	20.0	0.001	mg/kg
Volatiles	2-BUTANONE (MEK)	8260B/C	Solid	44.5-154	21.3	0.01	mg/kg
Volatiles	2-CHLOROETHYL VINYL ETHER	8260B/C	Solid	16.7-162	23.7	0.05	mg/kg
Volatiles	2-CHLOROTOLUENE	8260B/C	Solid	74.6-127	20.0	0.001	mg/kg
Volatiles	2-HEXANONE	8260B/C	Solid	62.7-150	20.0	0.01	mg/kg
Volatiles	2-METHYLNAPHTHALENE	8260B/C	Solid	63.3-137	21.5	0.01	mg/kg
Volatiles	4-CHLOROTOLUENE	8260B/C	Solid	79.5-123	20.0	0.001	mg/kg
Volatiles	4-ETHYLTOLUENE	8260B/C	Solid	78.0-127	20.0	0.001	mg/kg

**Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RLs**  
*This table is subject to revision without notice*

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	4-METHYL-2-PENTANONE (MIBK)	8260B/C	Solid	61.1-138	20.0	0.01	mg/kg
Volatiles	ACETONE	8260B/C	Solid	25.3-178	22.9	0.01	mg/kg
Volatiles	ACROLEIN	8260B/C	Solid	41.0-182	20.0	0.05	mg/kg
Volatiles	ACRYLONITRILE	8260B/C	Solid	57.8-143	20.0	0.01	mg/kg
Volatiles	BENZENE	8260B/C	Solid	72.6-120	20.0	0.001	mg/kg
Volatiles	BROMOBENZENE	8260B/C	Solid	80.3-115	20.0	0.001	mg/kg
Volatiles	BROMOCHLOROMETHANE	8260B/C	Solid	79.7-123	20.0	0.001	mg/kg
Volatiles	BROMODICHLOROMETHANE	8260B/C	Solid	75.3-119	20.0	0.001	mg/kg
Volatiles	BROMOFORM	8260B/C	Solid	69.1-135	20.0	0.001	mg/kg
Volatiles	BROMOMETHANE	8260B/C	Solid	23.0-191	20.0	0.005	mg/kg
Volatiles	CARBON DISULFIDE	8260B/C	Solid	49.9-136	20.0	0.001	mg/kg
Volatiles	CARBON TETRACHLORIDE	8260B/C	Solid	69.4-129	20.0	0.001	mg/kg
Volatiles	CHLOROBENZENE	8260B/C	Solid	78.9-122	20.0	0.001	mg/kg
Volatiles	CHLORODIBROMOMETHANE	8260B/C	Solid	76.4-126	20.0	0.005	mg/kg
Volatiles	CHLOROETHANE	8260B/C	Solid	47.2-147	20.0	0.005	mg/kg
Volatiles	CHLOROFORM	8260B/C	Solid	73.3-122	20.0	0.025	mg/kg
Volatiles	CHLOROMETHANE	8260B/C	Solid	53.1-135	20.0	0.001	mg/kg
Volatiles	CIS-1,2-DICHLOROETHENE	8260B/C	Solid	76.1-121	20.0	0.001	mg/kg
Volatiles	CIS-1,3-DICHLOROPROPENE	8260B/C	Solid	77.3-123	20.0	0.001	mg/kg
Volatiles	DIBROMOMETHANE	8260B/C	Solid	78.5-117	20.0	0.005	mg/kg
Volatiles	DICHLORODIFLUOROMETHANE	8260B/C	Solid	50.9-139	20.0	0.005	mg/kg
Volatiles	DICHLOROFLUOROMETHANE	8260B/C	Solid	61.8-140	20.0	0.001	mg/kg
Volatiles	DICYCLOPENTADIENE	8260B/C	Solid	73.1-126	20.0	0.001	mg/kg
Volatiles	DI-ISOPROPYL ETHER	8260B/C	Solid	67.2-131	20.0	0.001	mg/kg
Volatiles	ETHYL ETHER	8260B/C	Solid	57.5-136	20.0	0.001	mg/kg
Volatiles	ETHYLBENZENE	8260B/C	Solid	78.6-124	20.0	0.001	mg/kg
Volatiles	HEXACHLORO-1,3-BUTADIENE	8260B/C	Solid	69.2-136	20.0	0.01	mg/kg
Volatiles	IODOMETHANE	8260B/C	Solid	63.3-136	20.0	0.001	mg/kg
Volatiles	ISOPROPYLBENZENE	8260B/C	Solid	79.4-126	20.0	0.002	mg/kg
Volatiles	M&P-XYLENE	8260B/C	Solid	77.3-124	20.0	0.001	mg/kg
Volatiles	METHYL TERT-BUTYL ETHER	8260B/C	Solid	70.2-122	20.0	0.005	mg/kg
Volatiles	METHYLENE CHLORIDE	8260B/C	Solid	68.2-119	20.0	0.005	mg/kg
Volatiles	NAPHTHALENE	8260B/C	Solid	69.9-132	20.0	0.001	mg/kg
Volatiles	N-BUTYLBENZENE	8260B/C	Solid	74.2-134	20.0	0.01	mg/kg
Volatiles	N-HEXANE	8260B/C	Solid	59.9-125	20.0	0.001	mg/kg

**Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RLs**  
*This table is subject to revision without notice*

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	N-PROPYLBENZENE	8260B/C	Solid	80.2-124	20.0	0.001	mg/kg
Volatiles	O-XYLENE	8260B/C	Solid	78.5-124	20.0	0.001	mg/kg
Volatiles	P-ISOPROPYLTOLUENE	8260B/C	Solid	75.4-132	20.0	0.0025	mg/kg
Volatiles	PROPENE	8260B/C	Solid	10.0-192	26.1	0.001	mg/kg
Volatiles	SEC-BUTYLBENZENE	8260B/C	Solid	77.8-129	20.0	0.001	mg/kg
Volatiles	STYRENE	8260B/C	Solid	79.4-124	20.0	0.001	mg/kg
Volatiles	TERT-BUTYLBENZENE	8260B/C	Solid	77.2-129	20.0	0.001	mg/kg
Volatiles	TETRACHLOROETHENE	8260B/C	Solid	71.1-133	20.0	0.005	mg/kg
Volatiles	TETRAHYDROFURAN	8260B/C	Solid	63.4-122	20.0	0.005	mg/kg
Volatiles	TOLUENE	8260B/C	Solid	76.7-116	20.0	0.50	mg/kg
Volatiles	TPH (GC/MS) LOW FRACTION	8260B/C	Solid	61.5-138	20.0	0.001	mg/kg
Volatiles	TRANS-1,2-DICHLOROETHENE	8260B/C	Solid	70.7-124	20.0	0.001	mg/kg
Volatiles	TRANS-1,3-DICHLOROPROPENE	8260B/C	Solid	73.0-127	20.0	0.0025	mg/kg
Volatiles	TRANS-1,4-DICHLORO-2-BUTENE	8260B/C	Solid	58.4-125	20.0	0.001	mg/kg
Volatiles	TRICHLOROETHENE	8260B/C	Solid	77.2-122	20.0	0.001	mg/kg
Volatiles	TRICHLOROFLUOROMETHANE	8260B/C	Solid	51.5-151	20.0	0.005	mg/kg
Volatiles	VINYL ACETATE	8260B/C	Solid	39.8-156	20.0	0.01	mg/kg
Volatiles	VINYL CHLORIDE	8260B/C	Solid	58.4-134	20.0	0.001	mg/kg
Volatiles	XYLENES, TOTAL	8260B/C	Solid	78.1-123	20.0	0.002	mg/kg
Volatiles	DI-ISOPROPYL ETHER	8260B/C	Solid	70.4-133	20.0	0.001	mg/kg
Volatiles	ETHYL TERT-BUTYL ETHER	8260B/C	Solid	81.4-110	25.0	0.001	mg/kg
Volatiles	METHYL-TERT-BUTYL ETHER	8260B/C	Solid	73.0-129	20.0	0.001	mg/kg
Volatiles	TERT-BUTYL ALCOHOL	8260B/C	Solid	59.5-170	25.0	0.050	mg/kg
Volatiles	TERT-AMYL METHYL ETHER	8260B/C	Solid	82-115	25.0	0.001	mg/kg
Volatiles	2-PROPANOL	8260B/C	Solid	70.0-130	25.0	0.05	mg/kg
Volatiles	GRO	8015B/C/D	GW, WW	66.3-133	20.0	0.100	mg/L
Volatiles	BENZENE	8021B, 602, 6200C	GW, WW	70.0-130	20.0	0.0005	mg/L
Volatiles	TOLUENE	8021B, 602, 6200C	GW, WW	70.0-130	20.0	0.005	mg/L
Volatiles	ETHYLBENZENE	8021B, 602, 6200C	GW, WW	70.0-130	20.0	0.0005	mg/L
Volatiles	M&P-XYLENE	8021B, 602, 6200C	GW, WW	70.0-130	20.0	0.001	mg/L
Volatiles	O-XYLENE	8021B, 602, 6200C	GW, WW	70.0-130	20.0	0.0005	mg/L
Volatiles	MTBE	8021B, 602, 6200C	GW, WW	70.0-130	20.0	0.001	mg/L
Volatiles	GRO	8015B/C/D	Solid	63.6-136	20.0	0.10	mg/kg
Volatiles	BENZENE	8021B	Solid	70.0 - 130	20.0	0.0005	mg/kg

**Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RLs**  
*This table is subject to revision without notice*

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	TOLUENE	8021B	Solid	70.0 - 130	20.0	0.005	mg/kg
Volatiles	ETHYLBENZENE	8021B	Solid	70.0 - 130	20.0	0.001	mg/kg
Volatiles	M&P-XYLENE	8021B	Solid	70.0 - 130	20.0	0.001	mg/kg
Volatiles	O-XYLENE	8021B	Solid	70.0 - 130	20.0	0.0005	mg/kg
Volatiles	MTBE	8021B	Solid	70.0 - 130	20.0	0.001	mg/kg
Volatiles	1,1,1,2-TETRACHLOROETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,1,1-TRICHLOROETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,1,2,2-TETRACHLOROETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,1,2-TRICHLOROETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,1-DICHLOROETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,1-DICHLOROETHENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,1-DICHLOROPROPENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,2,3-TRICHLOROBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,2,3-TRICHLOROPROPANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,2,4-TRICHLOROBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,2,4-TRIMETHYLBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,2-DIBROMO-3-CHLOROPROPANE	524.2	DW	70.0-130	25.0	0.0010	mg/L
Volatiles	1,2-DIBROMOETHANE	524.2	DW	70.0-130	25.0	0.0010	mg/L
Volatiles	1,2-DICHLOROBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,2-DICHLOROETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,2-DICHLOROPROPANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,3,5-TRIMETHYLBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,3-DICHLOROBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,3-DICHLOROPROPANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	1,4-DICHLOROBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	2,2-DICHLOROPROPANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	2-CHLOROTOLUENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	4-CHLOROTOLUENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	4-ISOPROPYLTOLUENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	ACETONE	524.2	DW	70.0-130	25.0	0.01	mg/L
Volatiles	BENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	BROMOBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	BROMOCHLOROMETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	BROMODICHLOROMETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	BROMOFORM	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	BROMOMETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	CARBON TETRACHLORIDE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	CHLOROBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	CHLOROETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	CHLOROFORM	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	CHLOROMETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	CIS-1,2-DICHLOROETHENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	CIS-1,3-DICHLOROPROPENE	524.2	DW	70.0-130	25.0	0.0010	mg/L
Volatiles	DIBROMOMETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	DICHLORODIFLUOROMETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	ETHYLBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	HEXACHLOROBUTADIENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	ISOPROPYLBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	METHYLENE CHLORIDE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	METHYL-T-BUTYL ETHER	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	NAPHTHALENE	524.2	DW	70.0-130	25.0	0.0050	mg/L
Volatiles	N-BUTYLBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	N-PROPYLBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	SEC-BUTYLBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	STYRENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	TERT-BUTYLBENZENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	TETRACHLOROETHENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	TOLUENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	TRANS-1,2-DICHLOROETHENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	TRANS-1,3-DICHLOROPROPENE	524.2	DW	70.0-130	25.0	0.0010	mg/L
Volatiles	TRICHLOROETHENE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	TRICHLOROFLUOROMETHANE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	VINYL CHLORIDE	524.2	DW	70.0-130	25.0	0.0005	mg/L
Volatiles	XYLENES – TOTAL	524.2	DW	70.0-130	25.0	0.0015	mg/L

\*\* Specific organizations may require limits that supersede the values listed.

### 13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CARs are kept on file by the Regulatory Affairs Department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

#### 13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these limits are exceeded, corrective action must be taken. Calculated sample spike control limits are also used.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP.

##### 13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory, the method criteria takes precedence.

##### 13.2.2 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

Rejection Criteria - Blank reading is more than twice the background absorbance or more than ½RL.

Corrective Action - Blanks are re-analyzed and the response is assessed. Standard curves and samples are evaluated for any obvious contamination that is isolated or uniform throughout the run. If necessary, reagents are re-prepared. Analyses are not initiated until the problem is identified and solved. If samples have already been prepared or analyzed, the Department Supervisor is consulted to determine if data needs to be rejected or if samples need to be re-prepared.

##### 13.2.3 Out Of Control Laboratory Control Standards (LCS & LCSD)

Rejection Criteria - If the performance is outside of lab-generated control limits which are calculated as the mean of at least 20 data points +/- 3 times the standard deviation of those points (Listed in Section 12) and the marginal exceedence allowance is surpassed (see section 12.2).

Corrective Action - Instrument settings are checked and the LCS standard is re-analyzed. If the LCS is still out of control, instrumentation is checked for systemic problems and repaired (if necessary). Re-calibration is performed and the samples affected since the last in control reference standard are reanalyzed. The group leader or Department Supervisor is consulted for further action.

#### 13.2.4 Out Of Control Matrix Spike Samples

Rejection Criteria - If either the MS or MSD sample is outside the established control limits.

Corrective Action - Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance, however, is based on method blank and LCS performance, not on MS/MSD recoveries. Specific methods, customers, and programs may require further corrective action in some cases.

#### 13.2.5 Out Of Control Duplicate Samples

Rejection Criteria - Lab-generated maximum RPD limit (as listed under precision in Section 12)

Corrective Action - Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance, however, is based on method blank and LCS performance. Specific methods, customers, and programs may require further corrective action in some cases.

#### 13.2.7 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

Corrective Action - Instrument settings are checked, calibration verification standard is re-analyzed. If the standard is still out of control, re-calibration is performed, and samples affected since the last in control reference standard are rerun. The group leader or Department Supervisor is consulted for further action.

### 14.0 RECORD KEEPING

Record keeping is outlined in SOP #030230, *Standards Logger*, SOP #030227, *Data Review* and SOP #030201, *Data Handling and Reporting*

Volatile organics calibration data are recorded and integrated using HP Enviroquant software. Calibration data from the volatile analyses, in addition to the initial and daily calibration, includes GC/MS autotunes, BFB reports and surrogate recovery reports. PDF records of initial calibration and daily calibration are stored with chromatograms and integrated with sample data by date analyzed.



## 15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 13.0 and *SOP #010104, Internal Audits*.

## 16.0 REVISIONS

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of this appendix are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0 (Appendix VI)	Section 5.1 – Removed language about supervisor and backup reviewing and approving all data Section 6.1 – Removed reagent water is evaluated to 0.055uS/cm Section 8.1 – Updated equipment list Table 8.3B – Updated working standards Section 8.4 – Changed daily detector mass calibration using PFTBA to using BFB for GC/MS methods. Also clarified 8260C CCV criteria is +/- 20% Table 8.5 – Clarified 8260C CCV criteria is +/- 20% Section 9.1 – Clarified what system is used to generate the reagent water Table 10.1 – Removed SOP for 8260B in Air (Arizona) Section 13.2.4 – Reworded MS/MSD criteria and revised corrective action to just qualify unless method, customer, or program states to do something else. Section 13.2.5 - Revised duplicate corrective action to just qualify unless method, customer, or program states to do something else. Section 16 – New section for summary of revisions to previous version

1.0 SIGNATORY APPROVALS

# Semi-Volatile QUALITY ASSURANCE MANUAL

## APPENDIX VII TO THE ESC QUALITY ASSURANCE MANUAL

for


ESC LAB SCIENCES  
12065 LEBANON ROAD  
MT. JULIET, TENNESSEE 37122  
(615) 758-5858


Prepared by

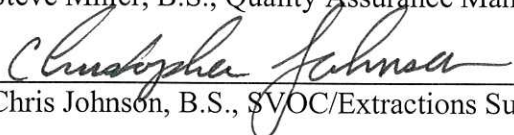
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**NOTE: The QAM has been approved by the following people.**

  
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### **3.0 SCOPE AND APPLICATION**

This appendix discusses specific QA requirements for general analytical protocols to ensure that analytical data generated from the Semi-Volatile (SVOC) laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in non-conforming work must be immediately evaluated and their corrective actions documented.

### **4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES**

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling guidance and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual Version 13.0*.

### **5.0 PERSONNEL AND TRAINING**

#### **5.1 PERSONNEL**

Chris Johnson, with a B.S. degree in Biology, is the SVOC/Extractions Supervisor and is responsible for the overall production of these laboratories; including the management of the staff and scheduling. Mr. Johnson has over 14 years of environmental laboratory experience.

In his absence, Blake Judge assumes responsibility for departmental decisions. Mr. Judge has a B.S. degree in Chemistry and is a Senior Chemist in the SVOC Department. He is proficient in semi-volatile organic analytical methods and has over 9 years of environmental laboratory experience.

#### **5.2 TRAINING**

- 5.2.1 All new analysts to the laboratory are trained by the Senior Chemist or Department Supervisor according to ESC protocol. ESC's training program is outlined in *SOP 030205 Technical Training and Personnel Qualifications*. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in SVOC analyses and preparation is also demonstrated by acceptable participation in multiple proficiency testing programs (PTs) and daily Quality Control sample analyses. Documentation of analyst training is maintained on file within the department.

## **6.0 FACILITIES AND LABORATORY SAFETY**

### **6.1 FACILITIES**

The main area of the instrumentation laboratory in Building #5 has nearly 1500 square feet with approximately 500 square feet of bench area. The 4000 square feet of area in the extraction laboratory includes roughly 330 square feet of bench area with 245 square feet of hood space. There is an additional 2000 square feet of storage for this laboratory. The air system is a 15-ton make-up unit plus 15-ton HVAC with electric heat. The physical and air-handling separations, between this laboratory and other ESC sections, prevent potential cross-contamination between solvent vapor generation and incompatible analytical processes. The laboratory reagent water is provided through the US Filter deionizer system. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's waste disposal carrier as discussed in detail in Section 6.0 of the ESC Quality Assurance Manual. ESC's building information guides and site plan are shown in Appendix I.

### **6.2 LABORATORY SAFETY**

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods.
- ESC's laboratory safety guidelines are detailed in the *ESC Chemical Hygiene Plan*.

## **7.0 SAMPLING PROCEDURES**

### **7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING**

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Matrices for SVOC environmental analyses include groundwater, wastewater, drinking water, soil, and sludge.
- Sample containers, preservation methods and holding times vary depending on analyses requested. Please see determinative procedures for specific directions.
- Plastic containers or lids may NOT be used for the storage of samples due to possible contamination from the phthalate esters and other hydrocarbons.
- Environmental sample containers should be filled carefully to prevent any portion of the sample from coming into contact with the sampler's gloves causing possible phthalate contamination.

## 8.0 EQUIPMENT

### 8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis						
<i>This table is subject to revision without notice</i>						
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Instrument Name</i>	<i>#</i>	<i>Serial #</i>	<i>Location</i>
Gas Chromatograph 2	HP	6890	svcompa	2	US00004397	SVOC
Gas Chromatograph 7	Agilent	6890	svcompe	7	US10350064	SVOC
Gas Chromatograph 8	Agilent	6890	svcompp	8	DE00022534	SVOC
Gas Chromatograph 9	HP	6890	svcompj	9	US00029095	SVOC
Gas Chromatograph 10	Agilent	6890	svcompk	10	US00039655	SVOC
Gas Chromatograph 11	Agilent	6890	svcompn	11	US00040550	SVOC
Gas Chromatograph 12	Agilent	6890	svcompo	12	US00034155	SVOC
Gas Chromatograph 13	HP	6890	svcomps	13	US00010364	SVOC
Gas Chromatograph 14	HP	6890	svcompt	14	US00020581	SVOC
Gas Chromatograph 16	Agilent	6890	svcompv	16	US10212071	SVOC
Gas Chromatograph 17	Agilent	6890	svcompw	17	US10344078	SVOC
Gas Chromatograph 18	Agilent	6890	svcompd	18	US10351038	SVOC
Gas Chromatograph 19	Agilent	6890	svcompaa	19	CN10516070	SVOC
Gas Chromatograph 20	Agilent	6890	svcompab	20	CN10543031	SVOC
Gas Chromatograph 21	Agilent	7890	svcompae	21	CN 10730070	SVOC
Gas Chromatograph 22	Agilent	7890	svcompaf	22	CN 10730081	SVOC
Gas Chromatograph 23	Agilent	6890	svcompag	23	CN 92174366	SVOC
Gas Chromatograph 24	Agilent	6890	svcompah	24	CN 92174369	SVOC
Gas Chromatograph 25	Agilent	7890	svcompaj	25	CN 10091009	SVOC
Gas Chromatograph 26	Agilent	7890	Svcompar	26	CN11501138	SVOC
Gas Chromatograph 27	Agilent	7890	Svcompas	27	CN11501139	SVOC
Gas Chromatograph 28	Agilent	7890	Svcompat	28	US11521018	SVOC
Gas Chromatograph 29	Agilent	7890	Svcompau	29	CN11521077	SVOC
Gas Chromatograph 30	Agilent	7890	svcompav	30	US11521020	SVOC
Gas Chromatograph 31	Agilent	7890	svcompba	31	CN13503096	SVOC
Gas Chromatograph 32	Agilent	7890	svcompbc	32	CN14423060	SVOC
Gas Chromatograph 33	Agilent	7890	svcompbd	33	CN15033026	SVOC
Gas Chromatograph 34	Agilent	7890	svcompbe	34	CN15033027	SVOC
Gas Chromatograph Detectors 3	Detectors	NPD/NPD	svcompo	3	N/A	SVOC
Gas Chromatograph Detectors 7	Detectors	FID	svcompe	7	N/A	SVOC
Gas Chromatograph Detectors 8	Detectors	FID	svcompp	8	N/A	SVOC
Gas Chromatograph Detectors 9	Detectors	FID	svcompj	9	N/A	SVOC
Gas Chromatograph Detectors 10	Detectors	ECD/ECD	svcompk	10	F) U11751 B) U11135	SVOC

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis</b>						
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<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Instrument Name</i>	<i>#</i>	<i>Serial #</i>	<i>Location</i>
Gas Chromatograph Detectors 11	Detectors	ECD/ECD	svcompn	11	F) U12482 B) U12481	SVOC
Gas Chromatograph Detectors 12	Detectors	FPD/FPD	svcompo	12	N/A	SVOC
Gas Chromatograph Detectors 13	Detectors	FID	svcomps	13	N/A	SVOC
Gas Chromatograph Detectors 14	Detectors	ECD/ECD	svcompt	14	F) U0418 B) U6632	SVOC
Gas Chromatograph Detectors 16	Detectors	FID	svcompu	16	N/A	SVOC
Gas Chromatograph Detectors 17	Detectors	FID	svcompv	17	N/A	SVOC
Gas Chromatograph Detectors 18	Detectors	ECD/ECD	svcompd	18	F) U8422 B) U11613	SVOC
Gas Chromatograph Detectors 19	Detectors	ECD/ECD	svcompaa	19	F) U2620 B) U11614	SVOC
Gas Chromatograph Detectors 20	Detectors	ECD/ECD	svcompab	20	F) U8422 B) U8423	SVOC
Gas Chromatograph Detectors 21	Detectors	FID	svcompae	21	N/A	SVOC
Gas Chromatograph Detectors 22	Detectors	ECD/ECD	svcompaf	22	N/A	SVOC
Gas Chromatograph Detectors 23	Detectors	ECD/ECD	svcompag	23	F) U11733 B) U11734	SVOC
Gas Chromatograph Detectors 24	Detectors	ECD/ECD	svcompah	24	F) U13989 B) U13988	SVOC
Gas Chromatograph Detectors 26	Detectors	FID	svcompar	26	N/A	SVOC
Gas Chromatograph Detectors 27	Detectors	FID	svcompas	27	N/A	SVOC
Gas Chromatograph Detectors 28	Detectors	ECD/ECD	Svcompat	28	F) U20406 B) U20407	SVOC
Gas Chromatograph Detectors 29	Detectors	ECD/ECD	svcompau	29	F) U20277 B) U20299	SVOC
Gas Chromatograph Detectors 30	Detectors	ECD/ECD	svcompav	30	F) U20425 B) U20424	SVOC
Gas Chromatograph Detectors 31	Detectors	FID	svcompba	31	N/A	SVOC
Gas Chromatograph Detectors 32	Detectors	FID	svcompbc	32	N/A	SVOC
Gas Chromatograph Detectors 33	Detectors	FID	svcompbd	33	N/A	SVOC
Gas Chromatograph Detectors 34	Detectors	FID	svcompbe	34	N/A	SVOC
Gas Chromatograph/Mass Spectrometer 1	Agilent	6890 GC/5973MSD	svcompf	1	GC CN10335001 MS US33220022	SVOC
Gas Chromatograph/Mass Spectrometer 2	Agilent	6890 GC/5973MSD	svcompc	2	GC US10409048 MS US35120400	SVOC
Gas Chromatograph/Mass Spectrometer 4	Agilent	6890 GC/5973MSD	svcomph	4	GC CN10403067 MS US35120308	SVOC
Gas Chromatograph/Mass Spectrometer 7	Agilent	6890 GC/5973MSD	svcompm	7	GC ----- MS US03940745	SVOC

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis						
<i>This table is subject to revision without notice</i>						
Item	Manufacturer	Model	Instrument Name	#	Serial #	Location
Gas Chromatograph/Mass Spectrometer 9	Agilent	6890 GC/5973MSD	svcompx	9	GC CN10344042 MS US33220158	SVOC
Gas Chromatograph/Mass Spectrometer 10	Agilent	6890 GC/5973MSD	svcompy	10	GC CN10340045 MS US33220183	SVOC
Gas Chromatograph/Mass Spectrometer 11	Agilent	6890 GC/5975MSD		11	GC CN10509031 MS US60532657	SVOC
Gas Chromatograph/Mass Spectrometer 12	Agilent	7890 GC/5975MSD	svcompai	12	GC CN10728074/ MS 12-0706-1325	SVOC
Gas Chromatograph/Mass Spectrometer 13	Agilent	7890 GC/5975MSD	svcompak	13	GC CN10301081/ MS US10313621	SVOC
Gas Chromatograph/Mass Spectrometer 14	Agilent	7890 GC/5975MSD	Svcompal	14	GC: CN11031022 MS: US11093726	SVOC
Gas Chromatograph/Mass Spectrometer 15	Agilent	7890 GC/5975MSD	Svcompam	15	GC: CN10301081 MS: US10313621	SVOC
Gas Chromatograph/Mass Spectrometer 16	Agilent	7890 GC/5975MSD	Svcompan	16	GC: CN10301152 MS: US10313616	SVOC
Gas Chromatograph/Mass Spectrometer 17	Agilent	7890 GC/5975MSD	Svcompao	17	GC: CN11191064 MS: US11363807	SVOC
Gas Chromatograph/Mass Spectrometer 18	Agilent	7890 GC/5975MSD	Svcompap	18	GC: CN11401093 MS: US11403903	SVOC
Gas Chromatograph/Mass Spectrometer 19	Agilent	7890 GC/5975MSD	Svcompaq	19	GC: CN11391051 MS: US11383838	SVOC
Gas Chromatograph/Mass Spectrometer 20	Agilent	7890 GC/5975MSD	Svcompaw	20	GC: CN12031161 MS: US11503941	SVOC
Gas Chromatograph/Mass Spectrometer 21	Agilent	7890 GC/5975MSD	Svcompax	21	GC: CN12031160 MS: US11513903	SVOC
Gas Chromatograph/Mass Spectrometer 22	Agilent	7890 GC/5975MSD	Svcompay	22	GC: CN11521157 MS: US12023909	SVOC
Gas Chromatograph/Mass Spectrometer 23	Agilent	7890 GC/5975MSD	Svcompaz	23	GC: CN12031114 MS: US11433926	SVOC
Gas Chromatograph/Mass Spectrometer 24	Agilent	7890 GC/5977MSD	Svcompbb	24	GC: CN10906031 MS: US11343905	SVOC
High Performance Liquid Chromatography	Agilent	1100 Series DAD/FLD	hplc1	1	DAD de01608402 FLD de23904489	SVOC
High Performance Liquid Chromatography	Agilent	1100 Series DAD/FLD	hplc2	2	DAD de30518420 FLD	SVOC
High Performance Liquid Chromatography (HPLC3)	Agilent	1100 Series DAD	hplc3	3	DAD us64400711	SVOC
High Performance Liquid Chromatography (HPLC4)	Agilent	1100 Series DAD/FLD	hplc4	4	DAD de43623013	SVOC
Analytical Balance	Mettler Toledo	PB1502-S		1	1126193668	Ext. Lab
Analytical Balance	Mettler Toledo	MS1602S		2	B243464732	Ext. Lab
Analytical Balance	Mettler Toledo	MS1602S		3	B115130112	Ext. Lab
Analytical Balance	Ohaus	ARA520		3	1202120618	Ext. Lab
Analytical Balance	Ohaus	ARA520		4	1202120814	Ext. Lab
Analytical Balance	Ohaus	Scout Pro			7132101108	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	1	2302	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	2	2304	Ext. Lab



<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis</b>						
<i>This table is subject to revision without notice</i>						
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Instrument Name</i>	<i>#</i>	<i>Serial #</i>	<i>Location</i>
Automatic Concentrators	Buchi	Syncore	Buchi	3	2303	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	4	0400000940	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	5	406583020005	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	6	1469	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	7	1461	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	8	417004020002	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	9	416870050003	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	10	1466	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	11	1463	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	12	1462	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	13	1468	Ext. Lab
Capping station	Horizon	MARS X			snxc2225	Ext. Lab
Capping station	Horizon	MARS X			snxc2215	Ext. Lab
Centrifuge	Sorvall	ST-40		2	2224	Ext. Lab
Centrifuge	Sorvall	ST-40		3	2225	Ext. Lab
Centrifuge	Sorvall	ST-40		5	2227	Ext. Lab
Concentration Chiller	Lauda	UC0300			64593	Ext. Lab
Concentration Chiller	Lauda	WKL 3200			2039	Ext. Lab
Furnace	Thermo Scientific				1882	Ext. Lab
Oven	Fisher	6556			166	Ext. Lab
Oven	VWR	1305U			0520	Ext. Lab
HAA Shaker	Eberbach				2159	Ext. Lab
RV shaker	Eberbach	F6010.00			041242	Ext. Lab
RV shaker	Eberbach	F6010.00			041250	Ext. Lab
LVI Shaker	Eberbach	6010-04			1834	Ext. Lab
HAA water Bath	Thermo Scientific	280 series			2033602-102	Ext. Lab
High Intensity Ultrasonic Processor	Misonix			1	2193	Ext. Lab
High Intensity Ultrasonic Processor	Misonix			2	1382	Ext. Lab
High Intensity Ultrasonic Processor	Misonix			3	1888	Ext. Lab
High Intensity Ultrasonic Processor	Misonix			4	1381	Ext. Lab
Microwave	CEM	MARS 6		3	2296	Ext. Lab
Microwave	CEM	MARS 6			MJ2518	Ext. Lab
OG concentrator	Horizon	SpeedVap III		1	1534	Ext. Lab
OG concentrator	Horizon	SpeedVap III		2	SN04-2020	Ext. Lab
OG concentrator	Horizon	SpeedVap III		3	2186	Ext. Lab
OG SPE extractor	Horizon	SPE-DEX 3000		1	2222	Ext. Lab
OG SPE extractor	Horizon	SPE-DEX 3000		2	2223	Ext. Lab
OG SPE extractor	Horizon	SPE-DEX 3000		3	2221	Ext. Lab

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis						
<i>This table is subject to revision without notice</i>						
Item	Manufacturer	Model	Instrument Name	#	Serial #	Location
OG SPE extractor	Horizon	SPE-DEX 3000		4	2220	Ext. Lab
OG SPE Controllers	Horizon	1000/3000XL		1	2125	Ext. Lab
OG SPE Controllers	Horizon	1000/3000XL		2	2659	Ext. Lab
OG SPE Controllers	Horizon	1000/3000XL		3	2127	Ext. Lab
OG SPE Controllers	Horizon	1000/3000XL		4	2128	Ext. Lab
Separatory funnel rotators	ATR				1514	Ext. Lab
Separatory funnel rotators	ATR				1515	Ext. Lab
Separatory funnel rotators	ATR				1516	Ext. Lab
Separatory funnel rotators	ATR				2055	Ext. Lab
Separatory funnel rotators	ATR				2056	Ext. Lab
Separatory funnel rotators	ATR				2057	Ext. Lab
Speed Vap	FMS				2471	Ext. Lab
Water Bath Sonicator	Branson	8510			RPA040384175E	Ext. Lab
Vacuum Pump	Gast				0908605639	Ext. Lab
Vacuum Pump	Gast				0913008139	Ext. Lab
Vacuum Pump	Gast			3	0311000841	Ext. Lab

## 8.2 E EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
Analytical Balances	•Check with Class "I" weights	Daily-tolerance $\pm 0.1\%$
Analytical Balances	•Service/Calibration (semi-annual contract maintenance and calibration check)	Semi-annually
Refrigerators & Incubators	•Maintenance service	As needed determined by daily temperature performance checks
Gas Chromatograph Detectors: ECD	•Bake off or Replace •Perform wipe leakage test	GC/detector maintenance is routinely completed as needed for each instrument. Analysts are responsible for performing and documenting maintenance on each component of the instrumentation based on daily performance of the instrument and its ability to meet certain method requirements. Senior analyst team is available to help with major maintenance issues.
Gas Chromatograph Detectors: FID	•Change Quartz jet; clean; replace flame tip	
Gas Chromatograph/Mass Spectrometer	•Autotune Report	
Gas Chromatograph/Mass Spectrometer	•Clean ion source	
Gas Chromatograph/Mass Spectrometer	•Replace vacuum pump oil	
Gas Chromatographs/Mass Spectrometer & Gas Chromatographs	•Replace septa and liner	
Gas Chromatographs/Mass Spectrometer & Gas Chromatographs	•Replace column	
High Intensity Ultrasonic Processor - Misonix	•Check tuning criteria	Daily with use
Infrared Spectrophotometer - Foxboro Miran 1A	•Optics alignment or replacement	As needed when response begins to deteriorate

### 8.3 S TANDARDS AND REAGENTS

<b>Table 8.3A: Standard stock sources, description and calibration information.</b>					
<i>This table is subject to revision without notice</i>					
<b>Method</b>	<b>Vendor*</b>	<b>Description</b>	<b>Calibration</b>	<b>Storage Req.</b>	<b>Expiration</b>
8310	Ultra	Aromatic Hydrocarbon	Primary	4° ± 2°C	6 months
	NSI	8310/610 Spike	Second Source	4° ± 2°C	6 months
DRO	NSI	DRO #2 Cal Mix	Primary	-10°C to -20°C	6 months
	NSI	DRO #2 Spike	Second Source	-10°C to -20°C	6 months
EPH TN DRO	NSI	TN-EPH Calibration Mix	Primary	-10°C to -20°C	6 months
	NSI	EPH-TN Spike	Second Source	-10°C to -20°C	6 months
RRO	NSI	30W Oil	Primary	-10°C to -20°C	6 months
PCB	Accustd	Aroclor PCB Kit	Primary	4° ± 2°C	6 months
	NSI	1260 Spike	Second Source	4° ± 2°C	6 months
Chlordane	Restek	Chlordane Mix	Primary	4° ± 2°C	6 months
Toxaphene	Restek	Toxaphene	Primary	4° ± 2°C	6 months
Pesticides	Ultra	Pest Mix	Primary	4° ± 2°C	6 months
	NSI	Pest Spike Mix	Second Source	4° ± 2°C	6 months
Herbicides	NSI	Custom Herbicide Mis	Primary	4° ± 2°C	6 months
	NSI	Herb Spike Mix	Second Source	4° ± 2°C	6 months
8141 OP Pest	Ultra/NSI	OP Cal Mix A, B	Primary	4° ± 2°C	6 months
	NSI	OP Spike Mix A, B	Second Source	4° ± 2°C	6 months
507 NP Pest	Ultra/NSI	507 Cal Mix	Primary	4° ± 2°C	2 months
	NSI	NP Pest Spike	Second Source	4° ± 2°C	2 months
THAA	Ultra/Accustd	HAA Cal Mix	Primary	-10°C to -20°C	6 months
	Accustd/NSI	HAA Spike	Second Source	-10°C to -20°C	6 months
8270	Ultra	Custom Std Mega Mix	Primary	4° ± 2°C	6 months
	Restek	Spike Mix	Second Source	4° ± 2°C	6 months
8330	Restek	Mix1, Mix2, PETN	Primary	4° ± 2°C	6 months
	Ultra, Chemservice	Mix1, Mix2, PETN	Second Source	4° ± 2°C	6 months
8011, 504.1	Accustd	504.1 Cal Mix	Primary	4° ± 2°C	1 month
	NSI	Spike Mix	Second Source	4° ± 2°C	1 month
Sulfolane, 8270C	Sigma Aldrich	Calibration Mix	Primary	4° ± 2°C	6 months
	Restek	Spike Mix	Second source	4° ± 2°C	6 months
Glycol, 8015	Chemservice	Calibration Mix	Primary	4° ± 2°C	6 months
	Chemservice	Spike Mix	Second source	4° ± 2°C	6 months

\*Equivalent Providers may be utilized.

**TABLE 8.3B: Working Standard Concentrations**

*This table is subject to revision without notice*

Organic Compounds	Method #	Standard Concentrations	Storage Requirements	Expiration
Semi-Volatiles	625, SM6410B 20 <sup>th</sup> , 8270C/D	1,2,4,8,12,16,20,30,40,50,80 (low level and regular)	4° ± 2°C	6 months
Semi-Volatiles: RV/LVI/NC SS	625, SM6410B 20 <sup>th</sup> , 8270C/D	10,20,50,100,200,500,1000,2000 ug/L	4° ± 2°C	6 months
PCBs: 1L/RV SS	608, SM6431B 20 <sup>th</sup> , 8082	2.0,4.0,5.0,10,20,50 µg/L	4° ± 2°C	6 months
Pesticides: 1L/RV/SS	608, SM 6630C, 8081A,	0.5,1.0,2.0,5.0,10,15,20 µg/L	4° ± 2°C	6 months
Chlordane and/or Toxaphene 1L/RV/SS	608, SM 6630C, 8081A,	10,20,50,100,150,200 µg/L	4° ± 2°C	6 months
Sulfolane	8270C/D	4,8,10,20,50,100,200,500 ug/L	4° ± 2°C	6 months
PCB Arochlors 1221, 1232, 1242, 1248, 1254	8082	10 ug/L	4° ± 2°C	6 months
Herbicides	8151A, SM6640C 20th	0.02, 0.05, 0.1, 0.2, 0.5, 1.0 mg/L	4° ± 2°C	6 months
OP and NP Pesticides	507 by dual-NPD, 1657A, 8141A by dual- FPD	0.2,1.0, 2.0, 5.0, 10.0, 15.0, 20.0 ug/L	4° ± 2°C	6 months
PAHs	8310, 610, SM6440B	0.04, 0.20,1.0,5.0,8.0,20.0,30.0, 40.0 ug/L	4° ± 2°C	6 months
PAHs: 1L/RV/LVI/ SS	8270C/D	4.0,20,40,100,160,400,600,800 ug/L	4° ± 2°C	6 months
	SIM	1.0,5.0,10,20,40,80,200 ug/L		
Nitroaromatics & Nitramines	8330	.05, 0.1, 0.25, 0.5, 2.0, 5.0, 10.0, 25.0 mg/L	NA*	NA*
EPHTN	EPH TN	10000, 6000, 4000, 2000, 1000, 400, 200, 100 mg/L	NA*	NA*
DRO	OA2 , 8015Mod, LA TPH D, LA TPH O, OHIO DRO	10000, 5000, 3000, 2000, 1000, 400, 200, 100 mg/L	NA*	NA*
Diesel/M.O: RV/LVI	EPH TN OA2 , 8015Mod, LA TPH D, LA TPH O, OHIO DRO	2.0,4.0,8.0,20,40,80,100,200 mg/L	NA*	NA*
DRO	DRO/CA LUFT/CO	2.0,4.0,10,20,40,60,100,200 mg/L	NA*	NA*
DROMO: LVI	MO DRO/PAH by 8270	5.0,10,20,40,80,120,160,200 mg/L	4° ± 2°C	6 months
PAHMO: LVI		4.0,20,40,100,160,400,600,800 ug/L		

**TABLE 8.3B: Working Standard Concentrations**

*This table is subject to revision without notice*

Organic Compounds	Method #	Standard Concentrations	Storage Requirements	Expiration
MADEP EPH	MADEP EPH	Aromatics C11-C22: 17, 85, 425, 850, 1700, 3400, 6800 mg/L Aliphatic C9 - C18: 6, 30, 150, 300, 600, 1200, 2400 mg/L Aliphatic C19 - C36: 8, 40, 200, 800, 1600, 3200 mg/L	NA*	NA*
EDB, DBCP, TCP	8011, 504.1	0.01, 0.02, 0.05, 0.10, 0.25, 0.5	NA*	NA*
THAAs	552.2	1, 2, 4, 10, 20, 30, 40, 50 ug/L	NA*	NA*
FL PRO	FL PRO	85, 850, 2550, 4250, 5950, 8500 mg/L	NA*	NA*
FL PRO RV	FL PRO	1.7, 3.4, 6.8, 13.6, 34, 85, 170 mg/L	NA*	NA-
Glycols	8015B/C/D - Modified	1.5, 7.5, 15, 30, 45, 60 ppm	NA*	NA*
TX TPH	TX1005	Individual Ranges- 4.5, 10, 25, 50, 125, 250, 500, 1250, 2500 ppm. Total Range- 9.0, 20, 50, 100, 250, 500, 1000, 2500, 5000 ppm.	NA*	NA*

\* indicates solutions are prepared fresh daily as needed.

## 8.4 I INSTRUMENT CALIBRATION

### 608/8081A or B/SM6630C - Chlorinated Pesticides – SOP Number 330344

The gas chromatograph is calibrated using either the internal or external standard calibration model. A standard curve is prepared using a minimum of three concentration levels for each compound of interest for method 608. A minimum of five concentration levels is necessary for methods 8081A/B and SM6630C. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration or ISTD response for each compound and calibration/response factors are calculated. If performing analysis by method 608 and the response factors of the initial calibration are < 10 % RSD for method 608 and 20% RSD for methods 8081A/B and 6630C over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990.

During the analytical sequence, the stability of the initial calibration curve is verified, following every 20<sup>th</sup> sample for external calibration and every 12 hours if monitoring

ISTD, by the analysis of a continuing calibration verification (CCV) standard. The CCV must recover within 15% of the expected concentration for each analyte.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of initial calibration verification standard (ICV). If the response for any analyte in this check varies from the predicted response by more than  $\pm 15\%$ , the analysis must be repeated using fresh standard. If the standard still does not meet the acceptance criteria, a new initial calibration curve must be generated.

An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should recover within  $\pm 20\%$  of the expected concentration for each analyte. When analyte responses in field samples exceed the calibration range, the sample is diluted and re-analyzed.

Degradation of DDT and Endrin are also verified at least every 12hr window. Breakdown should recover less 20% of the total injection.

#### **507 - Nitrogen/Phosphorus Pesticides - SOP Number 330348**

The gas chromatograph is calibrated using the external standard procedure. A standard curve is prepared using a minimum of three concentration levels for each compound of interest for method 507. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are  $\leq 20\%$  RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990.

During the analytical sequence the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of a continuing calibration verification (CCV) standard. The CCV must recover within 20% of the expected concentration for each analyte.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies by more than  $\pm 20\%$  from the initial calibration, the analysis must be repeated using fresh standard. If the standard still does not meet the criteria, a new initial calibration curve must be generated.

A Quality Control Sample (QCS) is analyzed at a minimum quarterly to verify calibration standards.

### **552.2 - HAA - SOP Number 330319**

The gas chromatograph is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of five concentration levels for each compound of interest. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are  $\leq 20\%$  RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990.

During the analytical sequence the stability of the initial calibration is verified, following every 10<sup>th</sup> sample and at the end of the sequence, by the analysis of a continuing calibration verification (CCV) standard. The response of the analytes in the CCV must not vary more than 30% from the initial calibration.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies by more than  $\pm 30\%$  from the initial calibration, the analysis must be repeated using fresh standard. If the standard still does not meet the criteria, a new initial calibration curve must be analyzed.

A Quality Control Sample (QCS) is analyzed at a minimum quarterly to verify calibration standards.

### **8151A, SM6640B – Herbicides - SOP Number 330320**

The gas chromatograph is calibrated using the external standard procedure. A standard curve is prepared using a minimum of five concentration levels for each analyte of interest. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are  $\leq 20\%$  RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990.

During the analytical sequence, the stability of the initial calibration is verified following every 10<sup>th</sup> sample and at the end of the sequence by the analysis of a continuing calibration verification (CCV) standard. The CCV must recovery within 15% of the expected concentration for each analyte for method 8151A and within 20% for method 6640C. The value of the CCV can exceed the criteria for a single compound provided that all samples in the analytical batch are BDL (below detection limit). The concentration of the continuing check standard must be routinely varied to verify the entire calibration range.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies from the predicted response by more than  $\pm 15\%$ , the analysis must be repeated using fresh standard. If the standard still does not meet the criteria, a new initial calibration curve must be generated.

An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should recover within  $\pm 20\%$  of the expected concentration for each analyte. When sample responses exceed the calibration range, the sample is diluted and re-analyzed.

#### **8141A, 1657A – Organophosphorus Pesticides - SOP Number 330318**

The gas chromatograph is calibrated using either the internal or external standard calibration model. A minimum of five concentration levels is necessary for methods 8141A and 1657A. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration or ISTD response for each compound and calibration/response factors are calculated. If the response factors of the initial calibration are  $\leq 20\%$  RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990.

During the analytical sequence, the stability of the initial calibration is verified following every 20<sup>th</sup> sample by the analysis of a continuing calibration verification (CCV) standard for external calibration or at the beginning of every 12hrs for ISTD calibration. The CCV must recovery within 15% of the expected concentration for each analyte. The concentration of the continuing check standard must be routinely varied to verify the entire calibration range.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies from the predicted



response by more than  $\pm 15\%$ , the analysis must be repeated using fresh standard. If the standard still does not meet the criteria, a new initial calibration curve must be generated. An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should recover within  $\pm 20\%$  of the expected concentration for each analyte. When sample responses exceed the calibration range, the sample is diluted and re-analyzed.

**625, 8270C or D, SM6410B - Base/Neutrals/Acids by GC/MS: Semivolatile Organics – SOP Number 330345**

Detector mass calibration is performed using the autotune function of the GC/MS analytical system and PFTBA (Perfluorotributylamine). Following verification of the appropriate masses, the instrument sensitivity is verified by injecting a tuning solution containing decafluorotriphenylphosphine (DFTPP), benzidine, pentachlorophenol and DDT. The DFTPP must meet the ion abundance criteria specified by the EPA published method. Benzidine and pentachlorophenol are reviewed for tailing and DDT is reviewed for breakdown to DDE and DDD. Successful tuning must occur every 12 hours for method 8270C/D and every 24 hours for method 625, except where noted in the determinative SOP.

Following successful tuning, the GC/MS is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of three standards for method 625 and five standards for method 8270C/D and SM6410B. The calibration standards are tabulated according to peak height or area against concentration and the concentrations and responses of the internal standard analytes. The results are used to determine a response factor for each analyte in each standard injected. A calibration curve is constructed and is determined to be acceptable if each analyte meets the criteria specified in the determinative method. When this condition is met, linearity through the origin can be assumed and the average RF can be used in place of a calibration curve. Initial calibration that does not meet these requirements will not be accepted and recalibration must be performed. Linear regression can be used for target compounds exceeding the 15% criteria, providing that the correlation coefficient is 0.990 or better. USACE projects must meet a correlation coefficient of 0.995 or better. The initial calibration range must represent the typical environmental sample and include the RL as the lowest calibration point. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range.

A second source calibration verification standard is analyzed after each calibration and should recover within 20% for all CCC compounds and within 50% for other analytes of interest for 8270C. All analytes must recover  $\pm 30\%$  for 8270D. Following successful calibration, the analysis of field and QC samples may begin. Analysis may be performed only during the timeframe of a valid tuning cycle (12 hours for 8270C/D and 24 hours for 625). Following the expiration of the tuning clock, the instrument must be retuned and either re-calibrated or existing calibration may be re-verified.

For 8270C/D analyses, daily calibration verification includes successful demonstration of DFTPP sensitivity and the injection of a mid-level CCV standard containing all the target analytes of interest. The DFTPP tune must meet the ion abundance criteria specified within the published method. Each internal standard in the CCV must recover between -50% to +100%, when compared to the same internal standard compound in the mid-point standard of the initial calibration curve. Additionally, if the retention time of an internal standard changes by more than 30 seconds from the retention time of the same internal standard in the mid-level standard of the most recent initial calibration, the system must be evaluated, corrected, and possibly re-calibrated.

For 625 analyses, daily calibration verification is accomplished by a successful demonstration of DFTPP sensitivity and the injection of a mid-level CCV standard containing all the target analytes of interest. The DFTPP tune must meet the same ion abundance criteria as the 8270C analysis and the CCV standard must recover within 20 % of predicted response for all analytes of interest.

**8310, 610, SM6640B - PAHs by HPLC - SOP Number 330322**

610: A standard curve is prepared using a minimum of three concentration levels for each compound of interest. If the response factors are < 10 % RSD over the working range, the average RF can be used for calculations

8310 & SM6640B: Perform calibration using a minimum of 5 points. If the response factors are < 20 % RSD over the working range, the average RF can be used for calculations or linear regression may be used providing that the correlation coefficient for each analyte of interest is 0.990 or better. USACE projects must meet a correlation coefficient of 0.995 or better. The regression line must never be forced through the origin.

The initial calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. Alternatively, the results can be used to plot a calibration curve of response ratios (Area/Ref. Area) vs (Amt./Ref Amt.). The calibration range must represent the typical environmental sample and include the RL as the lowest calibration point. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range. A second source calibration verification standard is analyzed after each calibration and should meet criteria of  $\pm 20\%$ .

A continuing calibration verification (CCV) must be run at the beginning of each run and every 10 samples thereafter. The continuing calibration standard is prepared from the same source as the calibration curve and must perform within  $\pm 15\%$  of the actual value. The CCV must represent the midpoint of the calibration range.

**8330A/B/C – Nitroaromatics/Nitrosamines - SOP Number 330323**

A standard curve is prepared using a minimum of five concentration levels for each compound of interest. Experience indicates that a linear calibration curve with zero intercept is appropriate for each analyte. Therefore, a response factor for each analyte can be taken as the slope of the best-fit regression line. The correlation coefficient for each analyte of interest is 0.990 or better. The calibration range must represent the typical environmental sample and include the RL as the lowest calibration point. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range. A second source calibration verification standard is analyzed after each calibration and should meet the criteria of  $\pm 20\%$ .

Daily calibration is accomplished through the analysis of midpoint calibration standards, at a minimum, at the beginning of the day, and singly after the last sample of the day (assuming a sample group of 10 samples or less). Obtain the response factor for each analyte from the mean peak heights or peak areas and compare it with the response factor obtained for the initial calibration. The mean response factor for the daily calibration must agree within  $\pm 20\%$  of the response factor of the initial calibration. If this requirement is not met, a new initial calibration must be obtained.

**8015B/C/D or State Specific Method - DRO/RRO - Various SOPs**

Certain state accreditation/registration programs may have specific requirements for calibration and analysis that must be met. Those requirements supersede the general guidance provided in this section and are addressed in the determinative SOP. Generally, for 8015B/C/D analysis, the gas chromatograph is calibrated using the external standard procedure. A standard curve is prepared using a minimum of five concentration levels for each analyte of interest. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are  $\leq 20\%$  RSD over the calibration range, or per state method, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990. USACE projects must meet a correlation coefficient of 0.995 or better.

During the analytical sequence, the stability of the initial calibration is verified following every 10<sup>th</sup> or 20<sup>th</sup> sample depending on method and at the end of the sequence by the analysis of a continuing calibration verification (CCV) standard. Typically, the CCV must recovery within 15% of the expected concentration for each analyte for method 8015B/C/D; however state specific limits for the CCV may vary. See the specific SOP or published method for more guidance. The concentration of the continuing check standard must be routinely varied to verify the entire calibration range.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies from the predicted response by more than  $\pm 15\%$  of the expected concentration for each analyte for method 8015B/C/D or more than state specified limits, the analysis must be repeated using fresh standard. If the standard still does not meet the criteria, a new initial calibration curve must be generated.

An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should meet criteria of  $\pm 20\%$  of the expected concentration for each analyte. When sample responses exceed the range of the standard curve, the sample is diluted to a concentration suspected to be within the calibration range and re-analyzed.

## 8.5 A ACCEPTANCE/REJECTION OF CALIBRATION

### Organic Chemistry

The initial calibration curve is compared with previous curves for the same analyte. All new standard curves are immediately checked with a secondary source or laboratory control standard prepared from a separate source than those used for calibration. All curves are visually reviewed to ensure that acceptable correlation represents linearity. Calibration curves may be rejected for nonlinearity, abnormal sensitivity, or poor response of the laboratory control standard.

Continuing calibration verification is performed on each day that initial calibration is not performed and following every 10<sup>th</sup> or 20<sup>th</sup> sample. If a check standard does not perform within established criteria then the instrument undergoes an evaluation to determine the cause. Once the issue is corrected, all samples between the last in control standard and the first out of control check are re-analyzed.

**TABLE 8.5: INSTRUMENT CALIBRATION**

Instrument (Analysis)	Calibration Type	Minimum Number of Standards	Type of Curve	Acceptance/Rejection Criteria	Frequency
Gas Chromatography  (Pest/PCB, Herbicides, Organophos/ Organonitrogen Pesticides)	Initial	3 (600 series methods) - 5 (other) cal.stds	Avg. RF or Linear	8081A, 8151A, 6640C, 8141A, 657A: Must be $\leq 20\%$ RSD 608 - $\leq 10\%$ RSD	As needed
	Second Source	1 Second Source		+/- 20% of true value	With each calibration
	Daily / Continuing	OPPEST/HER B1/10 P/PCB 1/20		Must be within 15% of the initial calibration curve, 20% for 6640C.	Beginning, every 20 samples and ending for external cal.
	Daily / Continuing	OPPEST/HER B1/10 P/PCB 1/20		Must be within 15% of the initial calibration curve, 20% for 6640C.	Every 12hrs samples for internal cal

**TABLE 8.5: INSTRUMENT CALIBRATION**

Instrument (Analysis)	Calibration Type	Minimum Number of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
HPLC  (PAH and Explosive)	Initial	3 (600 series methods) 5 (other) cal.stds	Avg. RF or Linear	8310, 8330: Must be $\leq 20\%$ RSD 610 - $\leq 10\%$ RSD	As needed
	Second Source	1 Second Source		+/- 20% of true value	With each calibration
	Daily / Continuing	1/10		Must be within 15% of the initial calibration curve.	Beginning, every 10 and ending.
GC/MS Semi-volatiles 8270C/D	Initial	At least 5 cal. stds	Avg. RF or Linear	8270C - Must be $\leq 15\%$ RSD, CCCs must be $\leq 30\%$ RSD, Linear regression: 0.990 per method or 0.995 for USACE	As needed
	Second Source	1 Second Source		8270D - Must be $\leq 20\%$ RSD for target analytes, Linear regression: 0.990 per method or 0.995 for USACE	With each calibration
	Daily / Continuing	Tune & CCV		8270C: Should recover within 20% for all CCC compounds and within 50% for other analytes of interest, with the exception of analytes known to perform poorly 8270D: Should recover w/in 30% for all  Must pass established method criteria. See SOP.	Every 12 hours per method
GC/MS Semi-volatiles 625	Initial	3 cal.stds	Avg. RF or Linear	625 - $\leq 35\%$ RSD all compounds	As needed
	Second Source	1 Second Source		Should recover within 20% for all CCC compounds and within 50% for other analytes of interest, with the exception of analytes known to perform poorly	With each calibration
	Daily / Continuing	Tune & CCV every 24 hours		Must pass established method tuning criteria; 625: CCV must be $\leq 20\%$ difference for all compounds,	Every 24 hours
HAA 552.2	Initial	5 cal.stds	Avg. RF or Linear	$\leq 30\%$ RSD all compounds	As needed
	Second Source(QCS)	1 Second Source		$\pm 30\%$ of true value	Extracted with each batch
	Daily / Continuing	1/10		CCV must be $\leq 30\%$ difference for all compounds,	Beginning, every 10 and ending
Pesticides 507	Initial	5 cal.stds	Avg. RF or Linear	$\leq 20\%$ RSD all compounds	As needed
	Second Source(QCS)	1 Second Source		$\pm 20\%$ of true value	Extracted with each batch
	Daily / Continuing	1/10		CCV must be $\leq 20\%$ difference for all compounds,	Beginning, every 10 and ending

TABLE 8.5: INSTRUMENT CALIBRATION					
Instrument (Analysis)	Calibration Type	Minimum Number of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
DRO –8015, State Programs* * Or per state requirement	Initial	5 cal.stds	Avg. RF or Linear	8015B/C/D - $\leq 20\%$ RSD all compounds	As needed
	Second Source	1 Second Source		$\pm 20\%$ of true value	With each calibration
	Daily / Continuing	1/10		CCV must be $\leq 15\%$ difference for all compounds,	Beginning, every 10/20 and ending

## 9.0 LABORATORY PRACTICES

### 9.1 REAGENT GRADE WATER

Reagent grade water is obtained from an Evoqua resin with Aquafine UV system.

### 9.2 GLASSWARE WASHING AND STERILIZATION PROCEDURES

Organic laboratory glassware is washed in a non-phosphate detergent and warm tap water. Before washing, all writing and large deposits of grease are removed with acetone. Glassware is then rinsed with: tap water, "No Chromix" solution, tap water, and deionized (DI) water. It is then solvent rinsed in the following order: acetone, and then methylene chloride. Glassware is stored in designated drawers or on shelves, inverted if possible. All glassware is rinsed with the required solvent for the particular extraction protocol prior to use.

## 10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOPs associated with the semi-volatile laboratory can be found in the following table:

**TABLE 10.1: SEMI-VOLATILE DEPARTMENT SOPS**

*This table is subject to revision without notice*

SOP #	Title
<b>Preparatory SOPs</b>	
330702B	RV Separatory Funnel Liquid-Liquid Extraction 3510C
330702A	Separatory Funnel Liquid-Liquid Extraction 3510C MN
330702	Separatory Funnel Liquid-Liquid Extraction 3510C
330705	Ultrasonic Extraction 3550B
330707	Microwave Extraction (3546)
330708	Buchi Syncore Concentration System
330709	Microextraction Procedure (3511)
330754	3580A Waste Dilution for SVOC's
330755	PCB in Oil Waste Dilution
<b>Extract Cleanup SOPs</b>	
330739	3630C Silica Gel Cleanup

SOP #	Title
330740	3665A Acid Clean up
330741	3660C Sulfur Clean up
330742	3620B Florisil Clean up
<b>Semi-Volatiles Analysis SOPs</b>	
330770A	TPH/O&G- Soxhlet extraction using Hexane
330771A	n-Hexane Oil and Grease Extraction by SPE for South Carolina
330771	n-Hexane Oil and Grease Extraction by SPE
340330A	Total Recoverable Oil and Grease Shake Extraction (9070A, SM5520B/F)
330317	Sulfolane (Modified EPA Method 8270C/D)
330318	8141 Organophosphorus Pesticides
330319	THAA's
330320	Chlorinated Herbicides by Gas Chromatography (Method 8151A)
330322	8310 PAH's by HPLC
330323	8330 Explosives by HPLC
330342	Carbamate Pesticides by HPLC (EPA Method 632)
330343	8082 PCB's
330344	Pesticides and PCBS by Gas Chromatography (608 and 8081A)
330345	Semi-volatile Organics by GC/MS using Capillary Column
330346	8011/504.1 EDB in Drinking Water by GC ECD
330346OH	8011 EDB in Drinking Water by GC ECD
330348	507 NP Pesticides in Drinking Water by GC NPD
330350A	Diesel Range Organics/Total Petroleum Hydrocarbons (Diesel) By Gas Chromatography
330352	TN - Extractable Petroleum Hydrocarbons / KY- Diesel Range Organics
330353	MA Extractable Petroleum Hydrocarbons
330355	Florida Pro and CT ETPH
330356	TXTPH 1005/1006
330358	OA2 & NWTPHDx
330359	AK102/AK103
330360	DROWM
330361	Glycols by GC/FID (8015)

## 11.0 QUALITY CONTROL CHECKS

**NOTE:** For specific guidance on each determinative method, including required quality control and specific state requirements/modifications, refer to the relevant laboratory standard operating procedure(s).

- 11.1 ESC participates in proficiency testing (PT's) in support of various laboratory accreditations/recognitions. Environmental samples are purchased from Environmental Resource Associates (ERA). The WS, WP and solid matrix studies are completed every 6 months. Proficiency testing samples are received and analyzed by method according to the vendor's instructions and according to the applicable analytical SOP.
- 11.2 Initial Demonstrations of Capability (IDOCs) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability (CDOCs) must be updated at least annually. The associated data is filed within the department and available for review.

11.3 Matrix Spike and Matrix Spike Duplicates are performed on each batch of samples analyzed depending on analytical method requested provided that sufficient volume is provided by the client.

11.4 A Laboratory Control Sample (LCS) and LCS Duplicate are analyzed one per batch of samples.

11.5 A method preparation blank is performed per batch of samples processed. If the acceptance criteria as listed in the determinative SOP is exceeded, the laboratory shall evaluate whether re-processing of the samples is necessary, based on the following criteria:

- The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch or
- The blank contamination is greater than 1/10 of the specified regulatory limit. The concentrations of common laboratory contaminants shall not exceed the reporting limit.

Any samples associated with a blank that fail these criteria shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in non-detected results for the failing analytes.

## 12.0 DATA REDUCTION, VALIDATION AND REPORTING

### 12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in SOP #030201, *Data Handling and Reporting*. A secondary review of the data package is performed according to ESC SOP #030227, *Data Review*. The reviewer verifies that the analysis has been performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required flags on test reports, etc.)

**TABLE 12.1 Data Reduction Formulas**

PARAMETER	FORMULA
GC and HPLC	$\frac{\text{response of sample analyte } \{area\} \times \text{final extract volume } \{mL\} \times \text{dilution}}{\text{response factor } \{area/(mg/mL)\} \times \text{initial extract volume-mass } \{mL \text{ or } g\}}$ <p style="text-align: center;"><i>Calculations performed by HP Enviroquant Software</i></p>
GC/MS	$\frac{\text{response of analyte } \{area\} \times \text{extract volume } \{mL\} \times \text{dilution} \times \text{int. std amt. } \{area\}}{\text{response factor } \{area/(mg/mL)\} \times \text{initial volume-mass } \{mL \text{ or } g\} \times \text{int. std cal. } \{area\}}$ <p style="text-align: center;"><i>Calculations performed by HP Enviroquant Software</i></p>



## 12.2 V ALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.3 by method for current QC targets and controls and current reporting limits.

**Marginal Excedence** – When a large number of analytes exist in the LCS, it is statistically possible for a few analytes to be outside established control limits while the analytical system remains in control. These excursions must be random in nature and, if not, a review of the control limits or analytical process is necessary.

Upper and lower marginal excedence (ME) limits are established as the mean of at least 20 data points  $\pm$  four times their standard deviations. The number of allowable marginal excedences per event is based on the number of analytes spiked in the LCS.

Allowable Marginal Excedence per Event	
Analytes in LCS:	ME Allowable
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

**Organic Control Limits** - The organic QC targets are statutory in nature; warning and control limits for organic analyses are initially established for groups of compounds based on preliminary method validation data. When additional data becomes available, the QC targets are reviewed. All QC targets are routinely re-evaluated at least annually (and updated, if necessary) against laboratory historical data to insure that the limits continue to reflect realistic, method achievable goals.

## 12.3 R EPORTING

Reporting procedures are documented in *SOP 030201 Data Handling and Reporting*.

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
Pesticides	AZINPHOS-METHYL	8141A, 1657A	GW	64.9-120	20.0	0.001	mg/L
Pesticides	BOLSTAR (SULPROFOS)	8141A, 1657A	GW	65.4-119	20.0	0.001	mg/L
Pesticides	CHLORPYRIFOS	8141A, 1657A	GW	65.3-113	20.0	0.001	mg/L
Pesticides	COUMAPHOS	8141A, 1657A	GW	62.2-121	20.0	0.001	mg/L
Pesticides	DEMETON,-O AND -S	8141A, 1657A	GW	65.9-110	20.0	0.002	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
Pesticides	DIAZINON	8141A, 1657A	GW	62.4-116	20.0	0.001	mg/L
Pesticides	DICHLORVOS	8141A, 1657A	GW	51.0-117	20.0	0.002	mg/L
Pesticides	DIMETHOATE	8141A, 1657A	GW	19.9-109	35.6	0.001	mg/L
Pesticides	DISULFOTON	8141A, 1657A	GW	63.3-113	20.0	0.001	mg/L
Pesticides	EPN	8141A, 1657A	GW	635-119	20.0	0.001	mg/L
Pesticides	ETHOPROP	8141A, 1657A	GW	63.7-113	20.0	0.001	mg/L
Pesticides	ETHYL PARATHION	8141A, 1657A	GW	71.8-112	20.0	0.001	mg/L
Pesticides	FENSULFOTHION	8141A, 1657A	GW	63.4-112	20.0	0.001	mg/L
Pesticides	FENTHION	8141A, 1657A	GW	61.5-114	20.0	0.001	mg/L
Pesticides	MALATHION	8141A, 1657A	GW	68.5-112	20.0	0.001	mg/L
Pesticides	MERPHOS	8141A, 1657A	GW	52.0-115	20.0	0.001	mg/L
Pesticides	METHYL PARATHION	8141A, 1657A	GW	70.6-114	20.0	0.001	mg/L
Pesticides	MEVINPHOS	8141A, 1657A	GW	58.8-111	20.0	0.001	mg/L
Pesticides	NALED	8141A, 1657A	GW	60.7-112	20.0	0.001	mg/L
Pesticides	PHORATE	8141A, 1657A	GW	64.1-113	20.0	0.001	mg/L
Pesticides	RONNEL	8141A, 1657A	GW	63.0-112	20.0	0.001	mg/L
Pesticides	STIROPHOS	8141A, 1657A	GW	65.3-118	20.0	0.001	mg/L
Pesticides	SULFOTEP	8141A, 1657A	GW	64.7-110	20.0	0.001	mg/L
Pesticides	TEPP	8141A, 1657A	GW	34.3-107	31.3	0.020	mg/L
Pesticides	TOKUTHION (PROTHIOFOS)	8141A, 1657A	GW	62.9-118	20.0	0.001	mg/L
Pesticides	TRICHLORONATE	8141A, 1657A	GW	67.1-112	20.0	0.001	mg/L
Pesticides	AZINPHOS-METHYL	8141A	SS	63.3-118	20.0	0.1	mg/Kg
Pesticides	BOLSTAR (SULPROFOS)	8141A	SS	67.3-119	20.0	0.1	mg/Kg
Pesticides	CHLORPYRIFOS	8141A	SS	67.1-117	20.0	0.1	mg/Kg
Pesticides	COUMAPHOS	8141A	SS	64.4-122	20.0	0.1	mg/Kg
Pesticides	DEMETON,-O AND -S	8141A	SS	60.9-111	20.0	0.1	mg/Kg
Pesticides	DIAZINON	8141A	SS	27.8-141	21.7	0.1	mg/Kg
Pesticides	DICHLORVOS	8141A	SS	43.8-117	20.0	0.1	mg/Kg
Pesticides	DIMETHOATE	8141A	SS	43.7-115	23.2	0.1	mg/Kg
Pesticides	DISULFOTON	8141A	SS	67.7-114	20.0	0.1	mg/Kg
Pesticides	EPN	8141A	SS	58.0-120	20.0	0.1	mg/Kg
Pesticides	ETHOPROP	8141A	SS	70.9-114	20.0	0.1	mg/Kg
Pesticides	ETHYL PARATHION	8141A	SS	66.0-115	20.0	0.1	mg/Kg
Pesticides	FENSULFOTHION	8141A	SS	41.1-121	24.9	0.1	mg/Kg
Pesticides	FENTHION	8141A	SS	63.8-119	20.0	0.1	mg/Kg
Pesticides	MALATHION	8141A	SS	66.9-117	20.0	0.1	mg/Kg
Pesticides	MERPHOS	8141A	SS	63.8-117	20.0	0.1	mg/Kg

<b>Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs</b> <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
Pesticides	METHYL PARATHION	8141A	SS	67.6-113	20.0	0.1	mg/Kg
Pesticides	MEVINPHOS	8141A	SS	49.7-120	20.0	0.1	mg/Kg
Pesticides	NALED	8141A	SS	17.4-116	25.9	0.1	mg/Kg
Pesticides	PHORATE	8141A	SS	67.03-114	20.0	0.1	mg/Kg
Pesticides	RONNEL	8141A	SS	66.3-113	20.0	0.1	mg/Kg
Pesticides	STIROPHOS	8141A	SS	66.1-113	20.0	0.1	mg/Kg
Pesticides	SULFOTEP	8141A	SS	67.8-117	20.0	0.1	mg/Kg
Pesticides	TEPP	8141A	SS	0-79	40.0	1.0	mg/Kg
Pesticides	TOKUTHION (PROTHIOFOS)	8141A	SS	67.2-118	20.0	0.1	mg/Kg
Pesticides	TRICHLORONATE	8141A	SS	65.4-121	20.0	0.1	mg/Kg
Pesticides	ALACHLOR	507	DW	70.0-130	25.0	0.0002	mg/L
Pesticides	ATRAZINE	507	DW	70.0-130	25.0	0.0001	mg/L
Pesticides	BUTACHLOR	507	DW	70.0-130	25.0	0.0001	mg/L
Pesticides	METOLACHLOR	507	DW	70.0-130	25.0	0.0002	mg/L
Pesticides	METRIBUZIN	507	DW	70.0-130	25.0	0.0002	mg/L
Pesticides	SIMAZINE	507	DW	70.0-130	25.0	7.00E-05	mg/L
Pesticides	4,4-DDD	608/8081A/B, 6630C	GW, WW	63.0-130	20.0	0.00005	mg/L
Pesticides	4,4-DDE	608/8081A/B, 6630C	GW, WW	59.3-125	20.0	0.00005	mg/L
Pesticides	4,4-DDT	608/8081A/B, 6630C	GW, WW	61.3-130	20.0	0.00005	mg/L
Pesticides	ALDRIN	608/8081A/B, 6630C	GW, WW	39.0-123	20.0	0.00005	mg/L
Pesticides	ALPHA BHC	608/8081A/B, 6630C	GW, WW	60.1-128	20.0	0.00005	mg/L
Pesticides	BETA BHC	608/8081A/B, 6630C	GW, WW	59.2-135	20.0	0.00005	mg/L
Pesticides	ALPHA CHLORDANE	608/8081A/B, 6630C	GW, WW	63.7-132	20.0	0.005	mg/L
Pesticides	DELTA BHC	608/8081A/B, 6630C	GW, WW	61.8-131	20.0	0.00005	mg/L
Pesticides	DIELDRIN	608/8081A/B, 6630C	GW, WW	61.4-130	20.0	0.00005	mg/L
Pesticides	ENDOSULFAN I	608/8081A/B, 6630C	GW, WW	61.8-131	20.0	0.00005	mg/L
Pesticides	ENDOSULFAN II	608/8081A/B, 6630C	GW, WW	54.8-138	20.0	0.00005	mg/L
Pesticides	ENDOSULFAN SULFATE	608/8081A/B, 6630C	GW, WW	61.9-139	20.0	0.00005	mg/L
Pesticides	ENDRIN	608/8081A/B, 6630C	GW, WW	53.8-125	20.0	0.00005	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
Pesticides	ENDRIN ALDEHYDE	608/8081A/B, 6630C	GW, WW	63.0-129	20.0	0.00005	mg/L
Pesticides	ENDRIN KETONE	608/8081A/B, 6630C	GW, WW	61.3-129	20.0	0.00005	mg/L
Pesticides	GAMMA BHC	608/8081A/B, 6630C	GW, WW	43.3-123	20.0	0.00005	mg/L
Pesticides	HEPTACHLOR	608/8081A/B, 6630C	GW, WW	61.8-130	20.0	0.00005	mg/L
Pesticides	HEPTACHLOR EPOXIDE	608/8081A/B, 6630C	GW, WW	48.3-110	20.0	0.00005	mg/L
Pesticides	HEXACHLOROBENZENE	608/8081A/B, 6630C	GW, WW	48.3-110	20.0	0.00005	mg/L
Pesticides	METHOXYCHLOR	608/8081A/B, 6630C	GW, WW	62.1-135	20.0	0.00005	mg/L
PCBs	PCB 1016	608, 6431B, 8082/A	GW, WW	55.5-103	20.0	0.0005	mg/L
PCBs	PCB 1260	608, 6431B, 8082/A	GW, WW	51.2-111	22.0	0.0005	mg/L
PCBs	PCB 1016	8082/A	SS	46.3-117	27.5	0.017	mg/Kg
PCBs	PCB 1260	8082/A	SS	46.5-120	27.0	0.017	mg/Kg
Pesticides	4,4-DDD	8081A/B	SS	70.8-120	20.0	0.02	mg/Kg
Pesticides	4,4-DDE	8081A/B	SS	70.9-121	20.0	0.02	mg/Kg
Pesticides	4,4-DDT	8081A/B	SS	68.1-124	20.0	0.02	mg/Kg
Pesticides	ALDRIN	8081A/B	SS	71.1-120	20.0	0.02	mg/Kg
Pesticides	ALPHA BHC	8081A/B	SS	69.9-121	20.0	0.02	mg/Kg
Pesticides	BETA BHC	8081A/B	SS	69.6-121	20.0	0.02	mg/Kg
Pesticides	DELTA BHC	8081A/B	SS	68.1-127	20.0	0.02	mg/Kg
Pesticides	DIELDRIN	8081A/B	SS	71.3-122	20.0	0.02	mg/Kg
Pesticides	ENDOSULFAN I	8081A/B	SS	71.6-122	20.0	0.02	mg/Kg
Pesticides	ENDOSULFAN II	8081A/B	SS	71.1-120	20.0	0.02	mg/Kg
Pesticides	ENDOSULFAN SULFATE	8081A/B	SS	67.4-125	20.0	0.02	mg/Kg
Pesticides	ENDRIN	8081A/B	SS	69.6-126	20.0	0.02	mg/Kg
Pesticides	ENDRIN ALDEHYDE	8081A/B	SS	59.9-114	20.0	0.02	mg/Kg
Pesticides	ENDRIN KETONE	8081A/B	SS	70.8-122	20.0	0.02	mg/Kg
Pesticides	GAMMA BHC	8081A/B	SS	70.1-121	20.0	0.02	mg/Kg
Pesticides	HEPTACHLOR	8081A/B	SS	63.3-126	20.0	0.02	mg/Kg
Pesticides	HEPTACHLOR EPOXIDE	8081A/B	SS	71.9-121	20.0	0.02	mg/Kg
Pesticides	HEXACHLOROBENZENE	8081A/B	SS	62.7-117	20.0	0.02	mg/Kg
Pesticides	METHOXYCHLOR	8081A/B	SS	69.3-122	20.0	0.02	mg/Kg

<b>Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs</b> <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
Herbicides	2,4,5-T	1658, 8151A, 6640C	GW, WW	50.0-121	26.5	0.002	mg/L
Herbicides	2,4,5-TP (SILVEX)	1658, 8151A, 6640C	GW, WW	46.3-127	29.5	0.002	mg/L
Herbicides	2,4-D	1658, 8151A, 6640C	GW, WW	31.1-136	28.6	0.002	mg/L
Herbicides	2,4-DB	1658, 8151A, 6640C	GW, WW	39.5-128	31.9	0.002	mg/L
Herbicides	DALAPON	1658, 8151A, 6640C	GW, WW	36.6-132	29.2	0.002	mg/L
Herbicides	DICAMBA	1658, 8151A, 6640C	GW, WW	53.7-134	20.0	0.002	mg/L
Herbicides	DICHLOROPROP	1658, 8151A, 6640C	GW, WW	42.5-109	26.8	0.002	mg/L
Herbicides	DINOSEB	1658, 8151A, 6640C	GW, WW	42.5-112	21.3	0.002	mg/L
Herbicides	MCPA	1658, 8151A, 6640C	GW, WW	30.5-137	31.4	0.1	mg/L
Herbicides	MCPP	1658, 8151A, 6640C	GW, WW	33.2-148	25.2	0.1	mg/L
Herbicides	2,4,5-T	8151A	SS	44.9-111	21.5	0.07	mg/Kg
Herbicides	2,4,5-TP (SILVEX)	8151A	SS	48.4-110	25.9	0.07	mg/Kg
Herbicides	2,4-D	8151A	SS	40.0-112	24.8	0.07	mg/Kg
Herbicides	2,4-DB	8151A	SS	33.8-126	27.8	0.07	mg/Kg
Herbicides	DALAPON	8151A	SS	36.7-119	28.0	0.07	mg/Kg
Herbicides	DICAMBA	8151A	SS	50.2-125	20.0	0.07	mg/Kg
Herbicides	DICHLOROPROP	8151A	SS	39.9-99.0	20.1	0.07	mg/Kg
Herbicides	DINOSEB	8151A	SS	15.6-109	40.0	0.07	mg/Kg
Herbicides	MCPA	8151A	SS	34.7-110	31.7	6.5	mg/Kg
Herbicides	MCPP	8151A	SS	41.0-121	24.9	6.5	mg/Kg
PAH	PYRENE	8310, 610, 6440B	GW, WW	69.2-96.9	20.0	0.0001	mg/L
PAH	PHENANTHRENE	8310, 610, 6440B	GW, WW	66.5-95.7	20.0	0.0001	mg/L
PAH	NAPHTHALENE	8310, 610, 6440B	GW, WW	47.5-86.6	20.2	0.0001	mg/L
PAH	INDENO(1,2,3-CD)PYRENE	8310, 610, 6440B	GW, WW	52.4-104	20.0	0.0001	mg/L
PAH	FLUORENE	8310, 610, 6440B	GW, WW	55.3-98.8	20.0	0.0001	mg/L
PAH	FLUORANTHENE	8310, 610, 6440B	GW, WW	70.4-102	20.0	0.0001	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
PAH	DIBENZ(A,H)ANTHRACENE	8310, 610, 6440B	GW, WW	38.6-111	22.2	0.0001	mg/L
PAH	CHRYSENE	8310, 610, 6440B	GW, WW	72.9-107	20.0	0.0001	mg/L
PAH	BENZO(K)FLUORANTHENE	8310, 610, 6440B	GW, WW	67.3-102	20.0	0.0001	mg/L
PAH	BENZO(G,H,I)PERYLENE	8310, 610, 6440B	GW, WW	41.9-115	20.0	0.0001	mg/L
PAH	BENZO(B)FLUORANTHENE	8310, 610, 6440B	GW, WW	68.5-102	20.0	0.0001	mg/L
PAH	BENZO(A)PYRENE	8310, 610, 6440B	GW, WW	58.8-106	20.0	0.0001	mg/L
PAH	BENZO(A)ANTHRACENE	8310, 610, 6440B	GW, WW	72.4-102	20.0	0.0001	mg/L
PAH	ANTHRACENE	8310, 610, 6440B	GW, WW	68.8-99.3	20.0	0.0001	mg/L
PAH	ACENAPHTHYLENE	8310, 610, 6440B	GW, WW	59.4-91.9	20.0	0.0001	mg/L
PAH	ACENAPHTHENE	8310, 610, 6440B	GW, WW	57.0-89.5	20.0	0.0001	mg/L
PAH	2-METHYLNAPHTHALENE	8310, 610, 6440B	GW, WW	45.7-92.1	20.0	0.0001	mg/L
PAH	1-METHYLNAPHTHALENE	8310, 610, 6440B	GW, WW	54.6-104	20.0	0.0001	mg/L
PAH	PYRENE	8310	SS	71.9-100	20.0	0.02	mg/Kg
PAH	PHENANTHRENE	8310	SS	66.9-97.1	20.0	0.02	mg/Kg
PAH	NAPHTHALENE	8310	SS	52.0-94.2	20.0	0.02	mg/Kg
PAH	INDENO(1,2,3-CD)PYRENE	8310	SS	64.6-101	20.0	0.02	mg/Kg
PAH	FLUORENE	8310	SS	58.6-100	20.0	0.02	mg/Kg
PAH	FLUORANTHENE	8310	SS	73.4-103	20.0	0.02	mg/Kg
PAH	DIBENZ(A,H)ANTHRACENE	8310	SS	72.1-100	20.0	0.02	mg/Kg
PAH	CHRYSENE	8310	SS	77.3-107	20.0	0.02	mg/Kg
PAH	BENZO(K)FLUORANTHENE	8310	SS	73.3-102	20.0	0.02	mg/Kg
PAH	BENZO(G,H,I)PERYLENE	8310	SS	67.1-110	20.0	0.02	mg/Kg
PAH	BENZO(B)FLUORANTHENE	8310	SS	73.9-103	20.0	0.02	mg/Kg
PAH	BENZO(A)PYRENE	8310	SS	66.5-104	20.0	0.02	mg/Kg
PAH	BENZO(A)ANTHRACENE	8310	SS	77.7-102	20.0	0.02	mg/Kg
PAH	ANTHRACENE	8310	SS	71.9-101	20.0	0.02	mg/Kg
PAH	ACENAPHTHYLENE	8310	SS	59.5-98.4	20.0	0.02	mg/Kg
PAH	ACENAPHTHENE	8310	SS	58.6-95.5	20.0	0.02	mg/Kg
PAH	2-METHYLNAPHTHALENE	8310	SS	54.9-95.3	20.0	0.02	mg/Kg

<b>Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs</b> <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
PAH	1-METHYLNAPHTHALENE	8310	SS	62.3-110	20.0	0.02	mg/Kg
BNA	PYRIDINE	8270C/D 625	GW,WW	13.0-54.0	32.8	0.01	mg/L
BNA	PYRENE	8270C/D 625	GW,WW	40.2-135	20.0	0.001	mg/L
BNA	PHENOL	8270C/D 625	GW,WW	10.0-77.3	24.6	0.01	mg/L
BNA	PHENANTHRENE	8270C/D 625	GW,WW	41.4-134	20.0	0.001	mg/L
BNA	PENTACHLOROPHENOL	8270C/D 625	GW,WW	17.0-117	34.3	0.01	mg/L
BNA	N-OCTADECANE	8270C/D 625	GW,WW	28.3-151	20.0	0.01	mg/L
BNA	N-NITROSODIPHENYLAMINE	8270C/D 625	GW,WW	41.1-134	20.0	0.01	mg/L
BNA	N-NITRODIPHENYLAMINE	8270C/D 625	GW,WW	40.1-157	20.0	0.01	mg/L
BNA	N-NITROSODI-N-PROPYLAMINE	8270C/D 625	GW,WW	35.6-125	20.0	0.01	mg/L
BNA	N-NITROSODIMETHYLAMINE	8270C/D 625	GW,WW	12.3-70.5	33.0	.01	mg/L
BNA	NITROBENZENE	8270C/D 625	GW,WW	34.4-121	21.2	.01	mg/L
BNA	N-DECANE	8270C/D 625	GW,WW	10.0-118	32.3	0.01	mg/L
BNA	NAPHTHALENE	8270C/D 625	GW,WW	33.0-117	20.0	0.001	mg/L
BNA	ISOPHORONE	8270C/D 625	GW,WW	30.5-109	20.0	0.01	mg/L
BNA	INDENO(1,2,3-CD)PYRENE	8270C/D 625	GW,WW	41.0-140	20.0	0.01	mg/L
BNA	HEXACHLOROETHANE	8270C/D 625	GW,WW	22.2-109	25.8	0.01	mg/L
BNA	HEXACHLOROCYCLOPENTADIENE	8270C/D 625	GW,WW	13.5-122	21.6	0.00	mg/L
BNA	HEXACHLOROBENZENE	8270C/D 625	GW,WW	34.1-125	20.0	0.001	mg/L
BNA	HEXACHLORO-1,3-BUTADIENE	8270C/D 625	GW,WW	24.9-121	22.0	0.01	mg/L
BNA	FLUORENE	8270C/D 625	GW,WW	39.9-132	20.0	0.001	mg/L
BNA	FLUORANTHENE	8270C/D 625	GW,WW	41.4-141	20.0	0.001	mg/L
BNA	DI-N-OCTYL PHTHALATE	8270C/D 625	GW,WW	39.8-146	20.0	0.003	mg/L
BNA	DI-N-BUTYL PHTHALATE	8270C/D 625	GW,WW	33.0-151	20.0	0.003	mg/L
BNA	DIMETHYL PHTHALATE	8270C/D 625	GW,WW	23.4-138	20.2	0.003	mg/L
BNA	DIETHYL PHTHALATE	8270C/D 625	GW,WW	36.0-140	20.0	0.003	mg/L
BNA	DIBENZOFURAN	8270C/D 625	GW,WW	37.9-128	20.0	0.01	mg/L
BNA	DIBENZ(A,H)ANTHRACENE	8270C/D 625	GW,WW	39.9-141	20.0	0.001	mg/L
BNA	CHRYSENE	8270C/D 625	GW,WW	40.5-140	20.0	0.001	mg/L
BNA	CARBAZOLE	8270C/D 625	GW,WW	41.0-137	20.0	0.01	mg/L
BNA	CAPROLACTAM	8270C/D 625	GW,WW	10.0-45.6	25.2	0.01	mg/L
BNA	BIS(2-ETHYLHEXYL)PHTHALATE	8270C/D 625	GW,WW	41.4-150	20.0	0.003	mg/L
BNA	BIS(2-CHLOROISOPROPYL)ETHER	8270C/D 625	GW,WW	33.6-115	21.3	0.01	mg/L
BNA	BIS(2-CHLOROETHYL)ETHER	8270C/D 625	GW,WW	29.8-114	25.3	0.01	mg/L
BNA	BIS(2-CHLOROETHOXY)METHANE	8270C/D 625	GW,WW	36.7-123	20.0	0.01	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	BIPHENYL	8270C/D 625	GW,WW	36.9-126	20.0	0.01	mg/L
BNA	BENZYL BUTYL PHTHALATE	8270C/D 625	GW,WW	29.2-146	20.7	0.003	mg/L
BNA	BENZYL ALCOHOL	8270C/D 625	GW,WW	26.0-104	21.0	0.01	mg/L
BNA	BENZOIC ACID	8270C/D 625	GW,WW	10.0-54.3	40.0	0.01	mg/L
BNA	BENZO(K)FLUORANTHENE	8270C/D 625	GW,WW	41.5-140	20.0	0.001	mg/L
BNA	BENZO(G,H,I)PERYLENE	8270C/D 625	GW,WW	38.8-137	20.0	0.001	mg/L
BNA	BENZO(B)FLUORANTHENE	8270C/D 625	GW,WW	40.5-137	20.0	0.001	mg/L
BNA	BENZO(A)PYRENE	8270C/D 625	GW,WW	41.7-138	20.0	0.001	mg/L
BNA	BENZO(A)ANTHRACENE	8270C/D 625	GW,WW	42.3-137	20.0	0.001	mg/L
BNA	BENZIDINE	8270C/D 625	GW,WW	10.0-75.5	40.0	0.01	mg/L
BNA	BENZALDEHYDE	8270C/D 625	GW,WW	10.0-93.4	27.8	0.01	mg/L
BNA	AZOBENZENE	8270C/D 625	GW,WW	37.2-129	20.0	0.01	mg/L
BNA	ATRAZINE	8270C/D 625	GW,WW	40.6-154	20.0	0.01	mg/L
BNA	ANTHRACENE	8270C/D 625	GW,WW	42.9-138	20.0	0.001	mg/L
BNA	ANILINE	8270C/D 625	GW,WW	22.5-99.1	28.3	0.01	mg/L
BNA	ACETOPHENONE	8270C/D 625	GW,WW	35.6-122	20.0	0.01	mg/L
BNA	ACENAPHTHYLENE	8270C/D 625	GW,WW	41.0-135	20.0	0.001	mg/L
BNA	ACENAPHTHENE	8270C/D 625	GW,WW	39.0-128	20.0	0.001	mg/L
BNA	4-NITROPHENOL	8270C/D 625	GW,WW	10.0-65.4	33.6	0.01	mg/L
BNA	4-NITROANILINE	8270C/D 625	GW,WW	37.3-159	20.0	0.01	mg/L
BNA	4-CHLOROPHENYL-PHENYLETHER	8270C/D 625	GW,WW	37.3-130	20.0	0.01	mg/L
BNA	4-CHLOROANILINE	8270C/D 625	GW,WW	29.8-128	20.9	0.01	mg/L
BNA	4-CHLORO-3-METHYLPHENOL	8270C/D 625	GW,WW	34.6-130	20.0	0.01	mg/L
BNA	4-BROMOPHENYL-PHENYLETHER	8270C/D 625	GW,WW	39.0-137	20.0	0.01	mg/L
BNA	4,6-DINITRO-2-METHYLPHENOL	8270C/D 625	GW,WW	28.2-134	29.2	0.01	mg/L
BNA	3-NITROANILINE	8270C/D 625	GW,WW	34.8-132	20.0	0.01	mg/L
BNA	3,3-DICHLOROBENZIDINE	8270C/D 625	GW,WW	33.1-134	20.0	0.01	mg/L
BNA	3&4-METHYLPHENOL	8270C/D 625	GW,WW	23.1-107	20.7	0.01	mg/L
BNA	2-NITROPHENOL	8270C/D 625	GW,WW	38.3-125	20.0	0.01	mg/L
BNA	2-NITROANILINE	8270C/D 625	GW,WW	41.9-143	20.0	0.01	mg/L
BNA	2-METHYLPHENOL	8270C/D 625	GW,WW	23.9-97	20.0	0.01	mg/L
BNA	2-METHYLNAPHTHALENE	8270C/D 625	GW,WW	35.6-124	20.0	0.001	mg/L
BNA	2-CHLOROPHENOL	8270C/D 625	GW,WW	31.2-103	20.0	0.01	mg/L
BNA	2-CHLORONAPHTHALENE	8270C/D 625	GW,WW	35.1-123	20.0	0.001	mg/L
BNA	2,6-DINITROTOLUENE	8270C/D 625	GW,WW	41.0-139	20.0	0.01	mg/L
BNA	2,4-DINITROTOLUENE	8270C/D 625	GW,WW	42.3-143	20.0	0.01	mg/L



Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
BNA	2,4-DINITROPHENOL	8270C/D 625	GW,WW	10.0-108	40.0	0.01	mg/L
BNA	2,4-DIMETHYLPHENOL	8270C/D 625	GW,WW	33.8-126	20.0	0.01	mg/L
BNA	2,4-DICHLOROPHENOL	8270C/D 625	GW,WW	39.6-121	20.0	0.01	mg/L
BNA	2,4,6-TRICHLOROPHENOL	8270C/D 625	GW,WW	35.9-129	22.4	0.01	mg/L
BNA	2,4,5-TRICHLOROPHENOL	8270C/D 625	GW,WW	35.4-136	20.0	0.01	mg/L
BNA	1-METHYLNAPHTHALENE	8270C/D 625	GW,WW	34.3-123	20.0	0.001	mg/L
BNA	1,4-DICHLOROBENZENE	8270C/D 625	GW,WW	24.8-105	25.2	0.01	mg/L
BNA	1,3-DICHLOROBENZENE	8270C/D 625	GW,WW	23.9-103	25.2	0.01	mg/L
BNA	1,2-DICHLOROBENZENE	8270C/D 625	GW,WW	26.1-107	25.4	0.01	mg/L
BNA	1,2,4-TRICHLOROBENZENE	8270C/D 625	GW,WW	26.6-109	20.0	0.01	mg/L
BNA	1,2,4,5-TETRACHLOROBENZENE	8270C/D 625	GW,WW	30.8-124	20.7	0.01	mg/L
BNA	PYRIDINE	8270C/D	SS	10.0-90.0	38.3	0.33	mg/Kg
BNA	PYRENE	8270C/D	SS	47.1-108	20.0	0.33	mg/Kg
BNA	PHENOL	8270C/D	SS	41.5-106	20.0	0.33	mg/Kg
BNA	PHENANTHRENE	8270C/D	SS	51.6-107	20.0	0.33	mg/Kg
BNA	PENTACHLOROPHENOL	8270C/D	SS	16.2-102	22.9	0.33	mg/Kg
BNA	N-OCTADECANE	8270C/D	SS	40.7-122	20.0	0.33	mg/Kg
BNA	N-NITROSODIPHENYLAMINE	8270C/D	SS	48.8-107	20.0	0.33	mg/Kg
BNA	N-NITROSODI-N-PROPYLAMINE	8270C/D	SS	43.3-109	20.0	0.33	mg/Kg
BNA	N-NITROSODIMETHYLAMINE	8270C/D	SS	18.1-1422	23.5	0.33	mg/Kg
BNA	NITROBENZENE	8270C/D	SS	40.7-109	21.0	0.33	mg/Kg
BNA	N-DECANE	8270C/D	SS	38.1-116	20.0	0.33	mg/Kg
BNA	NAPHTHALENE	8270C/D	SS	43.4-103	20.0	0.33	mg/Kg
BNA	ISOPHORONE	8270C/D	SS	28.8-104	20.0	0.033	mg/Kg
BNA	INDENO(1,2,3-CD)PYRENE	8270C/D	SS	47.5-109	20.0	0.33	mg/Kg
BNA	HEXACHLOROETHANE	8270C/D	SS	36.2-103	22.7	0.033	mg/Kg
BNA	HEXACHLOROCYCLOPENTADIENE	8270C/D	SS	13.5-123	20.7	0.33	mg/Kg
BNA	HEXACHLOROBENZENE	8270C/D	SS	43.2-104	20.1	0.33	mg/Kg
BNA	HEXACHLORO-1,3-BUTADIENE	8270C/D	SS	41.5-112	20.0	0.33	mg/Kg
BNA	FLUORENE	8270C/D	SS	51.1-109	20.0	0.33	mg/Kg
BNA	FLUORANTHENE	8270C/D	SS	53.7-110	20.0	0.33	mg/Kg
BNA	DI-N-OCTYL PHTHALATE	8270C/D	SS	49.6-112	20.0	0.33	mg/Kg
BNA	DI-N-BUTYL PHTHALATE	8270C/D	SS	49.7-113	20.0	0.33	mg/Kg
BNA	DIMETHYL PHTHALATE	8270C/D	SS	51.4-108	20.0	0.33	mg/Kg
BNA	DIETHYL PHTHALATE	8270C/D	SS	52.0-112	20.0	0.33	mg/Kg
BNA	DIBENZOFURAN	8270C/D	SS	48.6-104	20.0	0.33	mg/Kg

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
BNA	DIBENZ(A,H)ANTHRACENE	8270C/D	SS	45.7-111	20.0	0.33	mg/Kg
BNA	CHRYSENE	8270C/D	SS	54.4-110	20.0	0.33	mg/Kg
BNA	CARBAZOLE	8270C/D	SS	52.4-102	21.1	0.33	mg/Kg
BNA	CAPROLACTAM	8270C/D	SS	42.2-107	21.9	0.33	mg/Kg
BNA	BIS(2-ETHYLHEXYL)PHTHALATE	8270C/D	SS	48.1-116	20.5	0.33	mg/Kg
BNA	BIS(2-CHLOROISOPROPYL)ETHER	8270C/D	SS	40.4-99.0	20.7	0.33	mg/Kg
BNA	BIS(2-CHLOROETHYL)ETHER	8270C/D	SS	32.5-112	26.0	0.33	mg/Kg
BNA	BIS(2-CHLORETHOXY)METHANE	8270C/D	SS	44.9-108	20.0	0.33	mg/Kg
BNA	BIPHENYL	8270C/D	SS	45.6-103	20.0	0.33	mg/Kg
BNA	BENZYL BUTYL PHTHALATE	8270C/D	SS	47.5-115	20.0	0.33	mg/Kg
BNA	BENZYL ALCOHOL	8270C/D	SS	49.1-105	20.0	0.033	mg/Kg
BNA	BENZOIC ACID	8270C/D	SS	0.00-82.0	32.5	0.033	mg/Kg
BNA	BENZO(K)FLUORANTHENE	8270C/D	SS	52.9-107	20.0	0.33	mg/Kg
BNA	BENZO(G,H,I)PERYLENE	8270C/D	SS	45.8-108	20.0	0.33	mg/Kg
BNA	BENZO(B)FLUORANTHENE	8270C/D	SS	51.3-106	20.0	0.33	mg/Kg
BNA	BENZO(A)PYRENE	8270C/D	SS	51.9-106	20.0	0.33	mg/Kg
BNA	BENZO(A)ANTHRACENE	8270C/D	SS	52.3-106	20.0	0.33	mg/Kg
BNA	BENZIDINE	8270C/D	SS	0.00-48.0	40.0	0.033	mg/Kg
BNA	BENZALDEHYDE	8270C/D	SS	46.4-109	24.8	0.33	mg/Kg
BNA	AZOBENZENE	8270C/D	SS	45.0-131	20.0	0.33	mg/Kg
BNA	ATRAZINE	8270C/D	SS	45.0-131	20.0	0.33	mg/Kg
BNA	ANTHRACENE	8270C/D	SS	52.0-112	20.0	0.33	mg/Kg
BNA	ANILINE	8270C/D	SS	10.0-94.0	24.2	0.33	mg/Kg
BNA	ACETOPHENONE	8270C/D	SS	47.1-99.0	22.1	0.33	mg/Kg
BNA	ACENAPHTHYLENE	8270C/D	SS	49.2-111	20.0	0.033	mg/Kg
BNA	ACENAPHTHENE	8270C/D	SS	48.9-107	20.0	0.033	mg/Kg
BNA	4-NITROPHENOL	8270C/D	SS	34.8-109	20.0	0.033	mg/Kg
BNA	4-NITROANILINE	8270C/D	SS	38.6-133	21.7	0.033	mg/Kg
BNA	4-CHLOROPHENYL-PHENYLETHER	8270C/D	SS	48.1-108	20.0	0.033	mg/Kg
BNA	4-CHLOROANILINE	8270C/D	SS	24.5-101	24.5	0.33	mg/Kg
BNA	4-CHLORO-3-METHYLPHENOL	8270C/D	SS	51.1-113	20.0	0.33	mg/Kg
BNA	4-BROMOPHENYL-PHENYLETHER	8270C/D	SS	51.4-110	20.0	0.33	mg/Kg
BNA	4,6-DINITRO-2-METHYLPHENOL	8270C/D	SS	23.1-119	23.7	0.33	mg/Kg
BNA	3-NITROANILINE	8270C/D	SS	34.7-103	20.7	0.33	mg/Kg

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
BNA	3,3-DICHLORO BENZIDINE	8270C/D	SS	21.0-101	22.0	0.33	mg/Kg
BNA	3&4-METHYLPHENOL	8270C/D	SS	50.5-115	20.0	0.33	mg/Kg
BNA	2-NITROPHENOL	8270C/D	SS	44.2-113	20.9	0.33	mg/Kg
BNA	2-NITROANILINE	8270C/D	SS	56.2-117	20.0	0.33	mg/Kg
BNA	2-METHYLPHENOL	8270C/D	SS	53.8-107	20.0	0.33	mg/Kg
BNA	2-METHYLNAPHTHALENE	8270C/D	SS	42.4-100	20.0	0.033	mg/Kg
BNA	2-CHLOROPHENOL	8270C/D	SS	48.0-101	20.0	0.33	mg/Kg
BNA	2-CHLORONAPHTHALENE	8270C/D	SS	40.8-103	20.0	0.33	mg/Kg
BNA	2,6-DINITROTOLUENE	8270C/D	SS	47.1-105	20.0	0.33	mg/Kg
BNA	2,4-DINITROTOLUENE	8270C/D	SS	51.6-110	20.0	0.033	mg/Kg
BNA	2,4-DINITROPHENOL	8270C/D	SS	53.0-112	36.5	0.33	mg/Kg
BNA	2,4-DIMETHYLPHENOL	8270C/D	SS	10.0-105	20.0	0.33	mg/Kg
BNA	2,4-DICHLOROPHENOL	8270C/D	SS	42.2-109	20.0	0.33	mg/Kg
BNA	2,4,6-TRICHLOROPHENOL	8270C/D	SS	44.4-108	20.0	0.33	mg/Kg
BNA	2,4,5-TRICHLOROPHENOL	8270C/D	SS	43.3-110	20.0	0.33	mg/Kg
BNA	1-METHYLNAPHTHALENE	8270C/D	SS	49.8-104	20.0	0.33	mg/Kg
BNA	1,4-DICHLOROBENZENE	8270C/D	SS	36.5-97.0	20.0	0.33	mg/Kg
BNA	1,3-DICHLOROBENZENE	8270C/D	SS	35.0-94.0	20.0	0.33	mg/Kg
BNA	1,2-DICHLOROBENZENE	8270C/D	SS	37.2-98.0	20.0	0.33	mg/Kg
BNA	1,2,4-TRICHLOROBENZENE	8270C/D	SS	39.8-100	20.0	0.33	mg/Kg
BNA	1,2,4,5-TETRACHLOROBENZENE	8270C/D	SS	47.6-107	20.0	0.33	mg/Kg
BNA	PYRIDINE	8270C/D RV	GW,WW	13.5-58.9	32.5	0.01	mg/L
BNA	PYRENE	8270C/D RV	GW,WW	463-117	20.0	0.001	mg/L
BNA	PHENOL	8270C/D RV	GW,WW	10.0-57.9	35.0	0.01	mg/L
BNA	PHENANTHRENE	8270C/D RV	GW,WW	46.4-113	20.0	0.001	mg/L
BNA	PENTACHLOROPHENOL	8270C/D RV	GW,WW	10.9-97.4	35.1	0.01	mg/L
BNA	N-OCTADECANE	8270C/D RV	GW,WW	15.8-132	21.1	0.01	mg/L
BNA	N-NITROSODIPHENYLAMINE	8270C/D RV	GW,WW	44.4-113	20.0	0.01/ .05ug/L SIM	mg/L
BNA	N-NITROSODI-N-PROPYLAMINE	8270C/D RV	GW,WW	33.2-106	23.7	0.01	mg/L
BNA	N-NITROSODIMETHYLAMINE	8270C/D RV	GW,WW	33.2-106	37.5	0.01	mg/L
BNA	NITROBENZENE	8270C/D RV	GW,WW	31.4-106	25.7	0.01	mg/L
BNA	N-DECANE	8270C/D RV	GW,WW	10.0-95.2	40.0	0.01	mg/L
BNA	NAPHTHALENE	8270C/D RV	GW,WW	32.2-101	23.8	0.001	mg/L
BNA	ISOPHORONE	8270C/D RV	GW,WW	35.4-112	21.5	0.01	mg/L
BNA	INDENO(1,2,3-CD)PYRENE	8270C/D RV	GW,WW	45.0-116	20.0	0.01	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
BNA	HEXACHLOROETHANE	8270C/D RV	GW,WW	16.5-89.8	30.7	0.01	mg/L
BNA	HEXACHLOROCYCLOPENTADIENE	8270C/D RV	GW,WW	10.0-121	27.9	0.00	mg/L
BNA	HEXACHLOROBENZENE	8270C/D RV	GW,WW	38.5-116	20.1	0.001	mg/L
BNA	HEXACHLORO-1,3-BUTADIENE	8270C/D RV	GW,WW	16.1-104	31.2	0.01	mg/L
BNA	FLUORENE	8270C/D RV	GW,WW	41.0-112	20.2	0.001	mg/L
BNA	FLUORANTHENE	8270C/D RV	GW,WW	45.9-115	20.0	0.001	mg/L
BNA	DI-N-OCTYL PHTHALATE	8270C/D RV	GW,WW	39.7-112	21.1	0.003	mg/L
BNA	DI-N-BUTYL PHTHALATE	8270C/D RV	GW,WW	41.8-120	20.2	0.003	mg/L
BNA	DIMETHYL PHTHALATE	8270C/D RV	GW,WW	35.3-128	20.8	0.003	mg/L
BNA	DIETHYL PHTHALATE	8270C/D RV	GW,WW	36.5-129	20.0	0.003	mg/L
BNA	DIBENZOFURAN	8270C/D RV	GW,WW	42.4-105	20.0	0.01	mg/L
BNA	DIBENZ(A,H)ANTHRACENE	8270C/D RV	GW,WW	42.8-118	20.0	0.001	mg/L
BNA	CHRYSENE	8270C/D RV	GW,WW	54.6-120	20.0	0.001	mg/L
BNA	CARBAZOLE	8270C/D RV	GW,WW	49.0-110	20.0	0.01	mg/L
BNA	CAPROLACTAM	8270C/D RV	GW,WW	10.0-40.4	40.0	0.01	mg/L
BNA	BIS(2-ETHYLHEXYL)PHTHALATE	8270C/D RV	GW,WW	36.9-134	23.6	0.003	mg/L
BNA	BIS(2-CHLOROISOPROPYL)ETHER	8270C/D RV	GW,WW	32.9-100	25.1	0.01	mg/L
BNA	BIS(2-CHLOROETHYL)ETHER	8270C/D RV	GW,WW	22.6-108	27.9	0.01	mg/L
BNA	BIS(2-CHLORETHOXY)METHANE	8270C/D RV	GW,WW	37.2-111	24.1	0.01	mg/L
BNA	BIPHENYL	8270C/D RV	GW,WW	38.0-103	20.1	0.01	mg/L
BNA	BENZYL BUTYL PHTHALATE	8270C/D RV	GW,WW	31.8-123	20.7	0.003	mg/L
BNA	BENZYL ALCOHOL	8270C/D RV	GW,WW	30.1-89.2	24.8	0.01	mg/L
BNA	BENZOIC ACID	8270C/D RV	GW,WW	0.00-79.4	31.1	0.01	mg/L
BNA	BENZO(K)FLUORANTHENE	8270C/D RV	GW,WW	49.4-114	20.0	0.001	mg/L
BNA	BENZO(G,H,I)PERYLENE	8270C/D RV	GW,WW	45.2-117	20.0	0.001	mg/L
BNA	BENZO(B)FLUORANTHENE	8270C/D RV	GW,WW	47.6-110	20.0	0.001	mg/L
BNA	BENZO(A)PYRENE	8270C/D RV	GW,WW	45.6-106	20.0	0.001	mg/L
BNA	BENZO(A)ANTHRACENE	8270C/D RV	GW,WW	51.2-112	20.0	0.001	mg/L
BNA	BENZIDINE	8270C/D RV	GW,WW	10.0-165	40.0	0.01	mg/L
BNA	BENZALDEHYDE	8270C/D RV	GW,WW	11.7-132	25.2	0.01	mg/L
BNA	AZOBENZENE	8270C/D RV	GW,WW	37.6-111	21.1	0.01	mg/L
BNA	ATRAZINE	8270C/D RV	GW,WW	50.0-123	21.5	0.01	mg/L
BNA	ANTHRACENE	8270C/D RV	GW,WW	43.6-113	18.7	0.001	mg/L
BNA	ANILINE	8270C/D RV	GW,WW	25.8-88.1	26.3	0.01	mg/L
BNA	ACETOPHENONE	8270C/D RV	GW,WW	41.6-104	24.8	0.01	mg/L
BNA	ACENAPHTHYLENE	8270C/D RV	GW,WW	36.0-106	21.0	0.001	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
BNA	ACENAPHTHENE	8270C/D RV	GW,WW	38.7-109	21.5	0.001	mg/L
BNA	4-NITROPHENOL	8270C/D RV	GW,WW	10.0-52.7	40.0	0.01	mg/L
BNA	4-NITROANILINE	8270C/D RV	GW,WW	35.4-124	23.1	0.01	mg/L
BNA	4-CHLOROPHENYL-PHENYLETHER	8270C/D RV	GW,WW	39.0-113	20.9	0.01	mg/L
BNA	4-CHLOROANILINE	8270C/D RV	GW,WW	32.0-104	26.4	0.01	mg/L
BNA	4-CHLORO-3-METHYLPHENOL	8270C/D RV	GW,WW	35.7-100	22.9	0.01	mg/L
BNA	4-BROMOPHENYL-PHENYLETHER	8270C/D RV	GW,WW	40.7-116	21.0	0.01	mg/L
BNA	4,6-DINITRO-2-METHYLPHENOL	8270C/D RV	GW,WW	18.4-148	24.4	0.01	mg/L
BNA	3-NITROANILINE	8270C/D RV	GW,WW	33.6-103	21.8	0.01	mg/L
BNA	3,3-DICHLORO BENZIDINE	8270C/D RV	GW,WW	27.2-142	22.3	0.01	mg/L
BNA	3&4-METHYLPHENOL	8270C/D RV	GW,WW	27.9-92	27.0	0.01	mg/L
BNA	2-NITROPHENOL	8270C/D RV	GW,WW	25.9-106	26.9	0.01	mg/L
BNA	2-NITROANILINE	8270C/D RV	GW,WW	56.4-173	20.0	0.01	mg/L
BNA	2-METHYLPHENOL	8270C/D RV	GW,WW	35.6-113	20.9	0.01	mg/L
BNA	2-METHYLNAPHTHALENE	8270C/D RV	GW,WW	26.4-86.9	26.5	0.001	mg/L
BNA	2-CHLOROPHENOL	8270C/D RV	GW,WW	33.8-98.6	24.2	0.01	mg/L
BNA	2-CHLORONAPHTHALENE	8270C/D RV	GW,WW	26.2-91.5	26.5	0.001	mg/L
BNA	2,6-DINITROTOLUENE	8270C/D RV	GW,WW	33.6-105	23.0	0.01	mg/L
BNA	2,4-DINITROTOLUENE	8270C/D RV	GW,WW	30.6-106	23.1	0.01	mg/L
BNA	2,4-DINITROPHENOL	8270C/D RV	GW,WW	31.2-105	22.0	0.01	mg/L
BNA	2,4-DIMETHYLPHENOL	8270C/D RV	GW,WW	24.2-128	20.5	0.01	mg/L
BNA	2,4-DICHLOROPHENOL	8270C/D RV	GW,WW	31.9-107	25.7	0.01	mg/L
BNA	2,4,6-TRICHLOROPHENOL	8270C/D RV	GW,WW	31.4-103	24.9	0.01	mg/L
BNA	2,4,5-TRICHLOROPHENOL	8270C/D RV	GW,WW	29.8-107	24.1	0.01	mg/L
BNA	2,3,4,6-TETRACHLOROPHENOL	8270C/D RV	GW,WW	34.9-112	23.9	0.001	mg/L
BNA	1-METHYLNAPHTHALENE	8270C/D RV	GW,WW	34.7-102	24.9	0.01	mg/L
BNA	1,4-DICHLORO BENZENE	8270C/D RV	GW,WW	21.0-89.4	32.6	0.01	mg/L
BNA	1,3-DICHLORO BENZENE	8270C/D RV	GW,WW	20.9-86.7	32.4	0.01	mg/L
BNA	1,2-DICHLORO BENZENE	8270C/D RV	GW,WW	23.7-91.9	31.9	0.01	mg/L
BNA	1,2,4-TRICHLORO BENZENE	8270C/D RV	GW,WW	22.9-96.1	27.5	0.01	mg/L
BNA	1,2,4,5-TETRACHLORO BENZENE	8270C/D RV	GW,WW	30.7-102	27.7	0.01	mg/L
BNA	SULFOLANE	8270C/D	GW, WW	70.0-130	20.0	0.2	ug/L
BNA	SULFOLANE	8270C/D	SS	70.0-130	20.0	0.33	ug/kg
Glycols	ETHYLENE GLYCOL	8015	SS	70.0-130	20.0	5.0	mg/L
Glycols	PROPYLENE GLYCOL	8015	SS	70.0-130	20.0	5.0	mg/L
Glycols	ETHYLENE GLYCOL	8015	GW,WW	70.0-130	20.0	5.0	mg/L

<b>Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs</b> <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL Unit	
Glycols	PROPYLENE GLYCOL	8015	GW,WW	70.0-130	20.0	5.0	mg/L
Explosives	1,3,5-TRINITROBENZENE	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	1,3-DINITROBENZENE	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	2,4,6-TRINITROTOLUENE	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	2,4-DINITROTOLUENE	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	2,6-DINITROTOLUENE	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	2-NITROTOLUENE	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	3-NITROTOLUENE	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	4-NITROTOLUENE (4-NT)	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	HEXAHYDRO-1,3,5-TRINITRO-1,3,5-TRIAZINE	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	METHYL-2,4,6-TRINITROPHENYLNITRAMINE	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	NITROBENZENE	8330A/B	SS	70.0-130	20.0	0.5	mg/Kg
Explosives	OCTAHYDRO - 1,3,5,7 - TETRANITRO-1,3,5,7-TETRAZOCINE (HMX)	8330A/B	SS	70.0-130	20.0	0.0005	mg/Kg
Explosives	PENTAERYTHRITOL TETRANITRATE (PETN)	8330A/B	SS	70.0-130	20.0	2	mg/Kg
Explosives	NITROGLYCERINE	8330A/B	SS	70.0-130	20.0	2	mg/Kg
Explosives	NITROGUANIDINE	8330A/B	SS	70.0-130	20.0	8	mg/Kg
Explosives	1,3,5-TRINITROBENZENE	8330A/B	GW	70.1-98.5	20.0	0.0005	mg/L
Explosives	1,3-DINITROBENZENE	8330A/B	GW	50.8-88.7	24.2	0.0005	mg/L
Explosives	2,4,6-TRINITROTOLUENE	8330A/B	GW	61.4-102	20.0	0.0005	mg/L
Explosives	2,4-DINITROTOLUENE	8330A/B	GW	40.2-91.7	36.2	0.0005	mg/L
Explosives	2,6-DINITROTOLUENE	8330A/B	GW	47.0-94.4	29.4	0.0005	mg/L
Explosives	2-NITROTOLUENE	8330A/B	GW	43.3-93.9	30.4	0.0005	mg/L
Explosives	3-NITROTOLUENE	8330A/B	GW	36.8-89.5	37.3	0.0005	mg/L
Explosives	4-NITROTOLUENE (4-NT)	8330A/B	GW	41.1-93.1	34.4	0.0005	mg/L
Explosives	HEXAHYDRO-1,3,5-TRINITRO-1,3,5-TRIAZINE	8330A/B	GW	63.1-94.2	20.0	0.0005	mg/L
Explosives	METHYL-2,4,6-TRINITROPHENYLNITRAMINE	8330A/B	GW	57.6-104	20.0	0.0005	mg/L
Explosives	NITROBENZENE	8330A/B	GW	56.0-99.0	20.4	0.0005	mg/L
Explosives	OCTAHYDRO - 1,3,5,7 - TETRANITRO-1,3,5,7-TETRAZOCINE (HMX)	8330A/B	GW	58.1-100	20.0	0.0005	mg/L
Explosives	PENTAERYTHRITOL TETRANITRATE (PETN)	8330A/B	GW	67.1-110	20.0	0.0005	mg/L
Explosives	NITROGLYCERINE	8330A/B	GW	65.0-126	20.0	0.0005	mg/L
GC	1, 2 DIBROMOETHANE (EDB)	504/8011	DW,GW, WW	70.0-130	30	0.00002	mg/L

<b>Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs</b> <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
GC	1, 2 DIBROMO-3-CHLOROPROPANE	504/8011	DW,GW, WW	70.0-130	30	0.00002	mg/L
GC	1,2,3-TRICHLOROPROPANE	504/8011	DW,GW, WW	70.0-130	30	0.0005	mg/L
THAA	BROMOACETIC ACID	552.2	DW	70.0-130	30	0.001	mg/L
THAA	CHLOROACETIC ACID	552.2	DW	70.0-130	30	0.002	mg/L
THAA	DIBROMOACETIC ACID	552.2	DW	70.0-130	30	0.001	mg/L
THAA	DICHLOROACETIC ACID	552.2	DW	70.0-130	30	0.001	mg/L
THAA	TRICHLOROACETIC ACID	552.2	DW	70.0-130	30	0.001	mg/L
TPH	PETROLEUM RANGE ORGANICS (TRPH)	FL-PRO RV	GW,	50.0-150	20.0	0.1	mg/L
TPH	PETROLEUM RANGE ORGANICS (TRPH)	FL-PRO	SS	50.0-150	20.0	4.0	mg/Kg
TPH	PETROLEUM RANGE ORGANICS (TRPH)	EPH TN	GW	50.0-150	20.0	0.1	mg/L
TPH	PETROLEUM RANGE ORGANICS (TRPH)	EPH TN	SS	50.0-150	20.0	4.0	mg/Kg
TPH	PETROLEUM RANGE ORGANICS (TRPH) - C9-C18, C19-C36, C11-C22	MADEP EPH	GW, WW	50.0-150	20.0	0.1	mg/L
TPH	PETROLEUM RANGE ORGANICS (TRPH) - C9-C18, C19-C36, C11-C22	MADEP EPH	SS	50.0-150	20.0	5.5	mg/Kg
TPH	PETROLEUM RANGE ORGANICS (TRPH) - C10-C28	DRO, 8015Mod	GW, WW	50.0-150	20.0	0.1	mg/L
TPH	PETROLEUM RANGE ORGANICS (TRPH) - C10-C28	DRO, 8015Mod	SS	50.0-150	20.0	4.0	mg/Kg
TPH	PETROLEUM RANGE ORGANICS (TRPH) – C10-C20, C20-C34	OHIO DRO	GW, WW	50.0-150	20.0	0.1	mg/L
TPH	PETROLEUM RANGE ORGANICS (TRPH) – C10-C20, C20-C34	OHIO DRO	SS	50.0-150	20.0	4.0	mg/Kg
TPH	PETROLEUM RANGE ORGANICS (TRPH) – GAS, DIESEL, MOTOR OIL, ETC.	OA2	GW, WW	50.0-150	20.0	0.1	mg/L
TPH	PETROLEUM RANGE ORGANICS (TRPH) – GAS, DIESEL, MOTOR OIL, ETC.	OA2	SS	50.0-150	20.0	4.0	mg/Kg
TPH	PETROLEUM RANGE ORGANICS - C10-C28, C28-C40	DRORLA	GW, WW	50.0-150	20.0	0.1	mg/L
TPH	PETROLEUM RANGE ORGANICS - C10-C28, C28-C40	DRORLA	SS	50.0-150	20.0	4.0	mg/Kg
TPH	PETROLEUM RANGE ORGANICS – C10-C32	DROWY	GW, WW	50.0-150	20.0	0.1	mg/L
TPH	PETROLEUM RANGE ORGANICS – C10-C32	DROWY	SS	50.0-150	20.0	4.0	mg/Kg

<b>Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs</b> <i>This table is subject to revision without notice</i>							
Class An	alyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
TPH	PETROLEUM RANGE ORGANICS – GAS, DIESEL, MOTOR OIL, ETC.	NWTPH-Dx	GW, WW	50.0-150	20.0	0.25	mg/L
TPH	PETROLEUM RANGE ORGANICS – GAS, DIESEL, MOTOR OIL, ETC.	NWTPH-Dx	SS	50.0-150	20.0	25	mg/Kg
TPH	PETROLEUM RANGE ORGANICS – C10-C28	DROWM	GW, WW	75.0-115	20.0	0.1	mg/L
TPH	PETROLEUM RANGE ORGANICS – C10-C28	DROWM	SS	70.0-120	20.0	10	mg/Kg
TPH	PETROLEUM RANGE ORGANICS – C10-C22	TPHAZ	SS	70.0-130	20.0	30	mg/Kg
TPH	PETROLEUM RANGE ORGANICS – C22-C32	TPHAZ	SS	70.0-130	20.0	100.	mg/Kg
TPH	PETROLEUM RANGE ORGANICS – C10-C32	TPHAZ	SS	70.0-130	20.0	130.	mg/Kg
TPH	PETROLEUM RANGE ORGANICS - C6-C12, C12-C28, C28-C35, C6-C35	TX TPH	SS	75.0-125	20.0	50	mg/Kg
TPH	PETROLEUM RANGE ORGANICS - C10-C21, C21-C35	DROMO	GW, WW	75.0-125	20.0	1.0	mg/L
TPH	PETROLEUM RANGE ORGANICS - C10-C21, C21-C35	DROMO	SS	75.0-125	20.0	20	mg/Kg

### 13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CAR is kept on file by the Regulatory Affairs Department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

#### 13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these limits are exceeded, corrective action must be taken. Calculated sample spike control limits are also used.

All samples and procedures are governed by ESCs quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP.



### 13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria takes precedence.

### 13.2.2 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

Rejection Criteria - Blank reading is more than twice the background absorbance or more than RL.

Corrective Action - Blanks are re-analyzed and the response is assessed. Standard curves and samples are evaluated for any obvious contamination that is isolated or uniform throughout the run. If necessary, reagents are re-prepared. Analyses are not initiated until the problem is identified and solved. If samples have already been prepared or analyzed, the Senior Chemist and/or Department Supervisor are consulted to determine if data needs to be rejected or if samples need to be re-prepared.

### 13.2.3 Out Of Control Laboratory Control Standards (LCS & LCSD)

Rejection Criteria - If the performance is outside of lab-generated control limits which are calculated as the mean of at least 20 data points  $\pm 3$  times the standard deviation of those points (Listed in Section 12) and the marginal exceedance allowance is surpassed (see section 12.2).

Corrective Action - Instrument settings are checked and the LCS standard is reanalyzed. If the LCS is still out of control, instrumentation is checked for systemic problems and repaired (if necessary). Re-calibration is performed and the samples affected since the last in control reference standard are rerun. The Senior Chemist and/or Department Supervisor are consulted for further action.

### 13.2.4 Out Of Control Matrix Spike Samples

Rejection Criteria - If either the MS or MSD sample is outside the established control limits.

Corrective Action - Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance, however, is based on method blank and LCS performance, not on MS/MSD recoveries. Specific methods, customers, and programs may require further corrective action in some cases.

### 13.2.5 Out Of Control Duplicate Samples

Rejection Criteria - Lab-generated maximum RPD limit (as listed under precision in Section 12)

Corrective Action - Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance, however, is based on method blank and LCS performance. Specific methods, customers, and programs may require further corrective action in some cases.

### 13.2.7 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

Corrective Action - Instrument settings are checked, calibration verification standard is reanalyzed. If the standard is still out of control, recalibration is performed, and samples affected since the last in control reference standard are rerun. The the Senior Chemist and/or Department Supervisor are consulted for further action.

## 14.0 RECORD KEEPING

Record keeping is outlined in SOP #030230, *Standards Logger*, SOP #030227, *Data Review* and SOP #030201, *Data Handling and Reporting*. Semi-Volatile organics calibration data are recorded and integrated using HP Enviroquant software. Calibration data from the semi-volatile analyses, in addition to the initial and daily calibration, includes GC/MS autotunes, DFTPP reports and surrogate recovery reports. Hard copy records of initial calibration and daily calibration are stored with chromatograms and integrated with sample data by date analyzed.

## 15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 12.0 and SOP #010104, *Internal Audits*.

## 16.0 REVISIONS

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of this appendix are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0 (Appendix VII)	Section 5.1 – Removed language about supervisor and backup reviewing and approving all data Section 8.1 – Updated equipment list Table 8.3B – Added working standards for reduced volume FL PRO Section 8.4 – Clarified 8141/1657 CCV frequency is every 20 samples for external calibrations and every 12h for internal calibration Table 8.5 – Added every 20 samples for CCV frequency for DRO for those methods that allow

Document	Revision
	this frequency Section 9.1 – Clarified what system is used to generate the reagent water Section 13.2.4 – Reworded MS/MSD criteria and revised corrective action to just qualify unless method, customer, or program states to do something else. Section 13.2.5 - Revised duplicate corrective action to just qualify unless method, customer, or program states to do something else. Section 16 – New section for summary of revisions to previous version

1.0 SIGNATORY APPROVALS

# Air Laboratory QUALITY ASSURANCE MANUAL

## APPENDIX VIII TO THE ESC QUALITY ASSURANCE MANUAL

for


ESC LAB SCIENCES  
12065 LEBANON ROAD  
MT. JULIET, TENNESSEE 37122  
(615) 758-5858


Prepared by

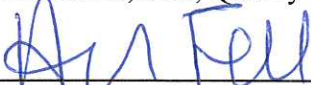
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**NOTE: The QAM has been approved by the following people.**

  
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### **3.0 SCOPE AND APPLICATION**

This appendix discusses specific QA requirements for general analytical protocols to ensure that analytical data generated from the Air Laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

### **4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES**

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling guidance and equipment, and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual*.

### **5.0 PERSONNEL AND TRAINING**

#### **5.1 PERSONNEL**

Heidi Ferrell, with a B.S. degree in Chemistry, is the Department Supervisor and is responsible for the overall production of the Air Laboratory; including the management of the staff and scheduling. Ms. Ferrell has 9 years of environmental laboratory experience.

In her absence, Matt Ferrell, with an A.S. of Applied Science, assumes responsibility for the Air Department decisions. Mr. Ferrell is the Primary Analyst for the Air Laboratory and is proficient in air analytical methods. He has 5 years of environmental laboratory experience.

#### **5.2 TRAINING**

The Supervisor trains new laboratory analysts according to ESC protocol. ESC's training program is outlined in *SOP 030205 Technical Training and Personnel Qualifications*. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in Air Laboratory is also demonstrated by acceptable participation in the Phenova proficiency testing program (PTs). Documentation of analyst training is maintained on file within the department.

## 6.0 FACILITIES AND LABORATORY SAFETY

### 6.1 FACILITIES

The main area of the laboratory has approximately 670 square feet of area with roughly 150 square feet of bench area. There are 670 square feet of additional storage and the lighting is fluorescence. The air system is a ten-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the US Filter deionizer system. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's hazardous waste disposal company. ESC's building information guides and site plan are shown in Appendix I.

### 6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods.

ESC's laboratory safety guidelines are detailed in the *ESC Chemical Hygiene Plan*.

## 7.0 SAMPLING PROCEDURES

### 7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedures are described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Samples for air analysis are collected in four ways:
  - Samples may be collected directly in evacuated Summa canisters fit with the appropriately adjusted regulators that control sampling flow to fill the canister over a given time period.
  - Summa canisters may also be collected as "grab" samples by simply opening the evacuated canister without the aid of a flow regulator and allowing the canister to fill quickly by virtue of the canister vacuum.
  - The third method entails collection of field samples using various sized bags specifically designed for air sampling (i.e. Tedlar). This type of sampling allows a pump connected via tubing to the bag's intake valve to sample the air at a controlled flow and over the appropriate timeframe needed by the client.
  - The headspace of containers housing water samples may also be analyzed for specific volatile components.

- Air samples taken in summa canisters should be shipped in bubble wrapped boxes. Tedlar bags and water samples can be shipped in a container or cooler that is sufficiently rigid and protects the samples from damage that may be incurred in transport. The chain of custody is also placed in the container. The shipping label containing the name and address of the shipper is affixed to the outside of the shipment container.
- Samples are received in the laboratory login area and are tracked using LIMS (Laboratory Information Management System). A Chain of Custody Form accompanies all samples received by the lab. This is necessary to prove the traceability of the samples and to document the change in possession from sampling to delivery to receipt by the laboratory. Prior to analysis samples are checked for integrity. Sample handling, tracking and acceptance procedures are outlined in *SOP #060105, Sample Receiving*.

## 8.0 EQUIPMENT

### 8.1 EQUIPMENT LIST

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Air Analysis</b>						
<i>This table is subject to revision without notice</i>						
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Instrument Name</i>	<i>#</i>	<i>Serial #</i>	<i>Location</i>
Gas Chromatograph	HP	6890N TCD	AIRGC3	1	US10726007	Air Lab
Gas Chromatograph/Mass Spectrometer	HP	6890 GC/5973MSD	AIRMS1	1	GCUS00024616 MSUS63810244	Air Lab
Gas Chromatograph/Mass Spectrometer	Agilent	6890N/5975	AIRMS2	2	CN10551083	Air Lab
Gas Chromatograph/Mass Spectrometer	Agilent	6890/5973	AIRMS3	3	US000011333 US91911078	Air Lab
Gas Chromatograph/Mass Spectrometer	Agilent	6890/5973	AIRMS4	4	US00024695 US82311265	Air Lab
Gas Chromatograph/Mass Spectrometer	Agilent	6890/5973	AIRMS5	5	GCUS0003961 MSUS0340681	Air Lab
Canister Autosampler	Entech	7016C			0203	Air Lab
Preconcentrator	Entech	7100A			1089	Air Lab
Preconcentrator	Entech	7200			1005	Air Lab
Canister Autosampler	Entech	7016CA			1039	Air Lab
Tedlar Autosampler	Entech	7032A-L			1019	Air Lab
Dynamic Diluter	Entech	Model 4600A			1086	Air Lab
Canister Cleaner	Entech	Model 3100A			1045	Air Lab
Canister Cleaner	Entech	Model 3100A			1178	Air Lab
Canister cleaner	Entech	Model 3100A			B33-02663	Air Lab
Preconcentrator	Entech	7100A			1137	Air Lab
Canister Autosampler	Entech	7016D			1422	Air Lab
Preconcentrator	Entech	7200			1162	Air Lab
Tedlar Autosampler	Entech	7032A			1044	Air Lab
Canister Autosampler	Entech	7016CA			1137	Air Lab
Tedlar Autosampler	Entech	7032A-L			1017	Air Lab



<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Air Analysis</b>						
<i>This table is subject to revision without notice</i>						
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Instrument Name</i>	<i>#</i>	<i>Serial #</i>	<i>Location</i>
GC/FID	Agilent	6890N	AIRGC2	2	US10137006	Air Lab
Headspace Autosampler	EST/PTS	LGX50			LGX108062315	Air Lab
Headspace Autosampler	EST/PTS	LGX50			LGX109062315	Air Lab
TO Canister	Restek/Entech	TO-Can/ SiloniteCan	1800 cans owned		N/A	Air Lab
Passive Sampling Kit	Restek		1100 owned		N/A	Air Lab
Field hand held PID	RAE Systems	MiniRae2000			110-012980	Air Lab
Field hand held PID	RAE Systems	MiniRAE2000				Air Lab

### 8.2 E EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

<i>INSTRUMENT</i>	<i>P. M. DESCRIPTION</i>	<i>FREQUENCY</i>
Gas Chromatograph Detectors: FID	Change Quartz jet; clean; replace flame tip	As needed - when deterioration is noticeable
Gas Chromatograph/Mass Spectrometer	•Autotune Report	Inspected daily
Gas Chromatograph/Mass Spectrometer	•Clean ion source	As needed to maintain high mass resolution
Gas Chromatograph/Mass Spectrometer	•Replace vacuum pump oil	Every 6 months
Gas Chromatographs	•Replace column	When separation begins to degrade

### 8.3 S STANDARDS AND REAGENTS

<b>Table 8.3A: Standard stock sources, description and calibration information.</b>					
<i>This table is subject to revision without notice</i>					
<b>Method Vend</b>	<b>or</b>	<b>Description</b>	<b>Conc.</b>	<b>Storage Req.</b>	<b>Expiration</b>
TO-15/8260B (VAP)/Method 8-mod. ISTD Stock Standard	Spectra Gases	ISTD and Tuning Mixture	1 ppmv	3395 L (2A) cylinder	1 year
TO-15/8260B (VAP)/Method 18-mod. Stock Standard*	Spectra Gases	Target Analytes except Bromoform at 3 ppmv, m&p Xylene at 2 ppmv and GRO at 40 ppmv	100 ppbv	3395 L (2A) cylinder	1 year
TO-15/8260B (VAP)/Method 18-mod. Laboratory Control Stock Standard*	Spectra Gases	Target Analytes – Second Source	100 ppbv	3395 L (2A) cylinder	1 year
Landfill Gases Stock (CO <sub>2</sub> , CO, CH <sub>4</sub> , O <sub>2</sub> , He)	Spectra Gases	Target Analytes	3 Levels	3395 L (2A) cylinder	1 year
Landfill Gases Laboratory Control Stock Standard	Spectra Gases	Target Analytes – Second Source	20%	3395 L (2A) cylinder	1 year
RSK-175 (Methane, Ethane, Ethene, Propane, Acetylene) Stock Standard	Scotty Gases	Target Analytes	1000 ppmv	3395 L (2A) cylinder	1 year
RSK-175 Laboratory Control Stock Standard	Scotty Gases	Target Analytes – Second Source	1000 ppmv	3395 L (2A) cylinder	1 year

**TABLE 8.3B: Intermediate/Working Standard Concentrations**  
*This table is subject to revision without notice*

Organic Compounds	Method #	Working Standard Concentrations	Volume of Stock Used	Final Volume	Expiration
ISTD and Tuning Intermediate Standard	TO-15/8260B (VAP)/Method 18.	20 ppbv	900 cc	45L in 15L Canister	1 year
Target Analytes* Intermediate Standard	TO-15/8260B (VAP)/Method 18	5 ppbv except Bromoform at 5ppbv, m&p Xylene at 10 ppbv and GRO at 200 ppbv	225 cc	45L in 15L Canister	1 year
TO-15/ 8260B(VAP)/ Method 18-mod. Laboratory Control* Intermediate Standard	TO-15/8260B (VAP)/Method 18	Second Source: 5 ppbv except Bromoform at 15ppbv, m&p Xylene at 10 ppbv and GRO at 200	225 cc	45L in 15L Canister	1 year

\* see analytes listed in Table 12.3.

#### 8.4 I INSTRUMENT CALIBRATION

##### **TO-15, 8260B (Ohio VAP Air), Gasoline Range Components (Method 18) – Volatiles in Air by GC/MS – SOP Numbers 330367, 330368, & 330369**

Detector mass calibration is performed daily using the autotune function of the GC/MS analytical system and BFB (Bromofluorobenzene). Following verification of the appropriate masses, the instrument sensitivity is verified by injecting a tuning solution containing Bromofluorobenzene (BFB). The BFB must meet the following ion abundance criteria:

Mass	Ion Abundance Criteria
50	8.0-40.0% of mass 95
75	30.0-66.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	50.0-120% of mass 95
175	4.0-9.0% of mass 174
176	> 93.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

Successful tuning must occur every 24 hours for method TO-15 and Method 18 and every 12 hours for method 8260B (OH VAP only).

Following successful tuning, the GC/MS is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of five standards. The calibration standards are tabulated according to peak height or area against concentrations of the target analytes and the concentrations and responses of the internal standard analytes. The results are used to determine a response factor for each analyte in each standard injected.

A TO-15 or Method 18 calibration curve is constructed and determined to be acceptable if each analyte is found to be constant over the working range (<30 % RSD with no more than 2 compounds being between 30 and 40 % RSD). When this condition is met, linearity through the origin can be assumed and the average RF can be used in place of a calibration curve.

When analyzing air by method 8260B, specific target analytes in the calibration standards are defined as calibration check compounds (CCCs) or system performance check compounds (SPCCs).

<b>SPCCs:</b>	
Analyte	Minimum Relative Response Factor
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

<b>CCCs:</b>	
1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl Chloride

Analytes identified by the method as SPCCs must meet the minimum average response factors listed above for successful initial calibration. Compounds identified as CCCs must have a %RSD of less than 30% in the initial calibration curve. The remaining target analytes in the calibration standards must be <15% RSD. Linear regression can be used for any target compound exceeding the 15% RSD criteria providing that the correlation coefficient is 0.990 or better. Initial 8260B calibration for the target analytes of interest for the client project that do not meet these requirements are not accepted and re-calibration must be performed.

For all methods, the initial calibration range must represent the typical air sample and include the lowest standard at or below the RL. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range. Following successful calibration, the analysis of field and QC samples may begin. Analysis may be performed only during the timeframe of a valid tuning cycle (12 hours for 8260B and 24 hours for TO-15 and Method 18). Following the expiration of the tuning clock, the instrument must be re-tuned and either recalibrated or the existing calibration may be verified prior to further sample analysis.

For 8260B analyses, daily continuing calibration verification (CCV) includes successful demonstration of BFB sensitivity and the injection of a mid-level CCV standard containing all the target analytes of interest, the CCC, and SPCC compounds. The BFB tune must meet the ion abundance criteria (see table above). Each SPCC in the calibration verification standard must meet a minimum response factors listed above. The CCCs must achieve the criteria of +/- 20% RSD. Each internal standard in the CCV must recover between -50% to + 100%, when compared to the same internal standard compound in the mid-point standard of the initial calibration curve. Additionally, if the retention time of an internal standard changes by more than 30 seconds from the retention time of the same internal standard in the mid-level standard of the most recent initial calibration, the system must be evaluated, corrected, and possibly re-calibrated.

For TO-15 and Method 18 analyses, daily calibration verification is accomplished by a successful demonstration of BFB sensitivity and the injection of a mid-level CCV standard containing all the target analytes of interest. The BFB tune must meet the same ion abundance criteria as previously listed and the CCV standard must recover within 30% of predicted response for all analytes of interest.

**Fixed Gases (Carbon Dioxide, Carbon Monoxide, Methane, Oxygen) based on ASTM D1946 – SOP Number 330372**

Optimize the conditions of the Gas Chromatograph with Thermal Conductivity Detection according to the manufacturer's specification to provide good resolution and sensitivity. Verify that the gas flows and column and detector temperatures are at optimum levels for analysis, based on peak resolution and chromatograph performance. Allow sufficient time between each temperature adjustment to attain a stable reading (typically one hour). Standards are injected at a minimum of five concentration levels from purchased certified standards. Generation of the initial calibration is performed using PC-based D.01 ChemStation software and a calibration factor or linear regression model. The calibration must meet 15% RSD for calibration factors or a correlation coefficient of at least 0.990. Instrument calibration must be verified initially on days when a full calibration curve is not analyzed, following every 10 injections during the analytical sequence, and at the end of each sequence by the analysis of a check standard. These standards must recover within 15% of the expected concentration.

**Methane, Ethane, Ethene, Propane, Acetylene based on RSK-175 – SOP Number 330370**

Optimize the conditions of the Gas Chromatograph with Thermal Conductivity Detection according to the manufacturer's specification to provide good resolution and sensitivity. Verify that the gas flows and column and detector temperatures are at optimum levels for analysis, based on peak resolution and chromatograph performance. Allow sufficient time between each temperature adjustment to attain a stable reading (typically one hour). Standards are injected at a minimum of five concentration levels. The target analytes in the calibration standards must be  $\leq 15\%$  RSD. Linear regression can be used for any

target compound exceeding the 15% RSD criteria providing that the correlation coefficient is 0.990 or better. Headspace is created in each field sample by forcing 20cc of helium into each sample vial. Following sufficient time for the sample and headspace to reach equilibrium, 100uL of air is removed from each vial and injected into the GC. Instrument calibration must be verified initially on days when a full calibration curve is not analyzed, following every 10 injections during the analytical sequence, and at the end of each sequence by the analysis of a check standard. These standards must recover within 15% of the expected concentration.

**Methanol and Ethanol (MEETAC) in soil and water samples based on EPA 8260B/C – SOP Number 330373**

Detector mass calibration is performed daily using the autotune function of the GC/MS analytical system and BFB (Bromofluorobenzene). Following verification of the appropriate masses, the instrument sensitivity is verified by injecting a tuning solution containing Bromofluorobenzene (BFB). The BFB must meet the following ion abundance criteria:

Mass	Ion Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

Successful tuning must occur every 12 hours.

Following successful tuning, the GC/MS is calibrated using the external standard procedure. A standard curve is prepared using a minimum of five standards. The calibration standards are tabulated according to peak height or area against concentrations of the target analytes. The results are used to determine a response factor for each analyte in each standard injected. A calibration curve is constructed and determined to be acceptable if each analyte is found to be constant over the working range (<15 % RSD). When this condition is met, linearity through the origin can be assumed and the average CF can be used in place of a calibration curve. Linear regression can be used for any target compound exceeding the 15% RSD criteria providing that the correlation coefficient is 0.990 or better.

The initial calibration range must represent the typical field sample and include the lowest standard at or below the RL. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range. Following successful calibration, the analysis of field and QC samples may begin. Analysis may be performed only during the timeframe of a valid tuning cycle (12 hours). Following the expiration of the tuning clock, the instrument must be re-tuned and either recalibrated or the existing calibration may be verified prior to further sample analysis.

Daily calibration verification is accomplished by a successful demonstration of BFB sensitivity and the injection of a mid-level CCV standard containing all the target analytes of interest. The BFB tune must meet the same ion abundance criteria as previously listed and the CCV standard must recover within 15% of predicted response for all analytes of interest.

### 8.5 A ACCEPTANCE/REJECTION OF CALIBRATION

The initial calibration curve is compared with previous curves for the same analyte. All new standard curves are immediately checked with a secondary source or laboratory control standard prepared from a separate source than those used for calibration. All curves are visually reviewed to ensure that acceptable correlation represents linearity. Calibration curves may be rejected for nonlinearity, abnormal sensitivity, or poor response of the laboratory control standard.

Continuing calibration verification is performed on each day that initial calibration is not performed and following every tenth sample. If a check standard does not perform within established criteria then the instrument will undergo evaluation to determine the problem. Once the problem is corrected, all samples between the last in control sample and the first out of control check will be re-analyzed.

**TABLE 8.5: INSTRUMENT CALIBRATION & QC**

Analysis/ Instrument	Calibration Type	Number of Standards	Acceptance/ Rejection Criteria	Frequency	
TO-15 & Method 18/ GC/MS	Initial/ Continuing	1 - Tuning Solution	<u>Mass</u> <u>m/z Abundance Criteria</u>	TO-15/ M-18: Every 24 hours  8260 VAP: Every 12 hours	
			50		8-40% of mass 95
			75		30-66% of mass 95
			95		Base peak, 100%
			96		5-9% of mass 95
			173		<2% of mass 174
			174		50-120% of mass 95
			175		4-9% of mass 174
			176		>93% but <101% of mass 174
177	5-9% of mass 176				

**TABLE 8.5: INSTRUMENT CALIBRATION & QC**

Analysis/ Instrument	Calibration Type	Number of Standards	Acceptance/ Rejection Criteria	Frequency
TO-15 & Method 18/ GC/MS	Initial	5 minimum	Average Response Factor: <30 % RSD with no more than 2 compounds being between 30 and 40 % RSD	As needed
8260B VAP/ GC/MS	Initial	5 minimum	Average Response Factor: Target analytes in the calibration standards must be <15% RSD, CCCs must have a %RSD of less than 30% & SPCCs must meet the minimum average response factors. Linear regression can be used for any target compound exceeding the 15% RSD	As needed
TO-15 & Method 18/ GC/MS	Continuing	1 cal. check verification (CCV)	Percent Difference for all compounds <30%	Daily, when init. calibration is not required.
TO-15 VAP/ GC/MS	Continuing	1 cal. check verification (CCV)	Average Response Factor: Target analytes in the calibration standards must be <15% RSD, CCCs must have a %RSD of less than 20% & SPCCs must meet the minimum average response factors.	Daily, when init. calibration is not required.
TO-15 & Method 18	Initial/ Continuing	1 - Blank	< ½ RL, concentrations of common laboratory contaminants shall not exceed the reporting limit	Following init. calibration or daily cal. verification
TO-15 & Method 18	Initial/ Continuing	2 – Second source (LCS/LCSD)	Must be within +/-30% with an RPD of <25.	Following initial calibration or daily cal. Verification
Landfill Gas	Initial	3	Average Response Factor: Target analytes in the calibration standards must be <15% RSD. Linear regression can be used for any target compound exceeding the 15% RSD	As needed
Landfill Gas	Continuing	1 - cal. check verification (CCV)	Target analytes in the calibration standards must be <15% RSD.	Daily, when init. calibration is not required, following every 10 <sup>th</sup> injection, and the end of the sequence.
Landfill Gas	Initial/ Continuing	1 - Blank	< ½ RL, concentrations of common laboratory contaminants shall not exceed the reporting limit	Following init. calibration or daily cal. verification
Landfill Gas	Initial/ Continuing	2 – Second source (LCS/LCSD)	Must be within +/-30% with an RPD of <25.	Following initial calibration or daily cal. verification
RSK-175	Initial	3	Average Response Factor: Target analytes in the calibration standards must be <15% RSD. Linear regression can be used for any target compound exceeding the 15% RSD	As needed

**TABLE 8.5: INSTRUMENT CALIBRATION & QC**

Analysis/ Instrument	Calibration Type	Number of Standards	Acceptance/ Rejection Criteria	Frequency
RSK-175	Continuing	1 - cal. check verification (CCV)	Target analytes in the calibration standards must be <15% RSD.	Daily, when init. calibration is not required, following every 10 <sup>th</sup> injection, and the end of the sequence.
RSK-175	Initial/ Continuing	1 - Blank	< ½ RL, concentrations of common laboratory contaminants shall not exceed the reporting limit	Following init. calibration or daily cal. verification
RSK-175	Initial/ Continuing	2 – Second source (LCS/LCSD)	Must be within +/-30% with an RPD of <25.	Following initial calibration or daily cal. verification
MEETAC	Initial/ Continuing	1 - Tuning Solution	<u>Mass m/z Abundance Criteria</u> 50 15.0-40.0% of mass 95 75 30.0-60.0% of mass 95 95 base peak, 100% relative abundance 96 5.0-9.0% of mass 95 173 < 2.0% of mass 174 174 > 50.0% of mass 95 175 5.0-9.0% of mass 174 176 > 95.0%, but less than 101% of mass 174 177 5.0-9.0% of mass 176	Every 12 hours
MEETAC	Initial	5 minimum	Average Response Factor: Target analytes in the calibration standards must be <15% RSD,	As needed
MEETAC	Continuing	1 cal. check verification (CCV)	Average Response Factor: Target analytes in the calibration standards must be <15% RSD,	Daily, when init. calibration is not required.
MEETAC	Initial/ Continuing	1 - Blank	< ½ RL, concentrations of common laboratory contaminants shall not exceed the reporting limit	Following init. calibration or daily cal. verification

## 9.0 LABORATORY PRACTICES

### 9.1 R REAGENT GRADE WATER

Reagent Grade water –Type II used in the Air Laboratory is generated in the Microbiology Laboratory and is periodically checked for contamination. Type II water is checked annually for single and total heavy metals. Monthly checks for total organic carbon, ammonia and organic nitrogen, total residual chlorine and a heterotrophic plate count are also conducted. Conductivity and pH are checked continuously or with each use.



## 9.2 S AMPLER CLEANING AND CERTIFICATION PROCEDURE

Canisters are cleaned in the laboratory using the Entech 3100 4-Position Canister Cleaner. Canisters are cleaned in batches of 4 to 8 per cleaning cycle. Prior to cleaning, canisters are inspected for integrity, damage and visible contamination. Acceptable canisters are connected to the manifold on the Entech cleaner and the cleaning cycle is controlled using Entech SmartLab software. Programmable cleaning cycles include: light, medium and heavy-duty and the cycle selected depends on the previous use of the dirtiest canister being cleaned. The cleaner automatically performs a leak check for the canisters and the manifold prior to the initial evacuation cycle. Heating bands are placed on each canister to elevate the temperature of the metallic canister to a level that provides for efficient cleaning. The typical cleaning cycle parameters are:

	Operating temperature = 120°C
1	Initial evacuation of canister to 1000 mtorr
2	Refill canister to 20psi
3	Evacuate the canister to 1000 mtorr
4	Repeat items 2 & 3 for 8 total cycles
5	Final zero air pressure in clean canister is 50 mtorr.

Following cleaning, a single canister is selected as a QC sample for the entire batch and the sample is filled with zero air or nitrogen and analyzed to verify that successful cleaning has occurred. If the analysis indicates that the batch is clean (i.e. <0.2 ppbv for target analytes and free of additional contamination), the QC sample is returned to the cleaner manifold. The entire batch is evacuated to less than 50 mtorr and clearly labeled as clean and ready for sample collection. If the QC sample indicates that canister contamination is still present, the batch is recycled through the cleaning process until residual contamination is no longer present. If following repeated cleaning cycles, residual contamination is still observed, canisters may be permanently removed from service and clearly identified as unusable.

Tedlar bags and vials, as used for headspace analyses, are purchased as certified pre-cleaned from approved providers and disposed of following the sample retention period.

## 9.3 TYPICAL ENTECH AUTOSAMPLER OPERATING PARAMETERS

These parameters are provided as an example and may be modified to improve analytical system performance or better address project needs.

Line Temp = 100°C	Module 2 Desorb = 180°C
Bulk Head 1 = 30°C	Module 2 Bake = 190°C
Bulk Head 2 = 30°C	Module 2 Desorb Time = 3.5 min
Module 1 Trap = -150°C	Module 3 Trap = -180°C
Module 1 Preheat = 20°C	Module 3 Inject = 2 min
Module 1 Desorb = 20°C	Module 3 Bake Time = 2 min

Module 1 Bake = 130°C	Module 3 Event = 3
Module 1 Bake Time = 5 min	Module 3 Wait Time = 25 min.
Module 2 Trap = -30°C	Pressure Comp Factor = 14
Module 2 Preheat = off	Loop Flush = 30 seconds

## 10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOPs associated with the air laboratory can be found in the following table:

**TABLE 10.1: AIR DEPARTMENT SOPs**

*This Table is subject to revision without notice*

SOP #	Title/Description
330367	Measurement of Volatile Organic Compound in Ambient Air by GC/MS (EPA TO-15)
330367OH	Measurement of Volatile Organic Compound in Ambient Air by GC/MS (EPA TO-15) (Ohio VAP only)
330368	Gasoline Range Organics in Ambient Air by GC/MS – Method 18 Modified
330369	Volatile Organic Compounds in Air by GC/MS 8260B for the Ohio VAP Program (with provisions for GRO determination based on 8015B)
330370	Method for Determination of Methane, Ethane, and Ethene (Based on RSK-175)
330371	Canister Cleaning, Certification and Storage
330372	The Analysis of Fixed Gases using GC/TCD
330373	Meetac – Methanol and Ethanol Based on EPA 8260B/C

10.2 Sample Dilutions:

Dilutions for air samples from summa canisters and Tedlar bags may take three forms depending on the level of dilution required. These dilution techniques are demonstrated below:

### Autosampler Dilution:

- First, a smaller sample volume can be analyzed using the capabilities of the Entech autosampler. For example, for a standard sample volume of 400cc, if 40cc were analyzed, that would be equivalent to a 10-fold dilution.
- The smallest sample volume that can be accurately analyzed using the autosampler method is 10cc (or a 40x).

### Pressurized Manual Dilution:

- Sometimes, a 40X dilution is not sufficient to bring the concentration of a target analyte within the calibration range. In those cases, the sample canister is pressurized resulting in a dilution of the target analytes present.
- The act of introducing more pure air into the canister performs a dilution.
- The canister can then be analyzed at 400cc or diluted using a lesser autosampler volume, if necessary.

**Secondary Manual Dilution:**

- In extreme cases, the canister may need to be diluted into a second evacuated canister.
- This is accomplished by using a gas tight syringe to remove an aliquot of sample (1-10mL) from the initial canister then injecting it into a clean evacuated second canister.
- The second canister is then analyzed and quantified taking into account the dilution based on the amount of sample injected and the total volume of the canister utilized.

**Tedlar Bag Dilutions:**

- Dilutions on Tedlar bags can be performed in much the same manner as summa canisters using either the autosampler dilution or the secondary manual dilution using a second Tedlar bag and filling it with pure air then adding an aliquot of field sample using a gas tight syringe.

## 11.0 QUALITY CONTROL CHECKS

**NOTE:** For specific guidance on each determinative method, including required quality control and specific state requirements/modifications, refer to the relevant laboratory standard operating procedure(s).

- 11.1 Initial Demonstrations of Capability (IDOCs) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability (CDOCs) must be updated at least annually for each analyst performing testing on field samples. The associated data is filed within the department and available for review.
- 11.2 A Laboratory Control Sample (LCS) and LCS Duplicate are analyzed per batch of samples and must yield recoveries within 70-130% of the expected concentration for all analytes and this pair must not exceed an RPD of 25%. Analytes specifically listed in each SOP as poor performers must yield recoveries as listed in each determinative SOP. LCS stock standards are prepared from sources independent of the calibration standards and also serve to verify the original calibration curve.
- 11.3 A method preparation blank is performed per batch of samples processed. If the acceptance criteria as listed in the determinative SOP is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary, based on the following criteria:
- The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch or
  - The blank contamination is greater than 1/10 of the specified regulatory limit. The concentrations of common laboratory contaminants shall not exceed the reporting limit.

Any samples associated with a blank that fail these criteria shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in non-detected results for the failing analytes.

## 12.0 DATA REDUCTION, VALIDATION AND REPORTING

### 12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in SOP #030201, *Data Handling and Reporting*. A secondary review of the data package is performed according to ESC SOP #030227, *Data Review*. The reviewer verifies that the analysis has been performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required qualifiers on test reports, etc.)

**TABLE 12.1 Data Reduction Formulas**

PARAMETER	FORMULA
GC/MS – Analyte Response Factor	$\frac{\text{response of analyte primary ion } \{area\} \times \text{concentration of analyte (ug/L)}}{\text{response of ISTD primary ion } \{area\} \times \text{concentration of ISTD (ug/L)}}$ <p style="text-align: center;"><i>Calculations performed by HP Enviroquant Software</i></p>
GC/MS – Sample Analyte Concentration	$\frac{\text{response of primary ion in analyte} \times \text{int. std concentration. } \{ppbv\} \times \text{dilution factor}}{\text{response factor } \{area/(mg/ml)\} \times \text{initial volume-mass } \{ml \text{ or } g\} \times \text{int. std cal. } \{area\}}$ <p style="text-align: center;"><i>Calculations performed by HP Enviroquant Software</i></p>

### 12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.3 by method for current QC targets and controls and current reporting limits.

**Organic Control Limits** - The organic QC targets are statutory in nature; warning and control limits for organic analyses are initially established for groups of compounds based on preliminary method validation data. When additional data becomes available, the QC targets are reviewed. All QC targets are routinely re-evaluated at least annually (and updated, if necessary) against laboratory historical data to insure that the limits continue to reflect realistic, method achievable goals.

## 12.3 REPORTING

Reporting procedures are documented in SOP #030201, *Data Handling and Reporting*.

Analyte Met	hod	Matrix	Accuracy (%)	Prec. (% RPD)	RL Unit	
1,1,1-Trichloroethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,1,2,2-Tetrachloroethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,1,2,2-Tetrachloroethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,1,2-Trichloroethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,1-Dichloroethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,1-Dichloroethene	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,2,4-Trichlorobenzene	TO-15	Air	53.6-154	25.0	0.63	ppbv
1,2,4-Trimethylbenzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,2-Dibromoethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,2-Dichlorobenzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,2-Dichloroethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,2-Dichloropropane	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,3,5-Trimethylbenzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,3-Butadiene	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,3-Dichlorobenzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,4-Dichlorobenzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
1,4-Dioxane	TO-15	Air	48.0-156	25.0	0.2	ppbv
1,1,1-Trichloroethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
2,2,4-Trimethylpentane	TO-15	Air	70.0-130	25.0	0.2	ppbv
2-Chlorotoluene	TO-15	Air	70.0-130	25.0	0.2	ppbv
2-Propanol	TO-15	Air	50.4-152	25.0	0.2	ppbv
4-Ethyltoluene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Acetone	TO-15	Air	70.0-130	25.0	1.25	ppbv
Allyl Chloride	TO-15	Air	70.0-130	25.0	0.2	ppbv
Benzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Benzyl Chloride	TO-15	Air	55.6-160	25.0	0.2	ppbv
Bromomethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
Bromodichloromethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
Bromoform	TO-15	Air	70.0-130	25.0	0.6	ppbv
Carbon Disulfide	TO-15	Air	70.0-130	25.0	0.2	ppbv
Carbon Tetrachloride	TO-15	Air	70.0-130	25.0	0.2	ppbv
Chlorobenzene	TO-15	Air	70.0-130	25.0	0.2	ppbv

<b>Table 12.3: QC Targets for Air Accuracy (LCS), Precision and RLs</b>						
<i>This table is subject to revision without notice</i>						
<b>Analyte Met</b>	<b>hod</b>	<b>Matrix</b>	<b>Accuracy (%)</b>	<b>Prec. (% RPD)</b>	<b>RL Unit</b>	
Chloroethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
Chloroform	TO-15	Air	70.0-130	25.0	0.2	ppbv
Chloromethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
Cis-1,2-Dichloroethene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Cis-1,3-Dichloropropene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Cyclohexane	TO-15	Air	70.0-130	25.0	0.2	ppbv
Dibromochloromethane	TO-15	Air	70.0-130	25.0	0.2	ppbv
Dichlorodifluoromethane	TO-15	Air	56.7-140	25.0	0.2	ppbv
Ethanol	TO-15	Air	34.3-167	25.0	0.63	ppbv
Ethyl Acetate	TO-15	Air	70.0-130	25.0	0.2	ppbv
Ethylbenzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Freon-11	TO-15	Air	70.0-130	25.0	0.2	ppbv
Freon-12	TO-15	Air	70.0-130	25.0	0.2	ppbv
Freon-113	TO-15	Air	70.0-130	25.0	0.2	ppbv
Freon-114	TO-15	Air	70.0-130	25.0	0.2	ppbv
Gasoline Range Organics	TO-15	Air	70.0-130	25.0	50	ppbv
Heptane	TO-15	Air	70.0-130	25.0	0.2	ppbv
Hexachloro-1,3-Butadiene	TO-15	Air	62.1-143	25.0	0.63	ppbv
Hexane	TO-15	Air	70.0-130	25.0	0.2	ppbv
Isopropylbenzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
M&P-Xylene	TO-15	Air	70.0-130	25.0	0.4	ppbv
Methyl Butyl Ketone	TO-15	Air	47.9-165	25.0	1.25	ppbv
Methyl Ethyl Ketone	TO-15	Air	70.0-130	25.0	1.25	ppbv
Methyl Isobutyl Ketone	TO-15	Air	55.3-154	25.0	1.25	ppbv
Methyl Methacrylate	TO-15	Air	70.0-130	25.0	0.2	ppbv
Methyl tert Butyl Ether	TO-15	Air	70.0-130	25.0	0.31	ppbv
Methylene Chloride	TO-15	Air	70.0-130	25.0	0.63	ppbv
Naphthalene	TO-15	Air	52.0-158	25.0	0.63	ppbv
N-butyl benzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
N-propyl benzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
o-Xylene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Propene	TO-15	Air	53.9-143	25.0	0.4	ppbv
Sec-butyl benzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Styrene	TO-15	Air	70.0-130	25.0	0.2	ppbv
t-Butyl Alcohol	TO-15	Air	70.0-130	25.0	0.2	ppbv

<b>Table 12.3: QC Targets for Air Accuracy (LCS), Precision and RLs</b>						
<i>This table is subject to revision without notice</i>						
Analyte Met	hod	Matrix	Accuracy (%)	Prec. (% RPD)	RL Unit	
Tert-butyl benzene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Tetrachloroethylene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Tetrahydrofuran	TO-15	Air	65.0-140	25.0	0.2	ppbv
Toluene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Trans-1,3-Dichloropropene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Trans-1,2-Dichloroethene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Trichloroethylene	TO-15	Air	70.0-130	25.0	0.2	ppbv
Vinyl Acetate	TO-15	Air	70.0-130	25.0	0.2	ppbv
Vinyl Bromide	TO-15	Air	70.0-130	25.0	0.2	ppbv
Vinyl Chloride	TO-15	Air	70.0-130	25.0	0.2	ppbv
Methane	RSK-175	Air/ Headspace	85.0-115	20.0	0.01	ppmv
Ethane	RSK-175	Air/ Headspace	85.0-115	20.0	0.0129	ppbmV
Ethene	RSK-175	Air/ Headspace	85.0-115	20.0	0.0127	ppmv
Propane	RSK-175	Air/ Headspace	85.0-115	20.0	0.0186	ppmv
Acetylene	RSK-175	Air/ Headspace	85.0-115	20.0	0.0208	ppmv
Carbon Dioxide	ASTM D1946	Air	70.0-130	20.0	0.50 / 200	% / ppmv
Carbon Monoxide	ASTM D1946	Air	70.0-130	20.0	0.50 / 200	% / ppmv
Methane	ASTM D1946	Air	70.0-130	20.0	0.50 / 200	% / ppmv
Nitrogen	ASTM D1946	Air	70.0-130	20.0	0.50 / 200	% / ppmv
Oxygen	ASTM D1946	Air	70.0-130	20.0	0.50 / 200	% / ppmv
Methanol	MEETAC	Water/Soil	70.0-130	20.0	20.0/100	ppb / ppm
Ethanol	MEETAC	Water/Soil	70.0-130	20.0	20.0/100	ppb / ppm

### 13.0 CORRECTIVE ACTION

- 13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CARs are kept on file by the Regulatory Affairs Department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

## 13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these limits are exceeded, corrective action must be taken. Calculated sample spike control limits are also used.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP

### 13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory, the method criteria takes precedence.

### 13.2.2 Calibration Verification Criteria Are Not Met.

Rejection Criteria – See Table 8.5.

Corrective Action – Instrument settings are checked. The standard is reviewed for obvious cause. The standard may require re-analysis or the instrument may require recalibration.

### 13.2.3 Out Of Control Blanks:

Rejection Criteria - Blank reading is more than ½ the RL.

Corrective Action - Instrument settings are checked. The Blank is re-analyzed. If the blank is still out of control, bakeout of the system is performed and the blank is re-analyzed.

### 13.2.4 Out Of Control Laboratory Control Standards (LCS)

Rejection Criteria - If the performance is outside of lab-generated control (Listed in Table 12.3).

Corrective Action - Instrument settings are checked. The LCS standard is re-analyzed. If the LCS is still out of control, re-calibration is performed, and samples affected since the last in control reference standard are re-analyzed.

## 14.0 RECORD KEEPING

Record keeping is outlined in SOP #030230, *Standards Logger*, SOP #030227, *Data Review* and SOP #030201, *Data Handling and Reporting*



## 15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 13.0 and in *SOP #010104, Internal Audits*.

## 16.0 REVISIONS

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of this appendix are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0 (Appendix VIII)	Section 5.1 – Removed language about supervisor and backup reviewing and approving all data Section 8.1 – Updated equipment list Section 8.4 – Changed daily detector mass calibration using PFTBA to using BFB for GC/MS Section 16 – New section for summary of revisions to previous version

1.0 SIGNATORY APPROVALS

# Aquatic Toxicity Laboratory QUALITY ASSURANCE MANUAL

## APPENDIX IX TO THE ESC QUALITY ASSURANCE MANUAL

for


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Prepared by


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### **3.0 SCOPE AND APPLICATION**

This appendix discusses specific QA requirements for general analytical protocols to ensure that analytical data generated from the Aquatic Toxicity laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in non-conforming work must be immediately evaluated and their corrective actions documented.

### **4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES**

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling guidance and materials and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual*.

### **5.0 PERSONNEL AND TRAINING**

#### **5.1 PERSONNEL**

Dr. Christabel Fernandes-Monteiro, with a Ph.D. in Applied Biology, is the Department Manager of Biology. She oversees supervision of laboratory operations in the Mold, Aquatic Toxicity, Microbiology, Protozoan and BOD laboratories. Her responsibilities include assurance of reliable data through monitoring of quality control, corroborating the analysis performed, protocol development, coordination with clients regarding sample analysis, scheduling of tests and overall production in all sections within the Biology Laboratory, including management of staff. In her absence, Shain Schmitt assumes her responsibilities in the Aquatic Toxicity laboratory.

Shain Schmitt with a B.S. degree in Conservation Biology, is the Primary Analyst for the Aquatic Toxicity laboratory. Mr. Schmitt is proficient in aquatic toxicity analytical methods and is responsible for sample analysis, review and approval of data associated with toxicity analyses. His responsibilities also include the coordination with clients regarding sample analysis, scheduling, data reductions, interpretation and validation of toxicity testing. In his absence, Brandon Etheridge assumes his responsibilities.

#### **5.2 TRAINING**

All new analysts to the laboratory are trained by the Primary Analyst or Manager according to ESC protocol. ESC's training program is outlined in *SOP 350355 Technical Training and Personnel Qualification for Biology–Aquatic Toxicity*. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in toxicity analysis is also demonstrated by acceptable participation in the Phenova proficiency testing

program (PTs) as well as by performing routine reference toxicant testing at the same concentrations and in the same dilution water as is used for field sample testing. Documentation of analyst training is maintained on file within the department.

## **6.0 FACILITIES AND LABORATORY SAFETY**

### **6.1 FACILITIES**

The main area of the laboratory has approximately 1440 square feet of area with roughly 280 square feet of bench area. There are 300 square feet of additional storage and the lighting is fluorescence. The air system is a five-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the Siemens Elga UltraPure deionizer system. Biohazard containers are located in the laboratory and Stericycle Waste Removal serves as ESC's biological waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

### **6.2 LABORATORY SAFETY**

- Laboratory access is limited when work is being performed.
- The following Biosafety Level 2 (BSL2) guidelines are adhered to:
  - Closed-toe shoes are worn in the laboratory
  - Floors and work surfaces are cleaned on a regular basis
  - Emergency numbers are posted in the laboratory
  - Laboratory personnel are trained in the use of the biological spill kit and emergency safety equipment
- ESC's laboratory safety guidelines are detailed in the ESC *Chemical Hygiene Plan*.

## **7.0 SAMPLING PROCEDURES**

### **7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING**

- Field Sampling procedures are described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Samples are received in the laboratory login area and are tracked using LIMS (Laboratory Information Management System). A Chain of Custody Form accompanies all samples received by the lab. This is necessary to prove the traceability of the samples and to document the change in possession from sampling to delivery to receipt by the laboratory. Prior to analysis samples are checked for integrity. Once samples are checked to confirm integrity, the samples are logged with unique sample identification information and a label is affixed to each container. Chronic Toxicity samples are uniquely identified with "sample 1, sample 2 and sample 3". A sample custodian then transports samples to the laboratory. Sample handling and tracking procedures are outlined in *SOP 060105, Sample Receiving*.

- Requirements for sample acceptance are located in *SOP 060105, Sample Receiving*. At a minimum, the following physical and chemical parameters are analyzed for each sample received:
  - Temperature
  - pH - initial and final measurements recorded
  - D.O. - initial and final measurements recorded
  - Specific Conductance
  - Alkalinity
  - Hardness
  - Total Residual Chlorine
- Samples must be immediately cooled and maintained at 0-6°C following sampling, during shipment and prior to testing.

**Residual Chlorine Treatment**

- Residual chlorine in biomonitoring samples is monitored using a pocket colorimeter and these checks are documented. Chlorine removal is not performed on submitted field samples.

**Dissolved Oxygen**

For acute tests, samples that are  $\leq 4.0$ mg/L are aerated until the sample reaches 90% saturation. For chronic tests, samples that are  $\leq 5.0$  mg/L are aerated until the sample reaches 90% saturation.

**8.0 EQUIPMENT**

**8.1 EQUIPMENT LIST**

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Aquatic Toxicity Lab</b>			
<i>This table is subject to revision without notice.</i>			
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Location</i>
Analytical Balance	Mettler	AT261 Delta Range	Aquatic Tox Lab
Class “I” weights (2)	Troemner		Aquatic Tox Lab
Conductivity Meter	Orion	150 A+	Aquatic Tox Lab
Dissolved Oxygen Meter	YSI	Model 50	Aquatic Tox Lab
Stereoscope	Olympus	SZX-IIIK100	Aquatic Tox Lab
Oven	Fisher	655F	Aquatic Tox Lab
Incubator	Thermo-Kool	Environmental chamber	Aquatic Tox Lab
Incubator	Percival Scientific	1-37 VL	Aquatic Tox Lab
Incubator	Precision Sci.	818	Aquatic Tox Lab
Incubator (2)	Precision Sci.	818	Aquatic Tox Lab
Microscope	Olympus	CHT	Aquatic Tox Lab

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Aquatic Toxicity Lab</b>			
<i>This table is subject to revision without notice.</i>			
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Location</i>
pH Meter	Orion	VersaStar	Aquatic Tox Lab
Refrigerator (2)	Beverage Air	E Series	Aquatic Tox Lab
Stereoscope	Olympus	SZH-ILLD	Aquatic Tox Lab
Stereoscope	Olympus	SZH-ILLD	Aquatic Tox Lab
Refrigerator	Frigidaire	FRC445GB	Aquatic Tox Lab
Refrigerator	True	T-49	Aquatic Tox Lab
Water Purifier	Siemens	Elga LabPure S4	Aquatic Tox Lab
Refrigerator	Fridgidaire	FRC 445GB	Aquatic Tox Lab
pH/Conductivity Benchtop meter	Thermo Scientific Orion	VSTAR 52	Aquatic Tox Lab
RDO Probe	Thermo Scientific Orion	VSTAR-RD	Aquatic Tox Lab
Oven (2)	VWR	13054	Aquatic Tox Lab
Stereoscope	Olympus	SZH-STS	Aquatic Tox Lab

## 8.2 E EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

<b>PREVENTATIVE MAINTENANCE FOR LABORATORY EQUIPMENT</b>		
<i>INSTRUMENT</i>	<i>P. M. DESCRIPTION</i>	<i>FREQUENCY</i>
Analytical Balances	•Check with Class "I" weights	Daily-tolerance 1 gm - ±0.0001 gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	10 gm - ±0.01 gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semi-annually
Refrigerators & Incubators	•Maintenance service	As needed - determined by twice daily temperature performance checks @ least 4 hours apart
Dissolved oxygen meter	•Calibrate with each use	Daily
Dissolved oxygen meter	•Change probe membrane	Every two to four weeks when in use
Conductivity Meter	•Check probe cables	As needed
Conductivity Meter	•Clean probe	As needed
Conductivity Meter	•Replace or replatinize probe	Poor response not corrected by above
Conductivity Meter	•Calibrate with each use	Daily (or prior to each use)
Microscope/Stereoscope	•Service/calibration of each ocular micrometer	Annually
Microscope/Stereoscope	• Clean optics and stage	As needed
pH Meters	•Reference junction & electrode replacement	As needed
pH Meters	•Probe stored in pH standard 4	At all times when not in use
pH Meters	•Other	As described in the manufacturer's manual
pH Meters	•Calibrate with each use	Daily (or prior to each use)

<b>PREVENTATIVE MAINTENANCE FOR LABORATORY EQUIPMENT</b>		
<i>INSTRUMENT</i>	<i>P. M. DESCRIPTION</i>	<i>FREQUENCY</i>
pH meter	•ATC checks	Annually
Bottle top dispenser/repipettor	•Calibrate	Quarterly
Bottle top dispenser/repipettor	•Clean to prevent residue buildup	As needed
Water Purifier	Tank Exchange, UV bulb and sleeve replacement ( service contract maintenance and check	As needed and annually
Water Purifier	•Replace cartridge and filter	As needed and semi-annual
RDO probe	•Replace sensor cap	Annually
RDO probe	•Clean sensor cap	As needed
RDO probe	•Other	As described in manufacturer's manual
pH/Conductivity/DO meter	•Calibrate with each use	Daily
Light Meter	•Calibrate	Annually

### 8.3 STANDARDS, REAGENTS AND ORGANISM CULTURES

All reagents and standards must meet the requirements listed in the analytical methods.

<b>Table 8.3A: Stock solution sources, description and related information.</b> (subject to revision as needed)				
<b>Description</b>	<b>Vend</b>	<b>or</b>	<b>Storage Req.</b>	<b>Expiration</b>
Conductivity standard 1413		NSI	Ambient	1 yr
pH buffer 7		-VWR	Ambient	1 yr
pH buffer 10		-VWR	Ambient	1 yr
Bromothymol blue solution		-VWR	Ambient	1 yr
Potassium phosphate monobasic		-VWR	Ambient	1 yr
Magnesium chloride		-VWR	Ambient in dessicator	1 yr
Potassium Chloride		-VWR	Ambient in dessicator	1 yr
Brine shrimp eggs		Brine Shrimp Direct (BSD)	Ambient, tightly sealed.	1 yr
Calcium sulfate		-VWR	Ambient in dessicator	1 yr
EDTA		-VWR	Ambient in dessicator	1 yr
Sodium thiosulfate		-VWR	Ambient in dessicator	1 yr
pH buffer 4		-VWR	Ambient.	1 yr
YCT		Made in-house	-10 to -20°C	14 days after thawing
<i>Raphidocelis subcapitata</i>		Aq. Biosystems	1-6°C	One month from concentration date
Vitamin B12		ICN	1-6°C	NA

**TABLE 8.3B: Working Solution Descriptions and Related Information.**  
(subject to change)

<b>Solution Concentrations</b>		<b>Storage Requirements</b>	<b>Expiration</b>
KCl stock solution	31.237g KCl to 2L of mod. Hard SDW	1-4°C	14 days
B12 Solution	0.01125g to 1L of DI Water	1-4°C	NA



**Source and Maintenance of in-house cultures:**

**Source of Biological Organisms (subject to change):**

The primary source for all fathead minnows is:

Aquatic Biosystems Inc.  
2821 Remington Street  
Fort Collins, CO 80525

The source for their organisms is documented on each packing slip received. ESC accepts the packing slip as documentation and verification by the supplier with regard to the taxonomic identification of the bioassay species. The packing slips for bioassay test organisms are kept on file.

The amount of food added to culture vessels depends upon the number of organisms within a given culture. As standard procedure, *Ceriodaphnia dubia* batch cultures are fed 4.5mL of YCT and algal suspension on the day of initiation. Batches are fed as needed. The date, time and the amount the organisms are fed is documented. All yeast purchased is at least food grade and has passed FDA standards. All (YCT) Yeast Trout Chow is made in-house. New lots are tested for pesticides, metals, and PCBs.

*Ceriodaphnia dubia*, fresh batch cultures are set up on Monday, Wednesday and Friday using newly hatched neonates less than 24 hours old. In addition, a minimum of 4 brood trays are set up daily in order to guarantee organisms of the right age to use in bioassay tests. The *C. dubia* brood trays are fed daily. The *C. dubia* are transferred into fresh water daily after their first brood of neonates is born. Third generation neonates, less than 24 hours old, are used for batch cultures and brood trays. Third generation neonates, less than 24 hours old and hatched within 8 hours of each other, are used for chronic tests. Adults are used as sources for neonates until 14 days of age.

*C. dubia* are taxonomically identified to species on a quarterly basis. All taxonomy information is documented and kept on file for a year.

*Pimephales promelas* batch cultures are cleaned as needed by siphoning off the excess food and waste from the bottom of the culture vessel and renewing the water. Cultures are aerated as needed to maintain adequate dissolved oxygen.

*Pimephales promelas* are taxonomically identified to species on a quarterly basis. All taxonomy information is documented and kept on file for a year.

The water used for culturing is moderately hard synthetic dilution water (SDW) and is prepared by diluting 1L synthetic freshwater concentrate to 20 L ultra-pure deionized water, and vigorously aerating for a minimum of 1 hour. The physical and chemical

parameters for each new tank of water prepared are recorded and should fall within the following acceptable range:

1. pH – 7.5- 8.5 units
2. D.O. - greater than 80% saturation in mg/L
3. Specific Conductance - ~250 micromhos/cm
4. Alkalinity - 57-64 mg CaCO<sub>3</sub>/L
5. Hardness - 80 to 100 mg CaCO<sub>3</sub>/L
6. Total Residual Chlorine - <0.1 mg/L

## 8.4 I INSTRUMENT CALIBRATION

### Lighting

All testing and culturing is maintained in incubators in which temperature is constant and the photoperiod is on a 16-hour light/8-hour dark cycle. The photoperiod is verified and documented quarterly. The light intensity must be within 50 – 100 foot candles (approximately 10-20  $\mu\text{E}/\text{m}^2/\text{s}$ ) and is verified and documented quarterly. All incubators are monitored at least weekly for proper light intensity.

### pH Meter

The pH meters are calibrated with each use according to manufacturer's instructions. The slope is documented on a daily basis. Ensure the acceptable pH slope range is within the manufacturer's acceptable range prior to use. Perform automatic temperature compensation (ATC) checks annually on the pH probe. All calibration information is documented.

### Volumetric Equipment

Equipment such as filter funnels, bottles, pipettes, non-Class A and other containers with graduations are calibrated once per lot prior to first use. The error of calibration must not exceed 3.0%.

### Analytical Balance

Analytical balances are checked and calibrated semi-annually by a certified technician. Calibration is checked before each use with Class I weights. Class I weights are calibrated annually.

### Stereoscope

Maintenance is performed by a trained technician on an annual basis.

### Conductivity Meter

The conductivity meter is calibrated with each use according to manufacturer's instructions.

#### **Dissolved Oxygen Meter**

The DO meter is calibrated according to manufacturer's instructions with each use. The electrochemical probe membrane is changed every two to four weeks to maintain accurate readings when in use. The RDO probe sensor cap is cleaned regularly, and replaced once per year. The RDO probe sensor cap must be stored in a moist environment.

#### **Test Chambers**

Each test chamber is rinsed with DI water prior to introducing the test organisms.

#### **Bottle Top Dispenser/Repipettor**

Repipettors are calibrated quarterly to ensure the instrument is dispensing the correct amount. Periodic cleaning is performed to maintain the accuracy and to prevent buildup of residue.

#### **Colorimeter Chlorine tester**

The colorimeter is calibrated before each use using standards to verify accuracy.

#### **Light Meter**

Calibrate the light meter annually to ensure it meets original performance specifications

## **9.0 LABORATORY PRACTICES**

### **9.1 REAGENT GRADE WATER**

Deionized water or reverse-osmosis produces water free from bactericidal and inhibitory substances and is used in the preparation of media, solutions and buffers. The quality of the water is monitored for chlorine residual, specific conductance, and heterotrophic bacteria plate count monthly (when in use), when maintenance is performed on the water treatment system, or at startup after a period of disuse longer than one month.

Analysis for metals is performed quarterly and the Bacteriological Water Quality Test (to determine presence of toxic agents or growth promoting substances) is performed annually. Results of these analyses meet the specifications of the required method and records of analyses are maintained for five years. (An exception to performing the Bacteriological Water Quality Test can be given to laboratories that can supply documentation to show that their water source meets the criteria, as specified by the method, for Type I or Type II reagent water.)

## 9.2 PH BUFFERS/CONDUCTIVITY STANDARDS

pH buffer and conductivity standard aliquots are used only once. Reagents containers are dated upon receipt and the date opened.

## 9.3 SECONDARY STANDARDS

Standards are used for retrieval and verification of the factory calibrated colorimeter and are used to verify consistent instrument calibration.

## 9.4 LABORATORY CONTROL WATER

Control water (moderately hard synthetic dilution water- SDW) is prepared by diluting 1L of synthetic freshwater concentrate with 20L deionized water and aerating for a minimum of 1 hour. The physical and chemical parameters for each new tank of water prepared are recorded and should fall within the following acceptable range:

1. pH – 7.5-8.5 units
2. D.O. - greater than 80% saturation in mg/L
3. Specific Conductance - ~250 micromhos/cm
4. Alkalinity - 57 to 64 mg CaCO<sub>3</sub>/L
5. Hardness - 80 to 100 mg CaCO<sub>3</sub>/L
6. Total Residual Chlorine - <0.1 mg/L

Control water (10% dilute mineral water-DMW) is prepared by diluting (3) 750mL bottles of Perrier to the 20 Liters mark of a 20 L NALGENE® carboy with ultra-pure deionized water and aerating for a minimum of 1 hour. The physical and chemical parameters for each new tank of water prepared are recorded and should fall within the following acceptable range:

1. pH – 6.5 to 8.5 units
2. D.O. - greater than 80% saturation in mg/L
3. Specific Conductance - ~95 micromhos/cm
4. Alkalinity - 60 to 70mg CaCO<sub>3</sub>/L
5. Hardness - 30 to 50mg CaCO<sub>3</sub>/L
6. Total Residual Chlorine - <0.1mg/L

A given batch of control water is not used for more than 14 days following preparation.

## 9.5 B BRINE SHRIMP

Artemia cysts are certified brine shrimp eggs from Brine Shrimp Direct. To determine the quality of the new lots of Brine shrimp, a side-by-side comparison test is performed using the new food and the food of known acceptable quality.

## 9.6 YCT

YCT-Yeast Cereal leaves and Trout chow is prepared in the laboratory. To determine the quality of the new lots of YCT a side-by-side comparison test is performed using the new food and the food of known acceptable quality.

## 9.7 A LGAE

Algae are commercially prepared. Upon arrival, each batch received has an accompanying Certificate of Algae Preparation History. The certificate provides the following quality control data: date prepared, species name, inoculation date, harvest date, concentration date and cell count.

## 9.8 G GLASSWARE WASHING, STERILIZATION PROCEDURES AND EQUIPMENT STERILITY CHECKS

Glassware washing and preparation/sterilization procedures are performed according to EPA guidelines and are outlined in *SOP 030701 Glassware Cleaning* and *SOP 350335, Quality Control and Quality Assurance of Microbiological Equipment and Testing Materials*. Before use, examine and discard items with chipped edges or etched inner surfaces. Reusable glassware is cleaned using the following protocol:

- Soak for 15 minutes in hot tap water with detergent and scrub. Rinse thoroughly with tap water. Rinse thoroughly with dilute nitric acid (10%). Rinse thoroughly with deionized water. Rinse thoroughly with pesticide grade acetone. Rinse well with deionized water.
- New glassware is cleaned according to the same procedure as listed above except the first step is preceded by soaking overnight in 10 % HNO<sub>3</sub>.

Inspect glassware after washing for excessive water beading and rewash, if necessary. Perform checks on pH and test for inhibitory residues on glassware and plastic ware. Use utensils and containers of borosilicate glass, stainless steel, aluminum, or other corrosion resistant material for media preparation. All biological glassware is purchased pre-sterilized. In-house sterilization of any auxiliary equipment is performed via autoclave.

Pipettes of all sizes are checked for sterility by drawing up non-selective media into the pipette and re-dispensing the volume back into original tube that contained the media. The tube is then incubated and monitored for growth. All results are recorded and maintained within the laboratory.

## 10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOPs associated with the Aquatic Toxicity laboratory can be found in the following table:

**TABLE 10.1: AQUATIC TOXICITY DEPARTMENT SOPs**

*This Table is subject to revision without notice*

SOP #	Title/Description
340312	Dissolved Oxygen Membrane Electrode Method
350301	Fathead Minnow, <i>Pimephales promelas</i> , Larval Survival and Growth Test, EPA Method 1000.0
350302	Cladoceran, <i>Ceriodaphnia dubia</i> , Chronic Survival and Reproduction Test, EPA Method 1002.0
350303	<i>Pimephales promelas</i> Acute Toxicity Testing, EPA Method 2000.0
350303NC	North Carolina <i>Pimephales promelas</i> Acute Toxicity Testing
350304	<i>Ceriodaphnia dubia</i> Acute Toxicity Testing EPA Method 2002.0
350304NC	North Carolina <i>Ceriodaphnia dubia</i> Acute Toxicity Testing
350317	WET Reference toxicant testing
350318	Mini Chronic <i>C. dubia</i> NC
350320	Acceptability Test for New Food Batches for WET Testing
350321	Pocket Colorimeter Chlorine Tester Maintenance and Calibration
350322	DO Meter Maintenance and Calibration
350323	Fluke Thermometer Operation and Maintenance
350324	Digital Light Meter Maintenance and Method of Operation
350325	pH Meter Maintenance and Calibration
350326	Thermometer Operation, Maintenance and Calibration Procedure
350327	Bottle Top Dispenser Maintenance and Method of Operation
350328	Conductivity Meter Maintenance and Calibration
350329	Taxonomic Verification/Identification of <i>Pimephales promelas</i> - Fathead Minnow
350330	Taxonomic Verification/Identification of <i>Ceriodaphnia dubia</i>
350345	Receipt and Maintenance of <i>Pimephales Promelas</i> (Fathead Minnow)
350346	<i>Ceriodaphnia Dubia</i> Culture Maintenance, Food Preparation, and Food Maintenance
350355	Technical Training and Personnel Qualifications for Biomonitoring-Aquatic Toxicity, Mold and Microbiology
350356	Water Bath and Incubator Temperature Stability and Load Testing
350362	Analytical Balance Operation and Verification in the Aquatic Toxicity Microbiology Lab
350364	North Carolina Phase II Chronic Whole Effluent Toxicity Test Procedure for <i>Ceriodaphnia dubia</i>

10.2 Additional information regarding Aquatic Toxicity testing can be found in:

Method Resources: EPA/821/R-02/013, EPA/821/R-02/012

- 7-Day Fathead Minnow (*Pimephales promelas*) Larval Survival and Growth Test; Test Method 1000.0 from "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (EPA 821-R-02-013).
- 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test; Test Method 1002.0 from "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (EPA 821-R-02-013).
- Fathead Minnow (*Pimephales promelas*) Acute Toxicity Test (24, 48 or 96 hour duration); referenced in "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms" (EPA 821-R-02-012, 10-02).
- *Ceriodaphnia dubia* Acute Toxicity Test (24, 48 or 96 hour duration); referenced in "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms" (EPA 821-R-02-012, 10-02)

## 11.0 QUALITY CONTROL CHECKS

11.1 At a minimum, the following physical and chemical parameters are analyzed for each biomonitoring sample received:

- Temperature - recorded up to twice daily.
- pH - initial and final measurements recorded
- D.O. - initial and final measurements recorded
- Specific Conductance
- Alkalinity
- Hardness
- Total Residual Chlorine

## 11.2 FEEDING REGIME

- 7-Day Fathead Minnow Larval Survival and Growth Test - Test organisms are fed 0.15mL, per container of 10 organisms. Newly hatched brine shrimp (*Artemia*) are fed to minnow batches 2-3 times daily. Batch cultures are fed depending on organism density.
- 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test - test organisms are fed 0.15mL of Yeast, Cereal leaves, Trout chow (YCT) and 0.15mL *Selenastrum capricornutum* algal suspension once daily.
- 24 and 48 Hour Acute Toxicity Tests - organisms are fed 2-5 hours prior to introduction into sample but are not fed for the duration of the test.
- 96-Hour Acute Toxicity Tests – organisms are fed at the 48 hour renewal period.

- 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test for North Carolina - test organisms are fed .05mL of YCT/15mL test solution and .05 *Raphidocelis subcapitata* algal concentrate once daily ( $1.7 \times 10^7$  to the 7th power cells/mL).

### 11.3 B BATCH CULTURES

Batch cultures are identified by date set up or date received. The set-up date is recorded for each batch.

*Ceriodaphnia dubia*, fresh batch cultures are set up on Monday, Wednesday and Friday using newly hatched neonates less than 24 hours old. In addition, a minimum of 4 brood trays are set up daily in order to guarantee organisms of the right age to use in bioassays. Condition of cultures is monitored daily and documented in the daily log. The *C. dubia* brood trays are fed daily. The *C. dubia* are transferred into fresh water daily after their first brood of neonates is born. Third generation neonates, less than 24 hours old, are used for batch cultures and brood trays. Third generation neonates, less than 24 hours old and hatched within 8 hours of each other, are used for chronic tests. Adults are used as sources for neonates until 14 days of age.

*Pimephales promelas*, organisms less than 36 hours old are obtained from a commercial supplier and are used immediately for chronic bioassays. Upon receipt, temperature, conductivity, pH, alkalinity and hardness are recorded and the organisms are slowly acclimated to a temperature of 25°C. If more than 10% mortality has occurred in the batch shipment, the batch is rejected and supplier is contacted. The date of the batch culture is recorded and batches are maintained for 14 days after receipt to use in acute tests. Batch cultures are monitored and fed daily. The number of organisms used is recorded in the daily log. Lots are cleaned as needed by siphoning off the excess food and waste from the bottom of the vessel and renewing the water. Minnow lots are aerated to maintain adequate dissolved oxygen. *Pimephales promelas* lots are fed 2.5 mL of newly-hatched brine shrimp per batch, 2-3 times daily. The date, time and the amount the organisms are fed are documented.

### 11.4 REFERENCE TOXICANT

The reference toxicant used at ESC is potassium chloride. Acute and chronic reference toxicant tests are performed at a minimum of once monthly and upper and lower control limits have been established.

## 12.0 DATA REDUCTION, VALIDATION AND REPORTING

### 12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in *SOP 030201 Data Handling and Reporting*. The primary analyst reviews the quality of data based on the following guidelines:



- The appropriate SOP has been followed
- Sample preparation is correct and complete
- Analytical results are correct and complete
- QC is within criteria and complete

All calculations are performed according to the EPA methods manual. When applicable, software is used to perform statistical analysis. All formulae are chosen appropriately depending on the conditions and outcome of each individual test. Due to the complexity of each formula please see EPA/821/R-02/013 for formulae pertaining to Chronic Toxicity tests and EPA/821/R-02/012 for formulae pertaining to Acute Toxicity tests.

**TABLE 12.1 Data Reduction Formulas**

PARAMETER	FORMULA
IC25, NOEC, LC50, AEC	Toxcalc 5.0 Software

For chronic tests the PMSD and the % CV is calculated and reported.

## 12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by reviewing all data entries and calculations for errors, reviewing all documentation to assure that sample information is correct, and that the tests have been performed appropriately and within the appropriate holding times. The secondary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP has been followed
- Sample preparation is correct and complete
- Analytical results are correct and complete

## 12.3 REPORTING

Reporting procedures are documented in *SOP 030201 Data Handling and Reporting* and *SOP 030227, Data Review*.

### 13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CARs are kept on file by the Regulatory Affairs Department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

#### 13.2 Required Corrective Action

All samples and procedures are governed by ESC's quality assurance program. Designated corrective actions are as follows:

##### 13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria takes precedence.

##### 13.2.2 Out of control acute toxicity tests.

Rejection Criteria – More than 10% mortality occurs in the control organisms within the specified time frame of the test.

Corrective Action – The test is considered invalid and must be repeated using fresh control water and fresh sample.

##### 13.2.3 Out of control 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test.

Rejection Criteria – If more than 10% mortality occurs in the control organisms within 96 hours or more than 20% mortality occurs in the test organisms in the 3-brood period (approx. 7 days)

Corrective Action – The test is considered invalid and must be repeated using fresh control water and fresh sample.

##### 13.2.4 Out of control 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test.

Rejection Criteria – If the average number of young produced in the control is less than 15 per organism

Corrective Action – The test is considered invalid and must be repeated using fresh control water and fresh sample.

13.2.5 Out of control 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test.

Rejection Criteria – A test is considered invalid if less than 60% (80% for NC tests) of the original number of adult daphnia loaded do not produce three broods within an eight day maximum (7 day maximum for NC tests).

Corrective Action – The test is considered invalid and must be repeated using fresh control water and fresh sample.

13.2.6 Out of control 7-Day *Pimephales promelas* Larval Survival and Growth Test.

Rejection Criteria – If more than 10% mortality occurs in the control organisms within 96 hours or more than 20% mortality occurs in the test organisms in 7 day period.

Corrective Action – The test is considered invalid and must be repeated using fresh control water and fresh sample.

13.2.7 Out of control 7-Day *Pimephales promelas* Larval Survival and Growth Test.

Rejection Criteria – The average weight of the control minnows is less than 0.2500 mg.

Corrective Action – The test is considered invalid and must be repeated using fresh control water and fresh sample.

13.2.8 Out of control Monthly Reference Toxicant:

Rejection Criteria – KCl is the reference toxicant used for acute and chronic testing for the following methods: 1000.0, 1002.0, 2000.0, and 2002.0. If reference toxicant test results fail to meet ESC in-house established criteria ( $\pm 2$  standard deviations from the mean & median).

Corrective Action – The test is deemed invalid and must be repeated twice. No test will be performed using organisms that fail to meet reference toxicant criteria.

13.2.9 Out of control PMSD 7-Day *Pimephales promelas* Larval Survival and Growth Test.

Rejection Criteria – The PMSD value is greater than the upper value of 30.

Corrective Action - The test may be deemed invalid and should be repeated.

13.2.10 Out of control PMSD 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test.

Rejection Criteria – The PMSD value is greater than the upper value of 47.

Corrective Action - The test may be deemed invalid and should be repeated.

13.2.11 Out of control %CV 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test and 7-Day *Pimephales promelas* Larval Survival and Growth Test.

Rejection Criteria – The %CV value is greater than the upper value of 40%.

Corrective Action - The test is deemed invalid and must be repeated.

## 14.0 RECORD KEEPING

Record keeping is outlined in SOP #030230, *Standards Logger*, SOP #030227, *Data Review* and SOP #030201, *Data Handling and Reporting*

## 15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 13.0 and SOP #010104, *Internal Audits*.

## 16.0 REVISIONS

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of this appendix are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0 (Appendix IX)	Section 8.1 – Updated equipment list Section 8.2 – Clarified frequency of preventative maintenance Table 8.3A – Updated stock solutions Section 8.3 – Minor clarifications about maintenance of in-house cultures Section 8.4 – Revised lighting check frequency to quarterly. Removed language about non-disposable volumetric equipment being verified annually. Other minor grammatical changes in various other paragraphs. Section 11.2 – Changed algal species used for 3-Brood <i>Ceriodaphnia dubia</i> Survival and Reproduction Test for North Carolina from <i>Selenastrum capricornutum</i> to <i>Raphidocelis subcapitata</i> Section 16 – New section for summary of revisions to previous version

1.0 SIGNATORY APPROVALS

# Microbiology Laboratory QUALITY ASSURANCE MANUAL

## APPENDIX X TO THE ESC QUALITY ASSURANCE MANUAL

for


ESC LAB SCIENCES  
12065 LEBANON ROAD  
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(615) 758-5858

Prepared by

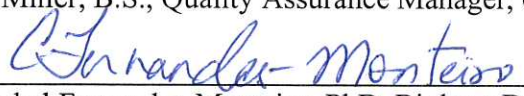
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**NOTE: The QAM has been approved by the following people.**

  
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### **3.0 SCOPE AND APPLICATION**

This appendix discusses specific QA requirements for general analytical protocols to ensure that analytical data generated from the Microbiology laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in non-conforming work must be immediately evaluated and their corrective actions documented.

### **4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES**

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling guidance and materials and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual*.

### **5.0 PERSONNEL AND TRAINING**

#### **5.1 PERSONNEL**

Dr. Christabel Fernandes-Monteiro, with a Ph.D. in Applied Biology, is the Department Manager of Biology. She oversees supervision of laboratory operations in the Mold, Aquatic Toxicity, Microbiology, Protozoan and BOD laboratories. Her responsibilities include assurance of reliable data through monitoring of quality control, corroborating the analysis performed, protocol development, coordination with clients regarding sample analysis, scheduling of tests and overall production in all sections within the Biology Laboratory, including management of staff. In her absence, Shain Schmitt assumes her responsibilities in the Microbiology laboratory.

Shain Schmitt with a B.S. degree in Conservation Biology, is the Primary Analyst for the Microbiology laboratory. Mr. Schmitt is proficient in microbiological analytical methods and is responsible for sample analysis, review and approval of data associated with microbiological analyses. In his absence, Brandon Etheridge assumes his responsibilities.

#### **5.2 TRAINING**

The Primary Analyst or Manager trains new laboratory analysts according to ESC protocol. ESC's training program is outlined in SOP #350355, *Technical Training and Personnel Qualification for Biomonitoring-Microbiology*. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in microbiological analysis is also demonstrated by acceptable participation in the Phenova proficiency testing program (PTs) and routine laboratory quality control practices. Documentation of analyst training is maintained on file within the department.

## **6.0 FACILITIES AND LABORATORY SAFETY**

### **6.1 FACILITIES**

The main area of the laboratory has approximately 1440 square feet of area with roughly 280 square feet of bench area. There are 300 square feet of additional storage and the lighting is fluorescence. The air system is a five-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the Siemens Elga Lab Pure deionizer system. Biohazard containers are located in the laboratory and Stericycle Waste Removal serves as ESC's biological waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

### **6.2 LABORATORY SAFETY**

- Laboratory access is limited when work is being performed.
- The following Biosafety Level 2 (BSL2) guidelines are adhered to:
  - Closed-toe shoes are worn in the laboratory
  - Floors and work surfaces are cleaned on a regular basis
  - Emergency numbers are posted in the laboratory
  - Laboratory personnel are trained in the use of the biological spill kit and emergency safety equipment
- ESC's laboratory safety guidelines are detailed in the ESC *Chemical Hygiene Plan*.

## **7.0 SAMPLING PROCEDURES**

### **7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING**

- Field Sampling procedures are described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Samples for bacterial analysis are collected directly into pre-sterilized high-density polyethylene (HDPE) sample containers preserved with sodium thiosulfate. The container should be kept closed until sample collection. Once the container is open, do not wash, rinse or contaminate the cap or the inside of the container. For microbiological samples, the container is filled allowing at least 1 inch of headspace per container.
- Sources for microbiological samples are surface waters, waste and drinking water, ground water and soil/sludge.
- Holding times for microbiological drinking water samples is generally 30 hours (except HPC which has a 8 hour holding time). Soil and sludge samples have a holding time of 24 hour and 8 hours depending on the method used. All other water samples have a 8-hour hold time.



- Microbiological samples are shipped in a cooler lined with a heavy-duty plastic bag. Once the sample container lids are secure, the samples are placed in appropriately sized polyethylene bags. The chain of custody is also placed in a plastic bag. The cooler liner is completely filled with ice and the plastic bag sealed tightly with a cable tie. The shipping label contains the name and address of the shipper and is affixed to the outside of the cooler.
- Samples are received in the laboratory login area and are tracked using LIMS (Laboratory Information Management System). A Chain of Custody Form accompanies all samples received by the lab. This is necessary to prove the traceability of the samples and to document the change in possession from sampling to delivery to receipt by the laboratory. Prior to analysis samples are checked for integrity. Sample handling, tracking and acceptance procedures are outlined in *SOP 060105, Sample Receiving*.

## 8.0 EQUIPMENT

### 8.1 EQUIPMENT LIST

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Microbiological Analysis</b>			
<i>This table is subject to revision without notice</i>			
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Location</i>
Analytical Balance	Mettler	AT261 Delta Range	Microbiology Lab
Class "I" weights	(2 sets) Troemner		Microbiology Lab
Conductivity Meter	Orion	150 A+	Microbiology Lab
Autoclave	Pelton and Crane	Validator 8	Microbiology Lab
Water Bath	Lindberg Blue	WB1130A	Microbiology Lab
Water Bath	Blue M	MW-1110A-1	Microbiology Lab
Oven	Fisher	655F	Microbiology Lab
Incubator	Percival Scientific	1-37 VL	Microbiology Lab
Incubator	VWR	2030 22MFG	Microbiology Lab
Quantitray Sealer	IDEXX	2X	Microbiology Lab
Incubator	Precision Sci.	818	Microbiology Lab
Colony Counter	Quebecor		Microbiology Lab
pH Meter	Beckman	pH/Temp/mV/ISE	Microbiology Lab
Refrigerator	True	T-49	Microbiology Lab
Stereoscope (2)	Olympus	SZH-ILLD	Microbiology Lab
UV light; short and long wave	UVP		Microbiology Lab
Water Bath	VWR Scientific	1295PC	Microbiology Lab
Autoclave	SterileMax	Harvey	Microbiology Lab
Stereoscope	Olympus	SZX-ILLK100	Microbiology Lab
Water Purifier	Siemens	Elga Lab Pure S4	Microbiology Lab
Oven	VWR	13054	Microbiology Lab
pH meter/Conductivity meter	Thermo Scientific Orion	VStar 52	Aquatic Tox Lab

## 8.2 E EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

<b>PREVENTATIVE MAINTENANCE FOR LABORATORY EQUIPMENT</b>		
<i>INSTRUMENT</i>	<i>P. M. DESCRIPTION</i>	<i>FREQUENCY</i>
Analytical Balances	•Check with Class "I" weights	Daily-tolerance 1 gm - $\pm 0.0001$ gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	10 gm - $\pm 0.01$ gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semi-annually
Refrigerators, Incubators, and Water Baths	•Maintenance service	Determined by twice daily temperature performance checks @ least 4 hours apart, when in use.
Water Bath	•Check thermometer vs. NIST traceable	Annually
Water Bath	•Remove from service when not maintaining temperature and send off for repair or replace	As needed
Autoclave	•Check sterilization efficiency	Monthly – Geobacillus stearothermophilus ampoule
Autoclave	•Check sterilization efficiency	With each use– Chemical Indicator Strip
Conductivity Meter	•Calibrate and clean probe	As needed
Conductivity Meter	•Replace or replatinize probe	When poor response not corrected by above
pH	Automatic Temperature Compensation of pH probe	Annually
Stereoscope	• Clean optics and stage	Each Use
pH Meters	•Reference junction & electrode replacement	As needed
pH Meters	•Probe stored in pH 4.0 Buffer	At all times when not in use.
pH Meters	•Other	As described in the manufacturer's O & M manual
Autoclave	•Check timing device	Quarterly
pH meter	•Calibrate and check slope (per manufacturer)	Daily
Quanti-Tray Sealer	•Check sealer for leaks	Monthly
Water Purifier	•Conductivity check using a calibrated conductivity meter	Monthly
Water Purifier	•Check for TOCs, ammonia, nitrogen, TRC and heterotrophic bacteria	Monthly
Water Purifier	•Check for single and heavy total metals	Annually
Incubators and Water Baths	Perform temperature stability and load testing	Annually
Autoclave	•Check pressure (annual contract maintenance)	Annually
Autoclave	Check mechanical timing device	Quarterly
Stereoscope	• Clean optics and stage; microscope alignment (annual maintenance contract)	Annually

### 8.3 S TANDARDS AND REAGENTS

All reagents and standards must meet the requirements listed in the analytical methods.

<b>Table 8.3A: Commercially prepared agar/broth, reagent sources, and storage information. (subject to revision as needed)</b>		
<i>Agar Type</i>	<i>Source</i>	<i>Storage</i>
M-FC Broth w/ Rosolic acid	Millipore	4 ± 2°C
A-1 Media (broth)	Hach	4 ± 2°C
mEndo Broth	Hach	4 ± 2°C
Lauryl Tryptose Broth	Hach	4 ± 2°C
Brilliant Green Lactose Broth	Hach	4 ± 2°C
EC media w/ mug broth	Hach	4 ± 2°C
HPC	Hach	4 ± 2°C
Colilert reagent powder	IDEXX	Room temp
Enterolert reagent powder	IDEXX	Room temp
Phosphate Buffer Solution	Weber Scientific	Room temp

All stock agar expirations are per manufacturer specification.

<b>Table 8.3B: In-house prepared agar/broth, reagent sources, and storage information. (subject to revision as needed)</b>						
<i>Agar Type-Stock</i>	<i>Source</i>	<i>Stock Storage</i>	<i>Stock Expiration</i>	<i>Preparation Components Media</i>	<i>Prepared Storage</i>	<i>Prepared Expiration</i>
Plate Count Agar	VWRDifco	Room Temp	As specified by Manufacturer	PCA + Water	4 ± 2°C	3 months
Tryptic Soy Agar	VWRDifco	Room Temp	As specified by Manufacturer	TSA + Water	4 ± 2°C	3 months
Tryptic Soy Broth (TSB)	VWRDifco	Room Temp	As specified by Manufacturer	TSB + Water	4 ± 2°C	3 months
Lauryl Tryptose Broth (LTB)	VWRDifco	Room Temp	As specified by Manufacturer	LTB + Water	4 ± 2°C	3 months
Buffered Rinse Water	VWRDifco	4 ± 2°C	As specified by Manufacturer	KH <sub>2</sub> PO <sub>4</sub> + MgCl <sub>2</sub> +Water	Room temp.	1 year
Heart Infusion Agar	VWR/BD	Room Temp	As specified by Manufacturer	HIA + Water	4 ± 2°C	2 weeks

### Membrane Filters and Pads

Membrane filters and pads are purchased and certified to meet the following specifications:

- Filter diameter - 47 mm, mean pore diameter - 0.45  $\mu\text{m}$ . Alternate filter and pore sizes may be used if the manufacturer provides data verifying performance equal to or better than that of 47mm-diam, 0.45- $\mu\text{m}$ -pore size filter. At least 70% of filter area must be pores.
- When filters are floated on reagent water, the water diffuses uniformly through the filters in 15s with no dry spots on the filters.
- Flow rates are at least 55 mL/min/cm<sup>2</sup> at 25°C and a differential pressure of 93kPa.
- Filters are nontoxic, free of bacterial-growth-inhibiting or stimulating substances, and free of materials that directly or indirectly interfere with bacterial indicator systems in the media. Ink grid is nontoxic. The arithmetic mean of five counts on filters must be at least 90% of the arithmetic mean of the counts on five agar spread plates using the same sample volumes and agar media.
- Filters retain the organisms from a 100mL suspension of *Serratia marcescens* containing  $1 \times 10^3$  cells.
- Water extractables in filters do not exceed 2.5% after the membrane is boiled in 100mL reagent water for 20min, dried, cooled, and brought to constant weight.
- Absorbent pad has diameter 47mm, thickness 0.8mm, and is capable of absorbing  $2.0 \pm 0.2\text{mL}$  Endo broth.
- Pads release less than 1mg total acidity calculated as CaCO<sub>3</sub> when titrated to the phenolphthalein endpoint with 0.02N NaOH.
- If the filter and absorbent pad are not sterile, they should not be degraded by sterilization at 121°C for 10min. Confirm sterility by absence of growth when a membrane filter is placed on a pad saturated with tryptic soy broth and incubated at  $35 \pm 0.5^\circ\text{C}$  for 24h.

## 8.4 I INSTRUMENT CALIBRATION

### Autoclave

Prior to first use, autoclaves must be initially evaluated for performance. All initial checks must be recorded and records must be retained on file. With each use, a record of items sterilized, temperature, pressure, and time is kept for each batch processed.

Operating temperature is checked and recorded at least weekly with a minimum/maximum thermometer. Performance is tested monthly with *Bacillus stearothermophilus* ampoules. Chemical strips are used with each use to verify that supplies and materials in each cycle have been sterilized. The autoclave mechanical timing device is checked quarterly against a stop watch and actual time elapsed documented. Records of autoclave operations are maintained for every cycle. Records include: date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time (may be recorded as time in and time out) and analyst's initials.

### **Quebecor Colony counter**

A dark field colony counter is used to count Heterotrophic Plate Count colonies. Maintenance is performed per manufacturer's instructions.

### **Quanti-tray Sealer**

The Quanti-tray sealer is checked monthly using 100mL of bromocresol purple, or equivalent dye. The solution is poured into a test tray, sealed, and tested for leaks.

### **pH Meter/Conductivity Meter**

With each use, calibrate the instrument according to the manufacturer's instructions. Verify that the slope of the calibration is within the manufacturer's acceptable range prior to use. Automatic temperature compensation (ATC) verifications are performed annually on the pH probe.

### **Incubators & Waterbaths**

Records of temperature checks are documented twice daily at least 4 hours apart when in use. Thermometers used for temperature checks are verified at least annually. Temperature stability and load testing is performed on an annual basis.

### **Analytical Balances**

Analytical balances are checked at least daily prior to each use with class "I" weights. Records of these verifications are maintained within the laboratory. Balances are also serviced and verified and/or calibrated by an external calibration service at least semi-annually.

### **Volumetric Equipment, IDEXX and Commercially Prepared Phosphate Buffer Bottles**

Equipment such as filter funnels, bottles, pipettes, non-Class A glassware and other containers with graduation must be calibrated once per lot prior to the first use. Mechanical hand pipettes, automatic dispensers and diluters are verified for accuracy quarterly. The error of calibration must not exceed 3%.

### **IDEXX Bottles and Quanti-trays**

Prior to first use, IDEXX bottles and Quanti-trays must be checked for fluorescence using a long wave UV light.

### Ultraviolet Lamp

The output of the UV lamp used to measure fluorescence for the identification of *E. coli* is tested quarterly with a UV light meter. The UV bulbs are replaced if the output is less than 70% of the original.

## 9.0 LABORATORY PRACTICES

### 9.1 REAGENT GRADE WATER

Reagent Grade water –Type II used in the Microbiology Laboratory is periodically checked for contamination. Type II water is checked annually for single and total heavy metals. Monthly checks for total organic carbon, ammonia and organic nitrogen, total residual chlorine and a heterotrophic plate count are also conducted. Resistivity and pH are checked continuously or with each use. Conductivity is also checked monthly using a calibrated conductivity meter. The Use test is performed quarterly and the Water Suitability test is performed annually.

### 9.2 GLASSWARE WASHING , STERILIZATION PROCEDURES AND EQUIPMENT STERILITY CHECKS

Glassware washing and preparation/sterilization procedures are performed according to EPA guidelines and are outlined in *SOP 030701 Glassware Cleaning and SOP 350335, Quality Control and Quality Assurance of Microbiological Equipment and Testing Materials*. Before use, examine and discard items with chipped edges or etched inner surfaces. Reusable glassware is cleaned using the protocol established by the EPA:

- Soak for 15 minutes in hot tap water with detergent and scrub. Rinse thoroughly with tap water. Rinse thoroughly with dilute nitric acid (10%). Rinse thoroughly with deionized water. Rinse thoroughly with pesticide grade acetone. Rinse well with deionized water.
- New glassware are cleaned according to the same procedure as listed above except the first step is preceded by soaking overnight in 10 % HNO<sub>3</sub>.

Inspect glassware after washing for excessive water beading and rewash, if necessary. Perform checks on pH and test for inhibitory residues on glassware and plastic ware. Use utensils and containers of borosilicate glass, stainless steel, aluminum, or other corrosion resistant material for media preparation. All biological glassware is purchased pre-sterilized. In-house sterilization of any auxiliary equipment is performed via autoclave.

Pipettes of all sizes are checked for sterility by drawing up non-selective media into the pipette and re-dispensing the volume back into original tube that contained the media. The tube is then incubated and monitored for growth. All results are recorded and maintained within the laboratory.

Inoculating loops are cultured by aseptically transferring the entire tip of the loop into a tube containing non-selective media. The tube is incubated and monitored for growth. Results are maintained within the laboratory.

A sterility check is performed on one container for each lot of purchased, pre-sterilized sample containers, and IDEXX containers. Results are maintained within the laboratory.

A check is performed on one container from each new lot of microbiological sample containers to ensure efficacy of sodium thiosulfate to 15 mg/L chlorine, and documented. A sterility check is performed on each batch of dilution and rinse water prepared in the laboratory and on each batch of commercially prepared water with non-selective growth media prior to first use.

In addition, stock solutions used for preparing rinse water are checked for turbidity prior to each use. If turbid, the stock buffer is discarded or re-sterilized.

### **9.3 MEDIA STERILITY VERIFICATION PROCEDURES**

A sterility check must be analyzed for each lot of pre-prepared media and for each lot of media prepared in the laboratory. This is done prior to the first use of the media used for membrane filtration, MPN, pour plate and chromofluorogenic methods. For media used in the pour plate analytical technique, sterility blanks of the media must be made by pouring an uninoculated plate for each run in addition to sterility and lot comparison tests being performed on each lot prior to first use. Reagents and containers used in chromofluorogenic method tests are checked for fluorescence prior to first use. All results of the sterility and lot comparison tests are documented.

### **9.4 POSITIVE AND NEGATIVE CONTROLS USING PURE CULTURES**

#### **ATCC Pure Cultures**

Positive culture controls demonstrate that the media can support the growth of the target organism(s), and that the media produces the specified or expected reaction to the target organism(s). All media must be tested with at least one pure culture of a known positive reaction. This must be done prior to first use of the media.

Negative culture controls demonstrate that the media does not support the growth of non-target organisms or does not demonstrate the typical positive reaction of the target organism(s). All batches of selective media in the laboratory must be analyzed with one or more known negative culture controls. This must be done prior to first use of the media.

## 10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOPs associated with the microbiology laboratory can be found in the following table:

**TABLE 10.1: MICROBIOLOGICAL DEPARTMENT SOPs**

*This Table is subject to revision without notice*

SOP #	Title/Description
350305	Fecal Coliform: Membrane Filter Technique (SM 9222D)
350315	Fecal Coliform Determination in Biosolids: Membrane Filter Technique (SM 9222D)
350316	Total Coliform (SM 9222B)
350321	Pocket Colorimeter Chlorine Tester Maintenance and Calibration
350322	DO Meter Maintenance and Calibration
350323	Fluke Thermometer Operation and Maintenance
350324	Digital Light Meter Maintenance and Method of Operation
350325	PH Meter Maintenance and Calibration
350326	Thermometer Operation, Maintenance and Calibration Procedure
350328	Conductivity Meter Maintenance and Calibration
350332	Laboratory Maintenance of Bacteria Reference Cultures
350333	QA/QC of Microbiological Equipment and Testing Materials
350343	Colilert (SM 9223B)
350348	Enterolert (ASTM 6503-99)
350354	HPC (SM 9215 B)
350355	Technical Training and Personnel Qualification for Biomonitoring-Microbiology
350356	Water bath and Incubator Temperature Stability and Load Testing
350359	Calibration and Maintenance of Autoclaves
350369	Sterilization, Sanitization and Residue Testing of Microbiological Glassware and Equipment
350380	Class "A" MPN Fecal Coliform Analysis (SM 9221E/C)
350381	Fecal Coliforms in Sewage Sludge (Biosolids) by MPN Fermentation using A-1 medium (EPA Method 1681)

10.2 Additional information regarding microbiological testing can be found in:

- Standard Methods for the Examination of Water and Wastewater, Sections 9020 through 9050.
- Heterotrophic Plate Count, SM 9215B
- Fecal Coliform Direct Test (A-1 Media), SM 9221E
- Standard Total Coliform Membrane Filter Procedure, SM 9222B.
- Fecal Coliform Membrane Filter Procedure, SM 9222D.
- Enzyme Substrate Test, SM 9223B.
- Environmental Regulations and Technology, Control of Pathogens and Vector Attraction in Sewage Sludge, Appendix F.
- Fecal Coliforms in Sewage Sludge, EPA 821-R-06-013



## 11.0 QUALITY CONTROL CHECKS

- 11.1 ESC participates in microbiological proficiency testing (PTs) in various matrices by analyzing samples provided by Phenova. Blind samples are received and analyzed according to instructions from Phenova and the standard operating procedure.
- 11.2 Plate count comparison between two analysts is conducted monthly. Acceptable plate count comparisons must be within 10%. Analyst deviations that are outside the 10% range are repeated. If the repeat inter-analyst count is unacceptable, additional procedural training and method reviews are conducted.
- 11.3 Duplicate analyses are performed on 10% of samples or at least one sample per month for total and fecal coliform and *E. coli* tests. Due to the infrequent laboratory receipt of some samples, duplicate analyses are conducted per sample. If the RPD exceeds 20%, the data is qualified.
- 11.4 For membrane filtration analyses, sterility control checks are conducted on the filter assembly at the beginning and end of each sequence and following every 10 samples analyzed. If QC blanks fails, the run is rejected or qualified.
- 11.5 Verification of total coliform and fecal coliform colonies must be conducted monthly (10 colonies/month for wastewater). Colonies found in drinking water samples must have at least five typical sheen colonies and five atypical colonies verified.
- 11.6 For HPC analysis, duplicate plates are run for each dilution. A positive control and an uninoculated plate performed for each run. If the QC fails, the run is rejected and qualified, and sample re-collected.
- 11.7 Duplicate counts are performed monthly for colony counts from membrane filtration or pour plated media on one positive sample for each month the test is performed. Each analyst counts typical colonies on the same plate and counts must be within 10% difference to be acceptable, if the laboratory has two or more analysts. The same plate is counted twice by the analyst and difference between counts must be no more than 5% in laboratories with only one analyst.
- 11.8 For biosolids testing, an Initial Precision and Recovery test (IPR) is performed prior to the first time the method is used and at any time the method or instrumentation is modified. The IPR consists of four Milorganite® samples spiked with *E. coli* (ATCC #25922), and must be accompanied by an acceptable method blank and appropriate sterility checks. Mean percent recoveries from the IPR must fall within the EPA approved QC limits of 1-312%, and Relative Standard Deviation of the recovery should be less than or equal to 96%.
- 11.9 For biosolids testing, an Ongoing Precision and Recovery sample (OPR) is analyzed after every 20 field and matrix spike samples, or one per week that samples are analyzed, whichever occurs more frequently. The OPR consists of one Milorganite® sample spiked with *E. coli* (ATCC #25922), and must be accompanied by an acceptable method blank and appropriate sterility checks. Recoveries from the OPR must fall within the EPA approved QC limits of 1- 371%.

- 11.10 For biosolids testing, a Method blank is analyzed everyday samples are processed. The Method Blank must be free of contamination from the target organism, and serve as a sterility control to verify the sterility of equipment, materials, and supplies.
- 11.11 For biosolids testing, a Matrix Spike (MS) is analyzed when samples are first received from a source from which the laboratory has not previously analyzed samples and subsequently, 5% of field samples to determine the effect of a particular matrix on fecal coliform recoveries. MS samples must be accompanied by the analysis of an unspiked field sample sequentially collected from the same sampling site, an acceptable method blank, media sterility checks, and an OPR sample if possible. MS percent recoveries must fall within the EPA approved QC limits: Class A Biosolid= 2-541%; Class B Biosolid= >0-6172%, and RSD less than or equal to 182% and 184% for Class A and Class B biosolids, respectively.
- 11.12 For biosolids testing, control charts for OPR, IPR, and MS are charted and maintained in the laboratory. The control charts graphically display the results of continuing performance when using Method 1681.

## ***12.0 DATA REDUCTION, VALIDATION AND REPORTING***

### **12.1 D ATA REDUCTION**

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in *SOP 030201 Data Handling and Reporting*. The primary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP has been followed
- Sample preparation is correct and complete
- Analytical results are correct and complete
- QC is within criteria and complete

### **12.2 V ALIDATION**

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by reviewing all data entries and calculations for errors, reviewing all documentation to assure that sample information is correct, and that the tests have been performed appropriately and within the appropriate holding times. The secondary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP has been followed
- Sample preparation is correct and complete
- Analytical results are correct and complete

## 12.3 REPORTING

Reporting procedures are documented in *SOP 030201 Data Handling and Reporting*. Microbiological data is reported as Colony Forming Units (CFU) per unit volume, Presence/Absence, or Most Probable Number (MPN)/100mL.

## 13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CARs are kept on file by the Regulatory Affairs Department. Corrective action procedures are documented in *SOP #030208, Corrective and Preventive Action*

### 13.2 Required Corrective Action

All samples and procedures are governed by ESC's quality assurance program. Designated corrective actions are as follows:

#### 13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria takes precedence.

#### 13.2.2 Out of control plate count comparisons between analysts.

Rejection Criteria – Comparisons must be within  $\pm 10\%$  for monthly plate count comparisons.

Corrective Action – Duplicate counts are repeated. If repeat counts are still beyond acceptance range, procedural training and method reviews are conducted.

#### 13.2.3 Out of control duplicate analyses for total and/or fecal coliform or *E. coli*.

Rejection Criteria – Duplicate RPDs must not exceed 20% for total and/or fecal coliform or *E. coli*.

Corrective Action – Data is qualified or the analysis is repeated. If repeat analysis is still beyond acceptance range, procedural training and method reviews are conducted.

#### 13.2.4 Out of control QC blank for membrane filtration analysis.

Rejection Criteria – Blank analyses performed either at the beginning or end of the analytical sequence is positive.

Corrective Action – The analytical sequence may be rejected and reprocessed or qualified based on the nature of the contamination.

#### 13.2.5 Out of Control QC Blank for HPC analysis

Rejection Criteria - Blank analysis performed during each run is positive for growth.

Corrective Action - The analytical run is rejected, and data qualified with an “R” for rejected data, and sample re-collected.

#### 13.2.6 Out of control IPR analyses

Rejection Criteria - Recoveries from IPR fall outside of the required range for recovery: 1 - 312%, and RSD of 96%.

Corrective Action - Identify the problem by evaluating each step in the analytical process, media, reagents, and controls, correct the problem and repeat the IPR.

#### 13.2.7 Out of Control OPR analyses

Rejection Criteria - Recoveries from OPR fall outside of the required range for recoveries: 1-371%.

Corrective Action - Identify the problem by evaluating each step in the analytical process, media, reagents, and controls, correct the problem and repeat the OPR.

#### 13.3.8 Out of Control MS analyses

Rejection Criteria - Recoveries from MS fall outside of the required range for recoveries: Class A Biosolid= 2-541%; Class B Biosolid= >0-6172%, and RSD less than or equal to 182% and 184% for Class A and Class B biosolids, respectively.

Corrective Action - Flag all associated filed data.

### **14.0 RECORD KEEPING**

Record keeping is outlined in SOP #030230, *Standards Logger*, SOP #030227, *Data Review* and SOP #030201, *Data Handling and Reporting*

### **15.0 QUALITY AUDITS**

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 13.0 and SOP #010104, *Internal Audits*.

## 16.0 REVISIONS

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of this appendix are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0 (Appendix X)	Section 7.1 – Updated holding time of HPC and other water samples to 8h. Section 8.1 – Updated equipment list Section 8.2 – Clarified frequency of preventative maintenance Table 8.3B – Changed source of some agars and broths Section 16 – New section for summary of revisions to previous version

1.0 SIGNATORY APPROVALS

# Mold/BOD Laboratory QUALITY ASSURANCE MANUAL

## APPENDIX XI TO THE ESC QUALITY ASSURANCE MANUAL

for


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Prepared by


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**NOTE: The QAM has been approved by the following people.**

  
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### **3.0 SCOPE AND APPLICATION**

This appendix discusses specific QA requirements for general analytical protocols to ensure that analytical data generated from the Mold laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

### **4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES**

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling guidance and materials and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in Section 4.0 in the *ESC Quality Assurance Manual*.

### **5.0 PERSONNEL AND TRAINING**

#### **5.1 PERSONNEL**

Dr. Christabel Fernandes-Monteiro, with a Ph.D. in Applied Biology, is the Department Manager for Biology. She oversees supervision of laboratory operations in the Mold, Aquatic Toxicity, Microbiology, BOD and Protozoan laboratories. Her responsibilities include assurance of reliable data through monitoring of quality control, corroborating the analysis performed, protocol development, coordination with clients regarding sample analysis, scheduling of tests and overall production in all sections within the Biology Laboratory, including management of staff. Dr. Fernandes-Monteiro oversees the review and approval processes of all data associated with all Biological laboratory sections. She gained experience in Mold analytical techniques at ESC, an AIHA accredited laboratory, and obtained additional training in microscopic techniques at the McCrone Research Institute. She also reviews AIHA and EPA online training modules related to the methods being performed in the Mold and BOD Laboratory. In her absence, Bridget Miller assumes responsibility for Mold/BOD departmental decisions.

Bridget Miller, with a BS degree in Biology, is the Primary Analyst in the Mold and BOD laboratory. She is proficient in Mold analytical methods as per AIHA guidelines. Bridget has gained analytical experience at ESC, an AIHA accredited laboratory, and obtained additional training in Mold analysis at the McCrone Research Institute. She reviews AIHA and EPA online training modules related to the methods being performed in the Mold and BOD Laboratory.



## 5.2 T TRAINING

All new analysts to the laboratory are trained by the Primary Analyst or Manager according to ESC protocol. ESC's training program is outlined in SOP #350355, *Technical Training and Personnel Qualification for Biomonitoring-Mold*. Analyst performance in the Mold/BOD Laboratory is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in mold analysis is demonstrated by acceptable participation in the AIHA proficiency testing programs (EMPAT-Direct Exam and EMPAT-Bacterial/Fungal), Round Robin analysis and daily Quality Control sample analysis. On-going acceptable capability in BOD analysis is demonstrated by acceptable participation in the Phenova proficiency testing program and daily Quality Control sample analyses. Documentation of analyst training, including a copy of college transcripts or degree, is maintained on file within the department.

## 6.0 FACILITIES AND LABORATORY SAFETY

### 6.1 FACILITIES

#### MOLD LAB

The main area of the Mold laboratory has approximately 532 square feet with 167 square feet of bench space. The lighting throughout the laboratory is fluorescence. The air system is a five-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the ELGA PureLab Ultra deionizer system. Biohazard containers are located in the laboratory and Stericycle Waste Removal serves as ESC'S biological waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

#### BOD LAB

The main area of the BOD laboratory has approximately 532 square feet of area with 151 square feet of bench space. The lighting standard throughout the laboratory is fluorescence. The air system is a five-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the ELGA PureLab Ultra deionizer system. Biohazard containers are located in the laboratory and Stericycle Waste Removal serves as ESC'S biological waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

### 6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where infectious aerosols or splashes may occur are conducted in biological safety II cabinets.
- The following Biosafety Level 2 (BSL2) guidelines are adhered to:
  - Closed-toe shoes are worn in the laboratory
  - Floors and work surfaces are cleaned on a regular basis
  - Emergency numbers are posted in the laboratory

- Biological safety hoods are tested and certified annually
- Laboratory personnel are trained in the use of the biological spill kit and emergency safety equipment
- ESC’s laboratory safety guidelines are detailed in the ESC *Chemical Hygiene Plan*.

## 7.0 SAMPLING PROCEDURES

### 7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Samples are received in the laboratory login area and are tracked using LIMS (Laboratory Information Management System). A Chain of Custody Form accompanies all samples received by the lab. This is necessary to prove the traceability of the samples and to document the change in possession from sampling to delivery to receipt by the laboratory. Prior to analysis samples are checked for integrity. Sample handling, tracking and acceptance procedures are outlined in SOP #060105, *Sample Receiving*.
- Sample storage procedures are followed using guidance from each approved method and associated department SOP.

## 8.0 EQUIPMENT

### 8.1 EQUIPMENT LIST

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Mold/ BOD Analysis</b>				
<i>This table is subject to revision without notice</i>				
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Serial #</i>	<i>Location</i>
Analytical Balance	Mettler	PL602-S	1125081657	Bacteriology Lab
Analytical Balance	Ohaus	Adventure Pro	8029211055	Bacteriology Lab
Autoclave	Tuttnauer	2540EK	2906170	Bacteriology Lab
Class I BSC	AirFiltronix	AirFiltronix HS 4500	41031	Mold Lab
Class II BSC	Labconco	Labconco 36213	60554894	Mold Lab
Class II BSC	Labconco	Labconco 36209	03076555	Bacteriology Lab
COD Reactor	HACH	45600	900903221	BOD
Microscope	NIKON	LABOPHOT	242008	Mold Lab
Microscope	NIKON	LABOPHOT	235267	Mold Lab
Microscope	Olympus	CH2	900216	Mold Lab
Microscope	Olympus	BH-2	708821	Mold Lab
Microscope	Leitz	Laborlux	512663	Mold Lab
Microscope	VWR Scientific	VWRC1	V167173	Mold Lab
Refrigerator	Whirlpool			Bacteriology Lab
Refrigerator	Whirlpool	EI05PPXMQ	EEP3524864	Mold Lab

<b>LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Mold/ BOD Analysis</b>				
<i>This table is subject to revision without notice</i>				
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>	<i>Serial #</i>	<i>Location</i>
Refrigerator	Whirlpool	EL7ATRRMQ07	EWR4973976	Mold Lab
Refrigerator	Frigidaire	FRT17G4BW9	BA703306	Mold Lab
Stereoscope	VWR Scientific	VWRS1	V168430	Mold Lab
Incubator	Labtronix	BOD2100D	21000010213	Mold Lab
Incubator	Quincy Lab	10-100	I11-2454	Mold Lab
Incubator	Precision Scientific	30M	9303590	Bacteriology Lab
Incubator	Precision Scientific	30M		Bacteriology Lab
Incubator	VWR	2030	802202	BOD
Incubator	Fisher	Not Visible	100212	BOD
Incubator	Thermo Scientific Precision	3271	317217-1241	BOD
Incubator	Precision	818	35AK-10	BOD
Waterbath	Blue M-MagniWhirlpool	MW-1110A	14991	Bacteriology Lab
Waterbath	Precision	Circulating 260	21-AJ11	BOD
Biolog MicroStation	Biolog, Inc.	Microlog 3	342689	Bacteriology Lab
Turbidimeter	Biolog, Inc.	21907	6093898	Bacteriology Lab
Plate Reader	Biotek	ELX808BLG	203222	Bacteriology Lab
Vortex Genie2 Mixer	VWR	G-560	2-223236	Mold Lab
Vortex Genie2 Mixer	VWR	G-560	2-223236	Bacteriology Lab
Stir Plate	Corning	PC-420D	023507102961	Bacteriology Lab
Stir Plate	Fisher	118	102	Bacteriology Lab
Stir Plate	VWR	205	7852	BOD
Stir Plate	VWR	220	5031	BOD
BOD SP Robotic Analyzer	Skalar	SP50	08124	BOD
BOD SP Robotic Analyzer	Skalar	SP50	08123	BOD
DO meter	YSI	5000	081C101451	BOD
DO meter	YSI	5000	081C101450	BOD
pH meter	VWR	Symphony B10P	12284S0009	BOD
Spectrophotometer	Hach	DR 2700	1388224	BOD

## 8.2 E EQUIPMENT PREVENTIVE MAINTENANCE

<i>INSTRUMENT</i>	<i>P. M. DESCRIPTION</i>	<i>FREQUENCY</i>
Analytical Balances	•Check with Class "I" weights	Daily-tolerance 1 gm - ±0.0001gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	10 gm - ±0.01 gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semiannually
Refrigerators, Waterbaths, & Incubators	•Maintenance service	As needed - determined by daily temperature performance checks twice daily and at least 4 hours apart

<i>INSTRUMENT</i>	<i>P. M. DESCRIPTION</i>	<i>FREQUENCY</i>
Water Bath	•Check thermometer vs. NIST	Annually
Water Bath	•Remove from service when not maintaining temperature and send off for repair or replace	As needed
Incubators and Waterbaths	Perform Temperature stability and load testing	Annually
Autoclave	•Check sterilization efficiency	Weekly – <i>G. stearothermophilus</i>
Autoclave	•Check sterilization efficiency	Per Use – Chemical Indicator
Autoclave	Check timing devices	Quarterly
Autoclave	Check pressure (annual Maintenance contract)	Annually
Class II Biosafety Cabinet	•Monitor air and UV lamps	Monthly
Class II Biosafety Cabinet	•Inspect for air flow	Quarterly
Class II Biosafety Cabinet	•Recertification according to NSF standard 49	Annually
Turbidimeter	•Maintenance Service	Annually
Turbidimeter	•Check for accuracy using NIST traceable stds	Per Use
Biolog MicroStation	•Maintenance Service	Annually
Microscope	•Service/calibration of each ocular micrometer	Annually
Microscope	•Clean optics and stage, Kohler Alignment	Each Use
pH meters	Calibrate and check slope (acceptable; range of	Daily
pH meters	Reference junction & electrode replacement	As needed
pH meters	Probe stored in KCl	At all times when not in use
pH meters	ATC checks	Annually
pH meters	Other	As described in manufacturer's O &
BOD SP Robotic Analyzer	Calibrate DO probe	Daily
BOD SP Robotic Analyzer	Clean and Change DO probe membrane	Weekly
BOD SP Robotic Analyzer	Rinse ATU (seed) dispenser using rinse pump option	As needed
BOD SP Robotic Analyzer	Clean rinsing vessel	Every three months or as needed
BOD SP Robotic Analyzer	Replace tubing for dispenser, diluent pump, and rinsing vessel	Annually or as needed

### 8.3 S TANDARDS AND REAGENTS

Table 8.3A lists commercially prepared agar sources. Table 8.3 B lists in-house prepared agar sources and storage information. Table 8.3C lists standard sources, receipt, and preparation information for BOD Analysis. Table 8.3D is designed to provide general calibration range information for BOD analysis. These tables may change depending on regulatory requirements, procedural changes, or project needs.

<i>Agar Type</i>	<i>Source</i>	<i>Storage</i>
Malt Extract Agar w/chloramphenicol (MEA)	HealthLink	4 ± 2°C
DG18 Agar	HealthLink	4 ± 2°C
Modified Cellulose Agar	HealthLink	4 ± 2°C
Tryptic Soy Agar w/Sheep Blood	HealthLink	4 ± 2°C
2 % Malt Extract	Biolog	4 ± 2°C
Biolog Universal Agar (BUG)	Biolog	4 ± 2°C

**Table 8.3A: Commercially prepared agar sources and storage information.**  
*(subject to revision as needed)*

<i>Agar Type</i>	<i>Source</i>	<i>Storage</i>
BUG w/BL	Biolog	4 ± 2°C
Biolog Universal Anaerobic Agar (BUA)	Biolog	4 ± 2°C
BUA w/BL	Biolog	4 ± 2°C
Biolog Universal Yeast Agar (BUY)	Biolog	4 ± 2°C
TSA w/SB contact	HealthLink	4 ± 2°C
BUG w/0.25% Maltose	Biolog	4 ± 2°C
Malt Extract Agar w/chloramphenicol contact	HealthLink	4 ± 2°C
Chocolate Agar	Biolog	4 ± 2°C
Czapek Yeast Extract Agar	HealthLink	4 ± 2°C
CNA w/5 % SB	HealthLink	4 ± 2°C
Saboraud's Dextrose Agar	HealthLink	4 ± 2°C

All stock agar expirations are per manufacturer specification.

**Table 8.3B: In-house prepared agar sources and storage information.**  
*(subject to revision as needed)*

<i>Agar Type-Stock</i>	<i>Source</i>	<i>Stock Storage</i>	<i>Stock Expiration</i>	<i>Preparation Components Media</i>	<i>Prepared Storage</i>	<i>Prepared Expiration</i>
Malt Extract Agar (MEA)	VWR/Difco	Room Temp	As specified by Manufacturer	MEA + Water	4 ± 2°C	2 weeks
Modified Saboraud's Agar (MSA)	VWR/Difco	Room Temp	As specified by Manufacturer	M-SAB Dex + Water	4 ± 2°C	2 weeks
R2A	VWR/Difco	Room Temp	As specified by Manufacturer	R2A + Water	4 ± 2°C	2 weeks
2 % Malt Extract	VWR/Difco	Room Temp	As specified by Manufacturer	Bacteriological Agar + Malt	4 ± 2°C	2 weeks
Biolog Universal Agar (BUG)	Biolog	Room Temp	As specified by Manufacturer	BUG + Water	4 ± 2°C	2 weeks
Biolog Universal Anaerobic Agar (BUA)	Biolog	Room Temp	As specified by Manufacturer	BUA + Water	4 ± 2°C	2 weeks
Biolog Universal Yeast Agar (BUY)	Biolog	Room Temp	As specified by Manufacturer	BUY + Water	4 ± 2°C	2 weeks
Biolog Universal Agar (BUG) with 0.25%	Biolog	Room Temp	As specified by Manufacturer	BUG + Water + Maltose	4 ± 2°C	2 weeks
Anaerobic Agar (ANA)	VWR	Room Temp	As specified by Manufacturer	ANA + water	4 ± 2°C	2 weeks
Tryptic Soy Agar (TSA)	VWR/Difco	Room Temp	As specified by Manufacturer	TSA + Water	4 ± 2°C	2 weeks
Tryptic Soy Broth (TSB)	VWR/Difco	Room Temp	As specified by Manufacturer	TSB + Water	4 ± 2°C	3 months

<b>Table 8.3C: Standard sources, description and calibration information.</b> (This table is subject to revision without notice)						
<i>Instrument Group</i>	<i>Standard Source</i>	<i>How Received</i>	<i>Source/Storage</i>	<i>Preparation from Source</i>	<i>Lab Stock Storage</i>	<i>Preparation Frequency</i>
BOD	Lab preparation	As dry glucose and glutamic acid	Dessicator	150mg each/L	Ambient	Made fresh daily
pH meter	Commercial source	pH 7.0 buffer	Ambient	No prep required	NA	Annual/Expiration Date
pH meter	Commercial source	pH 10.0 buffer	Ambient	No prep required	NA	Annual/Expiration Date
Turbidity meter	Commercial source	Turbidity standard	Ambient	No prep required	NA	Annual/Expiration Date

<b>Table 8.3D: Working Standard Calibration</b>	
<i>Analysis</i>	<i>Calibration Standard</i>
BOD	D.O.- Barometric pressure/temp, Glucose and glutamic acid reference standard

### Source of Fungi

A collection of fungi is maintained in the laboratory as training and reference material. The fungi are isolated from proficiency testing samples, laboratory contaminants and client samples, and stored as Malt Extract Agar slants for 3 months at  $4 \pm 2^{\circ}\text{C}$ . Cultures are sub-cultured every 3 months. Each culture is assigned an accession number, genus, specific epithet, authority, source, and name of collector. Records are maintained in the laboratory in the accession list database.

### Source of Bacteria

A collection of bacteria is maintained in the laboratory as training and reference material. The bacterial strains are purchased from an accredited microbiological supply company and are used as positive and negative reference controls. Alternatively, bacterial strains are collected from proficiency testing samples and laboratory contaminants, and stored as Tryptic Soy Agar slants for 3 months at  $4 \pm 2^{\circ}\text{C}$ .

## **8.4 I INSTRUMENT CALIBRATION**

### Autoclave

Operating temperature is checked and recorded with each use with a minimum/maximum thermometer. Performance is tested weekly with *Bacillus stearothermophilus* ampoules. Chemical strips are used with each batch processed to verify that supplies and materials have been sterilized. Records of autoclave operations are maintained for every cycle. Records include: date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time (may be recorded as time in and time out) and analyst initials.

### **Incubators & Waterbaths**

The record of temperature checks is documented twice daily at least 4 hours apart when in use. Thermometers used for temperature checks are verified at least annually. In addition temperature chart recorders are being used to continuously monitor the temperature in the incubators used for BOD analysis and the BOD Lab.

### **Analytical Balances**

Analytical balances are checked at least daily prior to each use with class “I” weights. Records of these verifications are maintained within the laboratory. Balances are also serviced and verified and/or calibrated by an external calibration service at least semi-annually.

### **Microscope**

A record of cleaning and alignment for each microscope is maintained in the laboratory. Each microscope has an ocular micrometer that is verified annually with a stage micrometer. All microscopes are serviced annually by an external microscope service.

### **Biochemical Oxygen Demand Robotic Analyzer – SOP Number 340303A**

The Dissolved oxygen meter is calibrated according to manufacturer’s instructions with each use. Air calibration is performed on the DO meter probes to correct DO for the ambient temperature and given local barometric pressure. The local barometric pressure is determined from information provided by the National Weather Service for the Nashville International Airport (BNA) by accessing <http://w1.weather.gov/obhistory/KBNA.html>. The air calibration is confirmed daily using the Winkler Test. During the analytical sequence, the calibration stability of the DO probes is verified after every ten samples and at the end of sequence, by the analysis of continuing calibration verification (CCV). If either of the readings differs from the initial readings by more than 0.2 mg DO/L., the instrument automatically re-calibrates the DO meters and re-reads everything after the last passing CCVs.

A laboratory control sample (LCS) is prepared from glucose and glutamic acid, and is analyzed in triplicate exactly like a field sample at the beginning of the workgroup, after every twenty samples throughout the run and at the end of the workgroup, to verify that the analytical process is performing accurately.

### **pH meter**

With each use of pH meters, calibrate the instrument according to manufacturer’s instructions. The slope is documented on a daily basis. Acceptable pH slope range is per the manufacturer’s operating manual. Automatic temperature compensation (ATC) verifications are performed annually on the pH probe.

### Turbidimeter

With each use, calibrate the instrument according to manufacturer's instructions. Adjust transmittance to a 100% using a blank reference test tube. Establish appropriate turbidity range on turbidimeter by adding or subtracting 2% T to the percent transmittance measured with the appropriate turbidity standard.

### Volumetric equipment

Equipment such as pipettes, non-Class A and other containers with graduations are calibrated once per lot prior to first use. Volumetric equipment that is not disposed of after use is calibrated on an annual basis. The error of calibration must not exceed 3%.

### Air Sampler

The air sampling pump used for laboratory environmental monitoring is verified monthly prior to use with a calibrator that is calibrated annually by an ISO 17025 accredited laboratory to ensure its measurement integrity.

## **9.0 LABORATORY PRACTICES**

### **9.1 REAGENT GRADE WATER**

Reagent Grade water –Type II used in the Mold Laboratory is periodically checked for contamination. Type II water is checked annually for single and total heavy metals. Monthly checks for total organic carbon, ammonia and organic nitrogen, total residual chlorine and a heterotrophic plate count are also conducted. Conductivity and pH are checked continuously or with each use. The water suitability test is performed annually and the USE test is performed quarterly.

Prior to first use, a sterility check with non-selective growth media is performed on each batch of reagent water prepared in the laboratory.

### **9.2 GLASSWARE WASHING AND STERILIZATION PROCEDURES**

Glassware washing and preparation/sterilization procedures are performed according to EPA guidelines and are outlined in SOP #030701, *Glassware Cleaning*. The glassware used in the mold laboratory is restricted to microscopic slides, cover slips, and screw capped bottles, vials or flasks for preparation of media. Before use, examine microscope slides, and discard items with chipped edges or etched inner surfaces. Prior to use, clean microscopic slides with 70 % isopropyl alcohol. Examine screw-capped bottles, vials or flasks for chipped inner edges that could leak. Screw-capped bottles, vials or flasks are cleaned using the following protocol:

- Prewash with hot tap water. Wash with hot tap water. Wash with non-foaming powder detergent. Rinse with tap water. Rinse with DI water. Dry and cool.



- New glassware is cleaned according to the same procedure as listed above.

Inspect glassware after washing for excessive water beading and re-wash, if necessary. Perform checks on pH and test for inhibitory residues on glassware and plastic ware. Use utensils and containers of borosilicate glass, stainless steel, aluminum, or other corrosion resistant material for media preparation. Sterilization of any auxiliary equipment is performed via autoclave.

Pipettes of all sizes are checked for sterility by drawing up non-selective media into the pipette and re-dispensing the volume back into original tube that contained the media. The tube is then incubated and monitored for growth. All results are recorded and maintained within the laboratory.

Inoculating loops are cultured by aseptically transferring the entire tip of the loop into a tube containing non-selective media. The tube is incubated and monitored for growth. Results are maintained within the laboratory.

BOD analysis is performed in disposable, pre-sterilized bottles. In the event that glass bottles must be used, the BOD glassware is washed in a commercial laboratory dishwasher using a phosphate free detergent, followed by a nitric acid rinse, with a final rinse of laboratory DI water.

### **9.3 MEDIA STERILITY VERIFICATION PROCEDURES**

A sterility check must be analyzed for each lot of pre-prepared media and for each lot of media prepared in the laboratory. This is done prior to the first use of the media used for membrane filtration, or MPN, or pour plate, and chromofluorogenic methods. For media used in the pour plate testing technique, sterility blanks of the media must be made by pouring an uninoculated plate for each run in addition to sterility and lot comparison tests performed on each lot prior to first use. All results are documented.

### **9.4 POSITIVE AND NEGATIVE CONTROLS USING PURE CULTURES**

Positive culture controls demonstrate that the media can support the growth of the target organism(s), and that the media produces the specified or expected reaction to the target organism(s). All prepared media must be tested with at least one pure culture of a known positive reaction. This must be done prior to first use of the media.

Negative culture controls demonstrate that the media does not support the growth of non-target organisms or does not demonstrate the typical positive reaction of the target organism(s). All batches of prepared selective media in the laboratory must be analyzed with one or more known negative culture controls. This must be done prior to first use of the media.

New lots of commercially-prepared media are evaluated for suitability using a known positive and negative culture prior to use.

## 10.0 ANALYTICAL PROCEDURES

A list of laboratory SOPs associated with the Mold and BOD laboratory can be found in the following table:

**TABLE 10.1: MOLD DEPARTMENT SOPs**

*This Table is subject to revision without notice*

SOP #	Title
340303	Biochemical Oxygen Demand
350306	Spore Traps
350307	Fungal Andersen
350308	Fungal Quantification
350309	Fungal RODAC
350310	Direct Exam Prep Procedure
350311	Fungal Identification
350312	Mold QA/QC
350313	Mold Lab Safety
350314	MUG – E. coli/Coliforms/Enterococcus
350319	Processing of Bacterial Andersen Samples for Quantification
350334	Microscope Usage
350335	Fungal Spore Identification
350342	BART Testing
350347	Processing of Bacterial Swabs, Bulk, Dust and Water Samples for Quantification
350349	Bacterial Identification Using Biolog
350351	Evaluation of Cosmetic Products
350352	Anaerobic Plate Count
350357	Actinomycetes Identification
350367	Labconco Flaskscrubber Operation and Maintenance
350370	Preparation of Culture Media
350371	Mold lab Autoclave Maintenance and Operation
350379	Mold Lab Reference Culture Maintenance

## 11.0 QUALITY CONTROL CHECKS

11.1 ESC participates in proficiency testing (PTs) in support of various laboratory accreditations/recognitions. For Mold analyses, PTs are administered quarterly by AIHA. ESC participates in both the EMPAT Fungal Direct Examination and Bacterial/Fungal Culturable proficiency testing. The samples are received and analyzed by method according to the vendor's instructions and according to the applicable analytical SOP.

For BOD analysis, environmental PTs are purchased from Phenova. The WP studies are completed every 6 months.

- 11.2 As part of the total spore analysis QC, the laboratory maintains a slide collection with various count levels and genera/groups of spores. Acceptance criteria for the slide collection include counts that are statistically determined (e.g.  $\pm 3\text{STD}$ ). Each analyst reviews one slide from this collection on each day of analysis. The slides are reviewed on a rotational basis such that a different slide is reviewed each day until the entire slide collection has been examined. The total spore count and acceptance criteria for each slide are calculated and compared with the statistically determined acceptance criteria.
- 11.3 Each week, a different pure culture is chosen from the culture collection and is identified by an analyst as part of training and continuing QC program.
- 11.4 Inter- and intra-analyst precision is determined by the re-analysis of samples by the same and different analysts (where possible). The rate of re-analysis by the same analyst (intra-analyst) and by a second qualified analyst (inter-analyst) is 5%, or at least one each month samples are received, for each field of testing. The laboratory uses control charts to compare the intra- and inter-analyst performance to an established control limit.
- 11.5 Media blanks for viable count analysis are used to monitor media and laboratory procedures for contamination. These blanks are utilized in two ways:
- Laboratory media blanks are unexposed fresh media (either recently received from the manufacturer or newly laboratory prepared) that is incubated under the same conditions as those used for analysis.
  - Field blanks are unopened media that is handled identically to field samples. These samplers are returned to the laboratory with sampled media to demonstrate that media utilized was not originally contaminated and did not become contaminated during transport.
- 11.6 Environmental monitoring of the laboratory air and the surfaces in the Mold laboratory is performed monthly. BSLII hoods are also monitored in the Mold laboratory.
- 11.7 Round Robin studies are performed for direct examination of fungal air samples in accordance with AIHA policy requirements. Results for these studies include raw counts and final concentrations for each fungal structure. Acceptance criteria include organism identification, ranking and quantification.
- 11.8 Analysts also participate in other continuing education activities, including attending seminars and conferences, in-house training meetings, reviews of journal publications and self-taught training on CD.
- 11.9 For BOD analysis, Initial Demonstrations of Capability (IDOCs) are performed during new analyst training and/or prior to acceptance and use of any new or modified method/instrumentation. Continuing Demonstration of Capability must be updated at least annually. The associated data is filed within the department and available for review.

- 11.10 For BOD analysis, samples are analyzed in batches of 1-20 samples. Each batch must include the following: method blank, seed blank, seed control, seed check, a laboratory control sample run in triplicate, 1 sample duplicate/ 10 samples. A calibration check (CCV) is performed every 10 samples and an additional LCS every twenty samples including the end of the sequence.
- 11.10.1 A method blank is analyzed for each probe at the beginning and end of the sequence. The method blank is used to define the level of laboratory background and reagent contamination. All blanks must meet method acceptance criteria. If method blanks fail, data is qualified. The depletion of the method blank must be  $< 0.20$ mg but no lower than  $-0.20$  mg. Multiple dilution water blanks in the same batch using the same dilution water are treated as replicates and averaged. The average of the dilution water blanks in a batch must not be more than 0.20 mg/L.
- 11.10.2 The Seed Blank/Seed Control/Seed Check must deplete to show that the microorganism population is viable. The seed correction factor should be 0.6-1 mg/L
- 11.10.3 The CCV should not vary more than 0.2g DO/L within a run.
- 11.10.4 The BOD value for the LCS must be within 167.5 and 228.5.
- 11.10.5 The RPD for the sample duplicate must be  $<30\%$  for high and low.

## ***12.0 DATA REDUCTION, VALIDATION AND REPORTING***

### **12.1 DATA REDUCTION**

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in SOP #030201, *Data Handling and Reporting* and ESC SOP# 030227, *Data Review*. The primary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP is followed
- Sample preparation is correct and complete
- Analytical results are correct and complete
- QC is within criteria and complete

For BOD analysis, the Laboratory Manager or Senior Analyst performs the secondary review of the data package using the ESC SOP# 030227, *Data Review*. The reviewer verifies that the analysis is performed as required and meets method criteria, All associated data is present and complete, and also ensures that any additional documentation is completed as required (i.e. required qualifiers on test reports, case narratives, etc.)

**TABLE 12.1 Data Reduction Formulas**

PARAMETER	FORMULA
Non-viable (Spore Traps) Mold	$\frac{\text{Spore Count}}{m^3} = \frac{\text{number on trace} \times 1000}{\text{Volume of air sampled in liters}}$
Andersen Fungal Viable (Culturable) Mold Spore Andersen Bacterial Viable (Culturable) Bacteria	$\frac{CFU}{m^3} = \frac{\text{raw counts} \times 1000}{\text{Volume of air sampled in liters}}$ $P_c = N [1/N + 1/N-1 + 1/N-2 + \dots + 1/N-r + 1]$
Quantitative Fungal/Bacterial	$\frac{CFU}{gm} \text{ or } \frac{CFU}{\text{Swab}} = \frac{\# \text{ of Colonies} \times \text{Dilution Factor}}{\text{Sample Amount}}$
BOD, 5-DAY	$\frac{\text{Initial D.O.} - \text{Final D.O.} - CF}{\% \text{ Dilution Sample}}$ <i>Calculations are performed by computer software</i> $CF = (\text{Depletion of Seed Control or Seed Check}) \times (\text{Vol of Seed in Samples}) / \text{Volume of Seed in Seed Control or Seed Check}$
Percent Recovery (%R)	$\%R = \frac{(\text{Observed Value}) \times (100\%)}{\text{True Value}}$
Relative Percent Difference (RPD)	$RPD = \frac{[ABS(\text{Result1} - \text{Result2})] \times (100\%)}{\text{Mean Result}}$
Reporting Limit (RL)	$RL (1 \text{ ppm}) \times \text{Final Volume (300 ml)} / \text{Initial volume} \times \text{Dilution Factor}$

## 12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by reviewing all data entries and calculations for errors, reviewing all documentation to assure that sample information is correct, and that the tests have been performed appropriately and within the appropriate holding times. The secondary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP is followed
- Sample preparation is correct and complete
- Analytical results are correct and complete

For BOD analysis, once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.3 for current QC targets, controls and current reporting limits for BOD analysis.

## 12.3 REPORTING

Reporting procedures are documented in SOP #030201, *Data Handling and Reporting*.

*BOD Control Limits:* BOD QC targets are statutory. The laboratory calculated limits verify the validity of the regulatory limits. The BOD QC targets are within the range of 5 to 15% for accuracy, depending on determinative method requirements, and, where applicable, <30% RPD for precision, unless laboratory-generated data indicate that tighter control limits can be routinely achieved. When using a certified reference material for QC sample analysis, the acceptance limits used in the laboratory conform to the provider's certified ranges for accuracy and precision.

<b>Table 12.3: QC Targets for BOD Lab Accuracy (LCS), Precision and RLs</b>					
<i>This table is subject to revision without notice</i>					
<b>Analyte</b>	<b>Analysis Method</b>	<b>Matrix</b>	<b>Accuracy Range (%)</b>	<b>Precision (RPD)</b>	<b>RL (ppm)</b>
Biochemical Oxygen Demand	SM5210B	W	85-115	≤30	1
Biochemical Oxygen Demand - Carbonaceous	SM5210B	W	85-115	≤30	1

### 13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CARs are kept on file by the Regulatory Affairs Department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

#### 13.2 Required Corrective Action

Control limits are established for each type of analysis. When these control limits are exceeded, corrective action must be taken.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are followed; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate SOP.

##### 13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria takes precedence.

##### 13.2.2 Out of Control RPD for inter- and/or intra-analyst reanalysis.

Rejection Criteria - RPD value of the original analysis is calculated and must be below the current control limit.

Corrective Action - Both first and second analysts re-analyze the sample until a consensus is reached and the RPD value falls within control limits.

#### 13.2.3 Out of Control RPD for inter-analyst analysis.

Rejection Criteria – All organisms must be accurately identified.

Corrective Action - Both first and second analysts review the sample. The second analyst results are reported to the client.

#### 13.2.4 Calibration Verification criteria are not met: BOD Analysis

Rejection Criteria see section 8.4

Corrective Action- If the CCV fails, the data may still be used. If the failure persists, check cleanliness of the equipment and stability of the DO probe for subsequent runs. If a problem persists, the group supervisor or Regulatory Affairs Department is notified for further action.

#### 13.2.5 Out of Control Blanks: Applies to Method Blank

Rejection Criteria- Blank depletion is greater than established limit of -0.2 mg and + 0.2 mg/L.

Corrective Action - If the average of the blanks fail, all data must be reported with a qualifier

#### 13.2.6 Out of Control Laboratory Control Standards (LCS)

Rejection Criteria - If the performance of associated laboratory control sample(s) is outside of lab-generated control limits calculated as the mean of at least 20 data points +/- 3 times the standard deviation of those points. (Listed in Section 12).

Corrective Action - All samples bracketed by the failed LCS must be reported with a qualifier.

#### 13.2.7 Out of Control Duplicate Samples

Rejection Criteria - Lab-generated maximum RPD limit (as listed under precision in Section 12)

Corrective Action - The sample and duplicate are reported with a qualifier.

### **14.0 RECORD KEEPING**

Record keeping is outlined in SOP #030230, *Standards Logger*, SOP #030227, *Data Review* and SOP #030201, *Data Handling and Reporting*

## 15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 13.0 and *SOP #010104, Internal Audits*.

## 16.0 REVISIONS

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of this appendix are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0 (Appendix XI)	Table 8.3B – Changed source of some agars and broths Section 8.4 – Clarified that the local barometric pressure is obtained from the airport for BOD Section 10 – Removed SOP# 340303A from SOP list Table 12.1 – Added some data reduction formulas Table 12.3 – Revised the RL for BOD Section 13.2.5 – Clarified MB acceptance criteria for BOD and clarified that if the average of the blanks fail then qualification is needed Section 16 – New section for summary of revisions to previous version



1.0 SIGNATORY APPROVALS

# Protozoa Laboratory QUALITY ASSURANCE MANUAL

## APPENDIX XII TO THE ESC QUALITY ASSURANCE MANUAL


for


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Prepared by

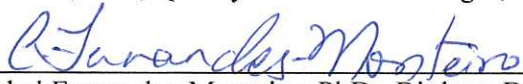
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**NOTE: The QAM has been approved by the following people.**

  
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### **3.0 SCOPE AND APPLICATION**

This manual discusses specific QA requirements for EPA Methods 1622 and 1623 to ensure that analytical data generated from the protozoan laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

### **4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES**

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling guidance and materials and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in Section 4.0 in the *ESC Quality Assurance Manual*.

### **5.0 PERSONNEL AND TRAINING**

#### **5.1 PERSONNEL**

Dr. Christabel Fernandes-Monteiro, with a Ph.D. in Applied Biology, is the Department Manager of Biology. She oversees supervision of laboratory operations in the Mold, Aquatic Toxicity, Microbiology, Protozoan and BOD laboratories. Her responsibilities include assurance of reliable data through monitoring of quality control, corroborating the analysis performed, protocol development, coordination with clients regarding sample analysis, scheduling of tests and overall production in all sections within the Biology Laboratory, including management of staff. In her absence, Stacy Kennedy assumes her responsibilities in the Protozoan laboratory.

Stacy Kennedy, with a M.S. degree in Biotechnology, is the Principal Analyst for the Protozoan laboratory. Ms. Kennedy is proficient in performing EPA Methods 1622 and 1623. She gained analytical experience from a certified EPA Protozoan Principal Analyst and obtained additional training on microscopic techniques. Also, she frequently reviews EPA online training modules related to the methods being performed. In her absence, Becky Rush assumes her responsibilities.

#### **5.2 TRAINING**

The Principal Analyst trains all new analysts in the Protozoan laboratory according to ESC protocol and EPA guidelines. ESC's training program is outlined in SOP #350405, *Training Protocol for Method 1622/1623* and is in accordance with *Supplement 2 to the 5th Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water*. Documentation of training received and authorizations to perform these analyses are maintained within the department.

## 6.0 FACILITIES AND LABORATORY SAFETY

### 6.1 FACILITIES

The main area of the laboratory is approximately 420 square feet and has roughly 67.5 square feet of bench area. The microscope dark room is located in the back of the laboratory is 36 square feet with 18 square feet of bench area. Additionally, there is 40 square feet of storage and fluorescent lighting throughout all areas. The air handling system is a five-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the Siemens Elga UltraPure deionizer system. Biohazard containers are located in the protozoan laboratory and Stericycle serves as ESC's biological waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

### 6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where infectious aerosols or splashes may occur are conducted in Biological Safety II cabinets.
- The following Biosafety Level 2 (BSL2) guidelines are adhered to:
  - Closed-toe shoes are worn in the laboratory
  - Floors and work surfaces are cleaned on a regular basis
  - Emergency numbers are posted in the laboratory
  - Biological safety hoods are tested and certified annually
  - Laboratory personnel are trained in the use of the biological spill kit and emergency safety equipment
- ESC's laboratory safety guidelines are detailed in SOP #350408, *Biosafety Guidelines for the Cryptosporidium Laboratory*.

## 7.0 SAMPLING PROCEDURES

### 7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- A description of field sample collection, containers, storage, temperature, and transport times are located in SOP #350402, *Method 1622/1623 Field-Filtering Sample Collection and Laboratory Delivery* and SOP #350403, *Method 1622/1623 Bulk Sample Collection and Laboratory Delivery*.
- Laboratory sample identification, handling, tracking and the information recording system are found in the following procedures: SOP #350404, *Method 1622/1623 Sample Receiving* and SOP #060105, *Sample Receiving*.

- A Chain of Custody and LT2 Sample Collection Form accompanies all compliance samples received by the lab. This is necessary to prove the traceability of the samples and to document the change in possession from sampling through receipt by the laboratory. Prior to analysis, all samples are checked for integrity.
- Following analysis, the slides are maintained for a minimum of 2 months and disposed of following all State and Federal regulations governing disposal.
- Requirements for sample acceptance are located in SOP #350404, Section 7.0, *Method 1622/1623 Sample Receiving*.

## 8.0 EQUIPMENT

Laboratory equipment specifications are outlined in SOP #350407, *Microscope Analyst Verification*, SOP #350410, *IEC CRU-500 Centrifuge Operation and Maintenance*, SOP #350411, *Lab-Line Multi-Wrist Shaker Operation and Maintenance* and SOP #350413, *Olympus BX40 Microscope Operation and Maintenance*.

### 8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Protozoan		
<i>Item</i>	<i>Manufacturer</i>	<i>Model</i>
Flow control valve	Plast-o-matic	FC050B
Centrifugal pump	Jabsco	18610-0271
Graduated container	Nalgene	20 Liter Carboy
Laboratory shaker	Lab-Line	3587-4
Laboratory shaker side arms	Lab-Line	3589
1500 XG swinging bucket centrifuge	Damon/IEC Division	CRU-5000
Sample mixer/rotator	DYNAL	Cat#: 947.01
Magnetic Particle Concentrator	DYNAL	MPC-1
Magnetic Particle Concentrator	DYNAL	MPC-S
Magnetic Particle Concentrator	DYNAL	MPC-6
Flat-sided sample tubes	DYNAL	Cat#: 740.03
Epifluorescence/differential interference contrast microscope	Olympus	BX-40
Excitation/band pass microscope for fluorescein isothiocyanate (FTIC)	C-Squared	UN3100
Excitation/band pass filters for 4',6-diamidino-2-phenylindole (DAPI)	C-Squared	UN41001

### 8.2 E EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

Calibration of equipment is conducted on an annual and/or semi-annual basis and is documented. Maintenance and cleaning is conducted on an as needed basis or per manufacturer's instructions. Equipment cleaning is specified in SOP #350412, *Cryptosporidium Laboratory Equipment Cleaning*.

<b>PREVENTATIVE MAINTENANCE AND CALIBRATION FOR LABORATORY EQUIPMENT</b>		
<i>INSTRUMENT</i>	<i>P. M. DESCRIPTION</i>	<i>FREQUENCY</i>
Balances (Top Loader or Pan)- capability of detection of 0.1 g for a load of 150 g, and 1 mg for a load of 10 g or less.	Service/Calibration (maintenance and calibration check)	Annually by a qualified independent service tech
	Verified using ASTM Class 1,2, or 3 weights	Monthly
	Non-reference weights should be calibrated	Every six months
pH meter	Electrodes should be maintained	Per manufacturer's instructions
	Slope determination	Monthly (Acceptable slope= 95-105%)
	Meter standardized with pH 7.0, and either 4.0 or 10.0 pH buffers	Each use period
Thermometer- Glass and Electronic	Calibration checked with NIST certified traceable reference thermometer or one traceable to a NIST reference thermometer	Annually
Continuous recording devices	Re-calibrated	Annually
Reference Thermometer	Re-calibrated	Annually by a certified service technician
Autoclave	Maintenance	Annually by a qualified independent technician
	Check Sterilization efficiency	Monthly- Geobacillus stearothermophilus ampoule With each use—Chemical Indicator Strip
	Maximum temperature registering	With each use
	Automatic timing mechanism	Quarterly
	Clean door seals, drain screen, remove debris	As needed
Conductivity Meter	Calibrated using a low level certified traceable standard or determine cell constant	Monthly per manufacturer instructions
Refrigerator	Record temperature	Daily when in use
Micropipettes	Calibrated	Annually
Hand Tally or Digital/Electronic Counter	Checked to confirm accuracy and operational status	Periodically as needed
Centrifuge	Clean and disinfect after spills/leakage	Periodically as needed
	Service/Calibration	Annually by a qualified service technician
Microscope	Service	Annually
	Alignment and adjustment of optics	With each use
	Stage Micrometer calibration	Annually
	Kohler illumination procedure	With each use
DI unit	Manufacturer's instructions	As needed

### 8.3 S TANDARDS AND REAGENTS

**Table 8.3A: Stock solution sources, description and related information.**  
*(subject to revision as needed)*

Description	Vend or	Concentration	Storage Req.	Expiration
Sodium Hydroxide (NaOH)	VWR	Concentrated	ambient	1 year
Hydrochloric Acid (HCl)	VWR	Concentrated	ambient	1 year
Laureth-12	VWR	--	ambient	1 year
Tris Stock	VWR	--	ambient	NA
EDTA	Supelco	0.5 M, pH 8.0	2 - 8°C	1 year
Antifoam A	Supelco	--	ambient	NA
Dynabeads® GC-Combo/Crypto	Idexx	--	2 - 8°C	2 years
Direct labeling kit for det. of oocysts and cysts, Merifluor Cryptosporidium/Giardia	VWR	--	2 - 8°C	1 year
Phosphate Buffered Saline (PBS) Solution, pH 7.4	Supelco	--	ambient	1 year
4', 6-diamidino-2-phenylindole (DAPI) stain	Waterborne, Inc	2mg/mL	2 - 8°C /Darkness	18 months/When positive control fails
Purified, live <i>Cryptosporidium</i> oocysts stock suspension	WSLH	--	2 - 8°C	1 month
Purified, live <i>Giardia</i> cysts stock suspension	WSLH	--	2 - 8°C	1 month

**Table 8.3B: Working Solution Descriptions and Related Information.**  
*(subject to change)*

Solution	Conc entrations	Storage Requirements	Expiration
Sodium Hydroxide (NaOH)	6.0 N	ambient	1 year
Sodium Hydroxide (NaOH)	1.0 N	ambient	1 year
Hydrochloric Acid (HCl)	6.0 N	ambient	1 year
Hydrochloric Acid (HCl)	1.0 N	ambient	1 year
Hydrochloric Acid (HCl)	0.1 N	ambient	1 year
Laureth-12 stock vials	10g/100mL	0°C to -20°C	1 year
Tris Working Solution	1 M, pH 7.4	ambient	3 months
Elution Buffer	--	ambient	1 week
1X SL Buffer A Solution	--	2 - 8°C	Prepared Daily
Staining 1X wash buffer	--	ambient	3 months
Phosphate Buffered Saline (PBS) Solution, pH 7.4	--	ambient	1 week
Working DAPI stain	10µL Stock/25ml Phosphate Buffer	Ambient/Dark container	1 day

## 9.0 LABORATORY PRACTICES

### 9.1 R REAGENT GRADE WATER

ASTM Type II grade water: Reagent water is analyzed for total chlorine, heterotrophic bacteria, specific conductance, pH, total organic carbon, ammonia and organic nitrogen on a monthly basis. Reagent water is tested for metals: Lead, Cadmium, Chromium, Copper, Nickel, and Zinc on an annual basis. A Use Test is performed on a quarterly basis. Reagent water used for preparing reagents must meet the following acceptance criteria:

Parameter	Limits	Frequency
Conductivity	>0.5 megaohms or <2 µmhos/cm (µseimens/cm) at 25 deg C	Monthly
Pb, Cd, Cr, Cu, Ni, Zn	Not greater than 0.05mg/L per contaminant. Collectively not greater than 0.1mg/L	Annually
Total Residual Chlorine	< 0.1 mg/L	Monthly
Heterotrophic Plate Count	<500 CFU/mL or MPN <500/mL	Monthly

### 9.2 G GLASSWARE WASHING AND STERILIZATION PROCEDURES

Glassware washing and preparation/sterilization procedures are outlined in SOP #350414, *Steamscrubber Operation and Maintenance*, SOP #350408, *Biosafety Guidelines for Cryptosporidium Laboratory* and SOP #350412, *Cryptosporidium Laboratory Equipment Cleaning*.

Laboratory glassware and plasticware are checked for acceptability prior to use. Glassware acceptance criteria are documented in SOP #350412, *Cryptosporidium Laboratory Equipment Cleaning*.

## 10.0 ANALYTICAL PROCEDURES

- 10.1 A list of laboratory SOPs associated with the protozoan laboratory can be found in the following table:

**TABLE 10.1: PROTOZOAN DEPARTMENT SOPs**

*This Table is subject to revision without notice*

SOP #	Title
350401	Isolation & Identification of <i>Giardia</i> and/or <i>Cryptosporidium</i> in Water
350402	Method 1622/1623 Field-Filtering Sample Collection and Laboratory
350403	Method 1622/1623 Bulk Sample Collection and Laboratory Delivery
350404	Method 1622/1623 Sample Receiving
350405	Training Protocol for Method 1622/1623
350406	Data Collection and Verification for Method 1622/1623



SOP #	Title
350407	Microscope Analyst Verification
350408	Biosafety Guidelines for <i>Cryptosporidium</i> Laboratory
350409	IPR, OPR and MS Spiking Procedures and Corrective Actions
350410	IEC CRU-5000 Centrifuge Operation and Maintenance
350411	Lab-Line Multi-Wrist Shaker Operation and Maintenance
350412	<i>Cryptosporidium</i> Laboratory Equipment Cleaning
350413	Olympus BX40 Microscope Operation and Maintenance
350414	Steamscrubber Dishwasher Operation and Maintenance

10.2 The following references are used for analytical procedures conducted in the laboratory:

- EPA. Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA, December 2005.
- EPA. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA, December 2005.
- EPA. Microbial Laboratory Guidance Manual for the Final Long Term 2 Enhanced Surface Water Treatment Rule. February 2006.
- Supplement 2 to the Fifth Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water, EPA 815-F-12-006, November 2012

## 11.0 QUALITY CONTROL CHECKS

- 11.1 ESC participates in proficiency testing (PT) through the analysis of spiked vials received from Wisconsin State Laboratory of Hygiene (WSLH) and analyzed according to study instructions and the ESC SOP. When the analysis is completed, the results are reported to the PT sample provider who issues the testing results as either a “pass” or “fail” to all regulatory agencies, as requested by ESC. If the laboratory fails a PT round, a follow-up test is performed in an attempt to meet the necessary requirements for proficiency. If the follow-up test results in a second failure, the laboratory takes part in re-training .
- 11.2 An Initial Precision and Recovery test (IPR) is performed prior to the first time the method is used and at any time the method or instrumentation is modified. The IPR consists of four reagent water samples spiked with 100-500 oocysts from a spiking vial received from Wisconsin State Laboratory. Recoveries from the IPR must fall within the EPA approved QC limits: Oocysts= 24- 100% and Cysts= 24-100%, and the Relative Standard Deviation (RSD) of the four recoveries should be less than or equal to 55% for *Cryptosporidium*, and less than or equal to 49% for *Giardia*.
- 11.2 An Ongoing Precision and Recovery sample (OPR) is analyzed once weekly or per 20 samples, and before any field samples are processed. The OPR is spiked with 100-500 cysts and/or oocysts from a spiking vial received from the WSLH. Recoveries from the OPR must fall within EPA approved QC limits: Oocysts = 33-100% and Cysts = 14-100%.

- 11.3 A Method Blank is also analyzed at least once weekly or per every 20 samples processed, and before any field samples are processed. The Method Blank must be free of test organisms and serves as a sterility control on the analytical system.
- 11.4 If either sample falls outside acceptance parameters, corrective action must be taken and the samples re-analyzed until the QC criteria are met. Client samples may only be analyzed following acceptable QC sample results. Quality control information is located in SOP #350409, *IPR (Initial Precision and Recovery)*, *OPR (Ongoing Precision and Recovery)* and *MS (Matrix Spike sample), Spiking Procedures and Corrective Actions*.
- 11.5 Clients are required to send a duplicate sample early in their sampling schedule and then again after every 20 field samples collected. This duplicate is utilized in the laboratory as a Matrix Spike (MS). The MS is spiked in the same manner and with the same number of organisms as the OPR to determine the effects of the matrix on the analytical process.
- 11.6 Inter/intra-analyst precision is determined, at least monthly for verification of analyst performance to assess and maintain consistency in slide examination among analysts. Quality Control information is located in SOP #350407, *Microscope Analyst Verification*.
- 11.7 Control charts of OPR and MS recoveries are maintained in the laboratory. The control charts graphically display the results of continuing performance when using Methods 1623 and 1622. If recoveries fall outside the control limits, or declining trends are observed, corrective action must be taken to investigate the potential causes of the outlying result.
- 11.8 Positive staining controls are used to verify that the FITC and DAPI stains are fluorescing at the appropriate intensity and uniformity. Negative staining controls are examined to verify that no oocysts or interfering particles are present. Both staining controls are examined using protocols as stated in ESC SOP # 350401 and meet criteria for EPA 1623 or EPA 1622.
- 11.9 IMS controls are used in the event of low recoveries to rule out any IMS steps as the cause. The IMS controls are processed beginning with the IMS procedure using protocols as stated in ESC SOP #350401, and meet criteria for EPA 1622 or EPA 1623.

## **12.0 DATA REDUCTION, VALIDATION AND REPORTING**

### **12.1 D ATA REDUCTION**

- The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in SOP #350401, *Isolation and Identification of Cryptosporidium and/or Giardia in Water* and SOP #350406, *Data Collection and Verification for Method 1622/1623*.

## 12.2 V ALIDATION

Guidelines for data validation are found in SOP #350406, *Data Collection and Verification for Method 1622/1623*. In general, data integrity involves reviewing all data entries and calculations for errors, reviewing all documentation to assure that sample information is correct and complete, and that the tests have been performed appropriately and within the appropriate sample holding times. The secondary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP is followed
- Sample preparation is correct and complete
- Analytical results are correct and complete

## 12.3 R EPORTING

Reporting procedures are documented in SOP #350406, *Data Collection and Verification for Method 1622/1623*. Depending on the needs of the client, one or more of the following may be included: Case narrative, Chain of Custody, Internal Chain of Custody, Final Report, Raw Data, etc. When the package involves more than just QC forms, it must contain a Table of Contents and Pagination. When the package is complete, it must be reviewed first by the Primary Analyst followed by the Department Manager or second qualified analyst. The final reviewer signs that the information is complete and the package is ready for submission to the client. A copy of the final package must be kept on file.

## 13.0 CORRECTIVE ACTION

- 13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CARs are kept on file by the Regulatory Affairs Department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

Corrective action procedures that are specific to *Cryptosporidium* and *Giardia* analyses are documented in the SOP #350409, *IPR (Initial Precision and Recovery)*, *OPR (Ongoing Precision and Recovery)* and *MS (Matrix Spike sample)*, *Spiking Procedures*.

### 13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these limits are exceeded, corrective action must be taken. Calculated sample spike control limits are also used.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP.

13.2.1 If a spiked sample or set of samples fails to meet quality control limits

Rejection Criteria - Recoveries from the OPR fall beyond the approved QC limits.

Corrective Action - Examine the spiking suspension organisms directly. To determine if the failure of the spike is due to changes in the microscope or problem with the antibody stain, re-examine the positive staining control, check Köhler illumination, and check the fluorescence and DAPI. To determine if the failure of the spike is attributable to the separation system, check the system performance by spiking a 10mL volume of reagent water with 100-500 cysts and/or oocysts and processing the sample through the IMS, staining and examination procedures. Recoveries should be greater than 70% of the expected concentration. If the failure of the spike is attributable to the filtration/elution/concentration system, check the system performance by processing spiked reagent water according to the method and filter, stain and examine the sample concentrate. This process is performed until the cause of the failure is isolated and corrected. The sample then must be re-analyzed until acceptable results are achieved.

13.2.2 Method Blank contains positive organism when analyzed.

Rejection Criteria – The Method Blank must be free of test organisms and serves as a sterility control on the analytical system.

Corrective Action - Equipment used to process the sample may be cleaned and/or replaced. Reagents used to process the sample may be disposed of and new reagents purchased or prepared. A new method blank is prepared and analyzed. This process is repeated until the method blank passes the acceptance criteria.

13.2.3 Inter/intra-analyst precision analyses are beyond  $\pm 10\%$ .

Rejection Criteria – Results for inter and/or intra-analyst precision must be within 10% of original results.

Corrective Action - The differences are discussed between analysts until a consensus is found.

13.2.4 Holding time on sample exceeded or not received at appropriate temperature.

Rejection Criteria - The sample not received on day of collection must be received at the laboratory at  $\leq 20\text{ }^{\circ}\text{C}$  and not frozen, and within 96 hours holding time.

Corrective Action - The samples must be re-collected.

13.2.5 Positive and Negative staining controls fail.

Rejection Criteria - If a positive and negative staining control fails all slides that were stained in that batch have failed and samples must be re-collected.

Corrective Action - If positive staining control fails due to faintness, fading or diffusion of the DAPI stain, the holding time may be reduced, or the concentration of the DAPI staining solution may be adjusted so that fading or diffusion does not occur. This process is performed until the cause of the failure is isolated and corrected. The sample then must be re-analyzed until acceptable results are achieved.

#### 14.0 RECORD KEEPING

Record keeping is outlined in SOP #030230, *Standards Logger*, SOP #030227, *Data Review* and SOP #030201, *Data Handling and Reporting*

Hard copy data of benchsheets and slide examination forms for all compliance monitoring samples, including both field and MS samples, and OPR samples and MB are archived. Benchsheets and slide examination forms for all ongoing PT samples are stored in the laboratory. Documentation for IPR and initial PT data for each method variation used for compliance samples is also archived in the laboratory.

#### 15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 13.0 and SOP #010104, *Internal Audits*.

#### 16.0 REVISIONS

The Regulatory Affairs Department has an electronic version of this Quality Assurance Manual with tracked changes detailing all revisions made to the previous version. This version is available upon request. Revisions to the previous version of this appendix are summarized in the table below.

Document	Revision
Quality Assurance Manual Version 14.0 (Appendix XII)	Section 16 – New section for summary of revisions to previous version. No other changes.

End of Document

## **Appendix F Decision Unit Random GPS Points for ISM Sampling**

DU	POINT	X_EASTING_FT	Y_NORTHING_FT	Long	Lat
DU1.1-R1	1	2353902.61025000000	2201799.91242000000	-116.84965263500	47.70099838610
DU1.1-R1	2	2354764.36399000000	2201769.39966000000	-116.84615170800	47.70094823100
DU1.1-R1	3	2354778.77824000000	2201776.48523000000	-116.84609358500	47.70096821230
DU1.1-R1	4	2354427.15740000000	2201795.17669000000	-116.84752242600	47.70100579870
DU1.1-R1	5	2354045.05050000000	2201809.08056000000	-116.84907478100	47.70102905840
DU1.1-R1	6	2354195.43119000000	2201791.31944000000	-116.84846313400	47.70098622140
DU1.1-R1	7	2353625.73604000000	2201809.95455000000	-116.85077746800	47.70101513190
DU1.1-R1	8	2354282.73820000000	2201797.98344000000	-116.84810900500	47.70100788130
DU1.1-R1	9	2354483.40994000000	2201780.67513000000	-116.84729317700	47.70096823350
DU1.1-R1	10	2353715.73985000000	2201807.03375000000	-116.85041183700	47.70101063140
DU1.1-R1	11	2354954.99245000000	2201775.32884000000	-116.84537799700	47.70097187570
DU1.1-R1	12	2353803.41994000000	2201817.41133000000	-116.85005640800	47.70104249110
DU1.1-R1	13	2354677.92246000000	2201782.69763000000	-116.84650347000	47.70098132800
DU1.1-R1	14	2354457.98152000000	2201781.06262000000	-116.84739645100	47.70096830810
DU1.1-R1	15	2354667.22096000000	2201770.19742000000	-116.84654620500	47.70094664870
DU1.1-R1	16	2353817.93136000000	2201810.17619000000	-116.84999706700	47.70102322390
DU1.1-R1	17	2354910.51786000000	2201778.50266000000	-116.84555876900	47.70097885120
DU1.1-R1	18	2354425.72127000000	2201788.64019000000	-116.84752788100	47.70098782580
DU1.1-R1	19	2353675.71199000000	2201822.17328000000	-116.85057524500	47.70105057100
DU1.1-R1	20	2353999.19222000000	2201793.87281000000	-116.84926011300	47.70098558900
DU1.1-R1	21	2354438.73724000000	2201790.31253000000	-116.84747512600	47.70099291540
DU1.1-R1	22	2354945.10572000000	2201767.81126000000	-116.84541771000	47.70095088610
DU1.1-R1	23	2354433.15666000000	2201784.82043000000	-116.84749747000	47.70097764440
DU1.1-R1	24	2354018.36090000000	2201797.32463000000	-116.84918247800	47.70099579640
DU1.1-R1	25	2353838.78945000000	2201799.09230000000	-116.84991173300	47.70099365410
DU1.1-R1	26	2353613.42490000000	2201808.18373000000	-116.85082735500	47.70100979840
DU1.1-R1	27	2354001.03909000000	2201810.97106000000	-116.84925360000	47.70103252840
DU1.1-R1	28	2354221.52243000000	2201798.69119000000	-116.84835761400	47.70100744210
DU1.1-R1	29	2354705.05218000000	2201772.28781000000	-116.84639271100	47.70095384660
DU1.1-R1	30	2353948.41549000000	2201810.52252000000	-116.84946725300	47.70102925150
DU1.2-R1	1	2355350.42240000000	2201784.69707000000	-116.84377288000	47.70101287220
DU1.2-R1	2	2355496.05807000000	2201772.38390000000	-116.84318081600	47.70098475660
DU1.2-R1	3	2355935.82693000000	2201677.41735000000	-116.84138968600	47.70074144520
DU1.2-R1	4	2355940.87136000000	2201697.48119000000	-116.84137035100	47.70079663660
DU1.2-R1	5	2355857.29101000000	2201722.19997000000	-116.84171114500	47.70086116420
DU1.2-R1	6	2355685.81632000000	2201711.15362000000	-116.84240679000	47.70082425820
DU1.2-R1	7	2355426.51575000000	2201729.65247000000	-116.84346074500	47.70086493570
DU1.2-R1	8	2356014.99092000000	2201675.07034000000	-116.84106810600	47.70073806900
DU1.2-R1	9	2355651.23755000000	2201707.56068000000	-116.84254699200	47.70081307280
DU1.2-R1	10	2356068.79584000000	2201645.91147000000	-116.84084796200	47.70066021950
DU1.2-R1	11	2355810.35628000000	2201695.75514000000	-116.84190021100	47.70078686330
DU1.2-R1	12	2356029.76615000000	2201685.05644000000	-116.84100868200	47.70076601220
DU1.2-R1	13	2355802.75408000000	2201698.54292000000	-116.84193124000	47.70079421110
DU1.2-R1	14	2355258.34552000000	2201751.70375000000	-116.84414486800	47.70091887030
DU1.2-R1	15	2355199.27979000000	2201742.77879000000	-116.84438419400	47.70089211890
DU1.2-R1	16	2355833.28438000000	2201726.19601000000	-116.84180885400	47.70087119010
DU1.2-R1	17	2355282.43969000000	2201778.02038000000	-116.84404854200	47.70099193910
DU1.2-R1	18	2355557.32157000000	2201751.59020000000	-116.84293086200	47.70093012930
DU1.2-R1	19	2355145.67387000000	2201759.69779000000	-116.84460283300	47.70093641890
DU1.2-R1	20	2355228.38413000000	2201736.21694000000	-116.84426563900	47.70087525950
DU1.2-R1	21	2355686.58064000000	2201753.80363000000	-116.84240612900	47.70094119480
DU1.2-R1	22	2355755.49105000000	2201737.68686000000	-116.84212539300	47.70089968110
DU1.2-R1	23	2356126.26591000000	2201598.42268000000	-116.84061189000	47.70053226730
DU1.2-R1	24	2356004.53627000000	2201685.73735000000	-116.84111116700	47.70076690450
DU1.2-R1	25	2355850.43004000000	2201706.30771000000	-116.84173809500	47.70081733710
DU1.2-R1	26	2355427.94594000000	2201762.42881000000	-116.84345681600	47.70095483360
DU1.2-R1	27	2355619.95029000000	2201752.48262000000	-116.84267660700	47.70093499760
DU1.2-R1	28	2356071.79359000000	2201658.61039000000	-116.84083651600	47.70069514400
DU1.2-R1	29	2356136.08107000000	2201622.41529000000	-116.84057340700	47.70059841180
DU1.2-R1	30	2355930.53973000000	2201655.73789000000	-116.84140991500	47.70068181590
DU1.3A	1	2356420.22123000000	2201577.10072000000	-116.83941706600	47.70048516130
DU1.3A	2	2356640.02912000000	2201495.41290000000	-116.83851987500	47.70026971920
DU1.3A	3	2357515.06409000000	2201240.60229000000	-116.83495231700	47.69960491430
DU1.3A	4	2356706.94901000000	2201470.76460000000	-116.83824674200	47.70020473370
DU1.3A	5	2357184.19384000000	2201326.35124000000	-116.83630067600	47.69982724780
DU1.3A	6	2356272.71179000000	2201619.23461000000	-116.84001843500	47.70059496520
DU1.3A	7	2357239.25826000000	2201301.69827000000	-116.83607568600	47.69976178850



DU1.3A	8	2356335.66105000000	2201598.63738000000	-116.83976165300	47.70054093450
DU1.3A	9	2356604.20696000000	2201498.19678000000	-116.83866549000	47.70027597000
DU1.3A	10	2356386.08035000000	2201575.16829000000	-116.83955558500	47.70047854810
DU1.3A	11	2356529.77731000000	2201530.11575000000	-116.83896953200	47.70036059460
DU1.3A	12	2356892.32308000000	2201420.00626000000	-116.83749114100	47.70007273750
DU1.3A	13	2357363.26281000000	2201277.90445000000	-116.83557081800	47.69970133260
DU1.3A	14	2356755.87122000000	2201463.40086000000	-116.83804767500	47.70018643300
DU1.3A	15	2357390.35847000000	2201262.50041000000	-116.83545992200	47.69966014970
DU1.3A	16	2356622.57468000000	2201502.43303000000	-116.83859115000	47.70028828960
DU1.3A	17	2356823.13676000000	2201440.41899000000	-116.83777323400	47.70012602750
DU1.3A	18	2357147.51199000000	2201326.77726000000	-116.83644964500	47.69982700530
DU1.3A	19	2356317.72547000000	2201598.25590000000	-116.83983445900	47.70053919710
DU1.3A	20	2357541.32878000000	2201236.09867000000	-116.83484541400	47.69959357790
DU1.3A	21	2356372.85973000000	2201572.54146000000	-116.83960911700	47.70047083790
DU1.3A	22	2356782.18421000000	2201441.51545000000	-116.83793958300	47.70012745640
DU1.3A	23	2356397.94866000000	2201569.12313000000	-116.83950704800	47.70046243540
DU1.3A	24	2357417.25791000000	2201260.38480000000	-116.83535057800	47.69965538390
DU1.3A	25	2357157.97944000000	2201328.64444000000	-116.83640724900	47.69983252580
DU1.3A	26	2356239.87298000000	2201631.12533000000	-116.84015245700	47.70062629180
DU1.3A	27	2357202.68136000000	2201309.45799000000	-116.83622464600	47.69978165260
DU1.3A	28	2357030.23559000000	2201371.62589000000	-116.83692839500	47.69994542910
DU1.3A	29	2357555.07368000000	2201244.41615000000	-116.83479007700	47.69961690440
DU1.3A	30	2356489.20461000000	2201551.75687000000	-116.83913551200	47.70041835100
DU1.3B	1	2357557.50433000000	2201216.19967000000	-116.83477860300	47.69953965400
DU1.3B	2	2356273.84374000000	2201597.85720000000	-116.84001261800	47.70053641160
DU1.3B	3	2357461.86983000000	2201236.34895000000	-116.83516806600	47.69959121290
DU1.3B	4	2356854.21894000000	2201409.84143000000	-116.83764528200	47.70004340830
DU1.3B	5	2357375.92190000000	2201240.54576000000	-116.83551729100	47.69959941560
DU1.3B	6	2357489.49894000000	2201222.57018000000	-116.83505509600	47.69955450510
DU1.3B	7	2356508.10289000000	2201525.81409000000	-116.83905729500	47.70034796810
DU1.3B	8	2356304.22335000000	2201594.64848000000	-116.83988907800	47.70052878810
DU1.3B	9	2357170.30458000000	2201304.97910000000	-116.83635585600	47.69976813100
DU1.3B	10	2356590.35807000000	2201483.53784000000	-116.83872088600	47.70023525510
DU1.3B	11	2357057.61805000000	2201335.38889000000	-116.83681514600	47.69984715360
DU1.3B	12	2356747.64255000000	2201449.71864000000	-116.83808030700	47.70014861210
DU1.3B	13	2356577.67197000000	2201486.75187000000	-116.83877258100	47.70024357620
DU1.3B	14	2356561.28170000000	2201503.41117000000	-116.83884008400	47.70028860920
DU1.3B	15	2357393.53392000000	2201249.34811000000	-116.83544627900	47.69962422010
DU1.3B	16	2356676.34239000000	2201462.16225000000	-116.83837052900	47.70017997520
DU1.3B	17	2356683.90592000000	2201458.20889000000	-116.83833959200	47.70016943000
DU1.3B	18	2357032.40753000000	2201346.03758000000	-116.83691811800	47.69987537290
DU1.3B	19	2356934.32922000000	2201375.84015000000	-116.83731805900	47.69995329100
DU1.3B	20	2356482.65353000000	2201520.03003000000	-116.83916030200	47.70033113260
DU1.3B	21	2357310.72142000000	2201271.90696000000	-116.83578381800	47.69968287430
DU1.3B	22	2356505.27763000000	2201511.23817000000	-116.83906793500	47.70030790540
DU1.3B	23	2357218.03453000000	2201295.50810000000	-116.83616151100	47.69974400500
DU1.3B	24	2356961.64634000000	2201372.36008000000	-116.83720694100	47.69994480290
DU1.3B	25	2357233.27730000000	2201287.48490000000	-116.83609916200	47.69972259860
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DU3.2C	26	2364142.41341000000	2198474.21810000000	-116.80788880900	47.69227324840
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DU1.1-R2	5	2354382.55003000000	2201787.86434000000	-116.84770313400	47.70098402220
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DU1.3B-alt	29	2356641.25746000000	2201466.20546000000	-116.83851322100	47.70018970650
DU1.3B-alt	30	2357228.90087000000	2201292.47130000000	-116.83611721600	47.69973609850
DU1.3C-alt	8	2356884.30439000000	2201371.65576000000	-116.83752094500	47.69993989620
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DU1.3C-alt	2	2356605.26384000000	2201463.22172000000	-116.83865920200	47.70018014120
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DU1.3C-alt	5	2357024.69234000000	2201325.51538000000	-116.83694827600	47.69981882310
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DU1.3C-alt	7	2356223.86675000000	2201598.12928000000	-116.84021556500	47.70053522940
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DU1.3C-alt	10	2356630.54102000000	2201466.25175000000	-116.83855673800	47.70018942060
DU1.3C-alt	11	2356324.30887000000	2201554.72989000000	-116.83980524000	47.70042014260
DU1.3C-alt	12	2356536.55554000000	2201488.02680000000	-116.83893960600	47.70024548650
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DU1.3C-alt	14	2357392.5593000000	2201233.2057600000	-116.83544931800	47.69957993510
DU1.3C-alt	15	2356966.3167100000	2201356.3110700000	-116.83718706200	47.69990099090
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DU3.1C-alt	4	2362865.49986000000	2199294.43496000000	-116.81311864600	47.69447363600
DU3.1C-alt	5	2362330.54929000000	2199632.71668000000	-116.81530946700	47.69538075690
DU3.1C-alt	6	2362869.33979000000	2199280.19420000000	-116.81310226200	47.69443474500
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DU2.2C-alt	6	2359681.06147000000	2200719.41628000000	-116.82612800500	47.69825912090
DU2.2C-alt	7	2359770.00203000000	2200719.72523000000	-116.82576689400	47.69826335430
DU2.2C-alt	8	2359353.70253000000	2200803.96716000000	-116.82746197000	47.69847840860
DU2.2C-alt	9	2360047.94645000000	2200702.40008000000	-116.82463736800	47.69822643990
DU2.2C-alt	10	2361039.10322000000	2200475.30578000000	-116.82060018500	47.69764157420
DU2.2C-alt	11	2358889.65648000000	2200921.24977000000	-116.82935279700	47.69878218170
DU2.2C-alt	13	2360112.06224000000	2200708.46216000000	-116.82437737700	47.69824549470
DU2.2C-alt	14	2360765.27672000000	2200627.49446000000	-116.82172055200	47.69804835880
DU2.2C-alt	15	2361436.67531000000	2200215.88162000000	-116.81897138500	47.69694551730
DU2.2C-alt	16	2359435.37780000000	2200775.21603000000	-116.82712871500	47.69840271280
DU2.2C-alt	17	2358836.88891000000	2200945.55303000000	-116.82956842900	47.69884678350
DU2.2C-alt	18	2359435.60620000000	2200782.69607000000	-116.82712821000	47.69842322500
DU2.2C-alt	19	2361146.08138000000	2200397.91598000000	-116.82016148000	47.69743349310
DU2.2C-alt	20	2361395.52171000000	2200241.83161000000	-116.81913993300	47.69701509200
DU2.2C-alt	21	2359252.39852000000	2200819.74486000000	-116.82787419100	47.69851779340
DU2.2C-alt	22	2361003.25371000000	2200496.42035000000	-116.82074693000	47.69769809300
DU2.2C-alt	23	2360434.72518000000	2200713.01716000000	-116.82306751100	47.69827024060
DU2.2C-alt	24	2361102.23316000000	2200434.42048000000	-116.82034156400	47.69753189500
DU2.2C-alt	25	2361389.19575000000	2200243.16875000000	-116.81916569300	47.69701851790
DU2.2C-alt	26	2358901.07865000000	2200913.83341000000	-116.82930599900	47.69876228910
DU2.2C-alt	27	2360461.73472000000	2200703.96327000000	-116.82295733400	47.69824644850
DU2.2C-alt	28	2360370.27556000000	2200711.51308000000	-116.82332911400	47.69826367000
DU2.2C-alt	29	2360897.33173000000	2200551.34594000000	-116.82118008900	47.69784463520
DU2.2C-alt	30	2360166.24517000000	2200703.19928000000	-116.82415708000	47.69823312840