October 9, 2012



Walter Coke 3500 35<sup>th</sup> Avenue North Birmingham, Alabama 35207

Attention: Ms. Meredith Anderson

Re: EPA Comments Dated 10/4/12 Groundwater IM Sampling and Analysis Work Plan (GW IM SAP) Walter Coke 3500 35<sup>th</sup> Avenue North Birmingham, Jefferson County, Alabama USEPA ID No. ALD 000 828 848 Terracon Project No. E1127095

Dear Ms. Anderson:

On behalf of Walter Coke, Inc. (Walter Coke), Terracon Consultants, Inc. (Terracon) is pleased to submit the enclosed revisions to the Interim Measures (IM) Groundwater Sampling and Analysis Plan (*Revision 1.0*) for the above-referenced site. These revisions have been prepared in response to Final Comments dated 10/4/12 for the GW IM Sampling and Analysis Work Plan from the United States Environmental Protection Agency (USEPA) Region 4. The individual comments and responses are provided below:

#### **General Comments**

#### **USEPA Comment No. 1**

The new monitoring well down gradient of CW-1 (MW-90) was missing from the text and tables of the GW IM SAP, although it is included in Figure 3. As this monitoring well is integral to the IM, please add MW-90 into the text of the GW IM SAP, to Table 2-1, and to other portions of the document, as appropriate.

## Walter Coke Response No. 1

Monitoring well MW-90 has been added to Table 2-1, and referenced in other portions of the document as appropriate.



Terracon Consultants, Inc. 110 12<sup>th</sup> Street North Birmingham, Alabama 35203 P [205] 942 1289 F [205] 443 5302 terracon.com



## **USEPA Comment No. 2**

GW IM SAP does not consistently refer to the type of equipment to be used for the low flow groundwater sampling and purging. It references the use of "submersible pumps", "pumps" and "peristaltic pumps" for the actual pumping element, while referencing the use of bailers for the (pre-sample) purging. The IM GW SAP should be revised to be consistent, as well as technically adequate with regard to the low flow sampling equipment and procedures to be used. For instance, peristaltic pumps are not recommended as they have inherent effective limits on use in deeper (i.e., greater than 30 feet) monitoring wells, while both peristaltic pumps and bailers will tend to volatilize a sample more than is desired.

## Walter Coke Response No. 2

The groundwater and purging Sections 6.2.1 through 6.2.3 have been revised to be consistent and reference the EPA Region 4 Field Branches Quality System and Technical Procedures found at <u>http://www.epa.gov/region4/sesd/fbqstp/index.html</u>. Groundwater pumps used will be consistent with the EPA guidance.

## **USEPA Comment No. 3**

For groundwater containment systems such as the one discussed here, it is important to differentiate between a zone of influence and a zone of capture of pumping well systems. All subsequent reports, especially performance monitoring reports, should focus on demonstrating that the capture zone is adequate to control site contaminants. Since capture zones are three-dimensional systems, it is important to collect water level data and analytical data for the deep wells in this area of the containment system (MW-49D and MW-89) to demonstrate that the plume is controlled in the vertical direction. Please add the following schedule for sampling of these deep wells to the GW IM SAP: 1) prior to system installation – analytical sampling and water level measurements; 2) semi-annual – water level measurements only; and 3) annual – analytical sampling and water level measurements. A groundwater flow and transport model may also be helpful in demonstrating the capture zone effectiveness. The following document can be referenced for this type of demonstration: *A Systematic Approach for Evaluation of Capture Zones at Pump and Treat Systems* (http://www.epa.gov/ada/pubs/reports.html).

## Walter Coke Response No. 3

Table 2-1 has been modified to add monitoring wells MW-49D and MW-89. Section 4.2 has also been modified to describe the sampling frequency of wells MW-49D and MW-89, and to describe the frequency of the water level measurements in all 18 monitoring wells.



## **USEPA Comment No. 4**

Sections 1.0 and 12.0 discuss a site-specific field Health and Safety Plan (HASP) for the work proposed in this work plan. The HASP should be submitted to EPA for approval prior to implementing this IM, and it should be a permanent part of this work plan.

#### Walter Coke Response No. 4

The HASP was prepared under separate cover and has been submitted to USEPA. The HASP is considered a permanent part of this work plan and has been prepared to serve as the HASP for all environmental work performed under the Administrative Order on Consent (AOC) for Walter Coke with effective date September 24, 2012.

#### Other Comments

#### **USEPA Comment No. 5**

Cover letter:

An EPA Region 9 guidance document is referenced in paragraph 2. Please ensure that this Work Plan and its implementation are consistent with EPA Region 4 guidelines (e.g., EPA Region 4 protocols for field sampling methods, quality assurance, etc.).

#### Walter Coke Response No. 5

The reference to EPA Region 9 has been removed. The Plan references USEPA Region 4 guidance as applicable.

#### **USEPA Comment No. 6**

p. 1 – Section 1.0:

Please reference the final, modified, and approved Groundwater IMWP, dated May 2012, prepared by CH2MHill. Also, please note at the end of paragraph 1 that "Project activities will focus on the area of the FCP located on the site <u>and in the off-site area east of the FCP</u>."

#### Walter Coke Response No. 6

These changes have been made to paragraph 1 of Section 1.0.

#### **USEPA Comment No. 7**

#### p. 1 – Section 1.1:

Amend this sentence to end after "...the former chemical plant (FCP)."



This change has been made to Section 1.1.

## **USEPA Comment No. 8**

p. 2-3 – Project Organizational Chart and Table:

Please add the Alabama Department of Environmental Management (ADEM) to the project organizational chart (dotted line from the EPA) and project organization table (Table 1).

## Walter Coke Response No. 8

The project Organizational Chart and Table have been revised to include ADEM.

## **USEPA Comment No. 9**

p. 4 – Section 1.5:

Please add a figure of the FCP that includes the approximate plume boundaries and the general groundwater flow directions.

## Walter Coke Response No. 9

Figure 3 has been modified to include the plume boundaries, the potentiometric surface and, the general groundwater flow direction.

## **USEPA Comment No. 10**

p. 4 – Section 2.0/last sentence:

The EPA approved the Groundwater Interim Measures Work Plan (Groundwater IMWP) in its letter dated **April 16, 2012**, not April 13, 2012. Also, please reference the final, modified, and approved Groundwater IMWP, dated May 2012, prepared by CH2MHill.

#### Walter Coke Response No. 10

These changes have been made to Section 2.0.

## USEPA Comment No. 11

p. 5 – Table 2-1:

The final Groundwater IMWP specifies that an additional monitoring well (MW-90) will be installed down-gradient from CW-1. Please add this well to Table 2-1. Also, please add a definition for "SB" to this table.



Well MW-90 has been added to Table 2-1 and a legend has been added to the bottom of the table. In addition, deep bedrock monitoring wells MW-49D and MW-89 have been added to the table. These wells were added for evaluation during the IM in USEPA Comment No. 3 above and in Walter Coke's Response No. 3.

## USEPA Comment No. 12

#### p. 7-10 – Section 3.0:

The IM GW SAP should include a detailed discussion regarding how the data will be used, what exact levels and criteria the data will be compared against, and the anticipated follow-up actions if a screening level is exceeded. The IM GW SAP should reference regional screening level (RSLs). MCLs, or other relevant levels of concern, if applicable (refer to http://www.epa.gov/reg3hwmd/risk/human/rb-concentration\_table/Generic\_Tables/index.htm for EPA RSLs). This step is important since the results of this sampling will be used to determine whether additional sampling or investigation would be warranted after the first year of IM performance/monitoring. A sub-group of VOCs/SVOCs of particular interest to the EPA at this time due to their presence in the FCP groundwater plume are: benzene, chlorobenzene, naphthalene, TCE, PCE, cis-1,2-dichloroethene, 1,2-dichloroethane, 1,2,4-trichlorobenzene, cresol, 1-methyl naphthalene, 2-methyl naphthalene, PAHs, 1,1-dichloroethene, and vinyl chloride.

## Walter Coke Response No. 12

Paragraph 2 of Section 3.2 has been modified to include this discussion.

#### **USEPA Comment No. 13**

p. 7-10 – Section 3.0:

Please assure that the laboratory analytical methods and limits will allow the resulting data to be reliably compared against the appropriate screening levels (i.e., method detection limits (MDLs) are equal to or less than the RSLs). If the laboratory is unable to achieve MDLS at or below RSLs for any constituent, please present the corrective actions that will be taken.

## Walter Coke Response No. 13

This comment has been address at the bottom of the 3<sup>rd</sup> paragraph of Section 3.2.



## **USEPA Comment No. 14**

#### p. 7-10 – Section 3.0:

Please clarify in Section 3.1, paragraph 2 that groundwater samples will be analyzed for VOCs using <u>EPA method 8260B</u>, <u>SVOCs using EPA method 8270D</u>, and <u>PAHs using EPA Method</u> <u>8270SIM</u>. Also, please update Tables 5-1 and 5-2 with this information.

#### Walter Coke Response No. 14

Paragraph 2 of Section 3.1 and tables 5-1 and 5-2 have been revised with this information.

### **USEPA Comment No. 15**

#### p. 7-10 – Section 3.0:

A discussion of project DQOs should be consistent with the EPA guidelines for DQOs and quality assurance plans. Section 3.2 should reference these EPA guidance documents found at: <u>http://www.epa.gov/quality1/qs-docs/g4-final.pdf</u> and <u>http://www.epa.gov/QUALITY/qs-docs/r5-final.pdf</u>.

#### Walter Coke Response No. 15

The first paragraph of Section 3.2 has been modified to include the references.

#### **USEPA Comment No. 16**

p. 7-10 – Section 3.0:

Section 3.3 should be clearly defined regarding data validation (not verification), including an indication of the level of validation that will be performed (Tier 1A/1B level of validation is appropriate).

#### Walter Coke Response No. 16

Section 3.3 has been revised to define validation and discuss the Tier 1A/1B level of data validation.

#### **USEPA Comment No. 17**

p. 7-10 – Section 3.0: Please add a sub-section defining "unusable data", listed as Section 3.3.3.



Section 3.3.3 Unusable Data has been added.

#### **USEPA Comment No. 18**

p. 7-10 – Section 3.0:Section 3.4 is missing (missing in the Table of Contents also).

#### Walter Coke Response No. 18

Section 3.5 Data Management has been renumbered to Section 3.4.

#### **USEPA Comment No. 19**

p. 7-10 – Section 3.0:

Section 3.5 should describe how and when analytical data will be reported/submitted to the EPA. The EPA requests data be submitted in the EPA Region 4 electronic data delivery (EDD) format. This format and the submittal procedures can be found at: <u>http://www.epa.gov/region4/</u> superfund/allresource/edd/edd.html.

#### Walter Coke Response No. 19

Section 3.5 was renumber to Section 3.4. The last paragraph of Section 3.4 describes the submittal of the data to USEPA Region 4 in the EDD format.

#### **USEPA Comment No. 20**

p. 11 – Section 4.2/1<sup>st</sup> sentence: <u>16</u>monitoring wells will be sampled, including the new monitoring well, MW-90.

#### Walter Coke Response No. 20

Due to the addition of deep bedrock monitoring wells MW-49D and MW-89. The first paragraph in Section 4.2 has been modified to read:

Based on the approved IMWP and Addendum, groundwater samples will be collected from the 18 monitoring wells listed in Table 2-1 prior to system startup. Groundwater samples from the 16 shallow bedrock wells and a groundwater sample will be collected from the combined flow of groundwater being recovered from the containment wells on a quarterly schedule for a period of one year. Groundwater samples from the two deep bedrock monitoring wells will also be collected at the end of one year.



## USEPA Comment No. 21

p. 11 - Section 5.1/1<sup>st</sup> sentence:

Again, groundwater samples will be collected from <u>16</u> monitoring wells, not 15.

## Walter Coke Response No. 21

This change has been made, but the paragraph was changed to also incorporate the addition of deep bedrock monitoring wells MW-49D and MW-89. The new 1<sup>st</sup> paragraph of Section 5.1 reads:

Groundwater samples will be collected prior to system start up and quarterly from the 16 shallow bedrock monitoring wells listed in Table 2-1. A groundwater sample will be collected quarterly from the combined flow of groundwater being recovered from the containment wells, and a groundwater sample will be collected prior to system start up and at one year after start up for the two deep bedrock monitoring wells listed in Table 2-1.

## **USEPA Comment No. 22**

p. 12 – Table 5-1: Please correct the second column to indicate that <u>16</u> monitoring wells will be sampled.

## Walter Coke Response No. 22

Table 5-1 has been corrected.

## **USEPA Comment No. 23**

p. 12 – Section 5.2:

Please add <u>MDLs</u> to the list of items to be reported on lab deliverables. It is important to that the MDLs are lower than regional screening levels (RSLs) for each chemical.

## Walter Coke Response No. 23

MDLs have been added to the list of items to be reported on lab deliverables.

## **USEPA Comment No. 24**

p. 12 – last paragraph:

Please remove the word "selected" from the first sentence of this paragraph.

## Walter Coke Response No. 24



The word "selected" has been removed from the first sentence of the third paragraph of Section 5.2.

### **USEPA Comment No. 25**

#### p. 13-16 - Section 6.0:

This section refers to groundwater well sampling and purging procedures. Please reference the appropriate EPA-Region 4 field sampling and decontamination methods, as appropriate, in these sub-sections, and indicate that all field procedures will be conducted in a manner consistent with these methods. See <u>http://www.epa.gov/region4/sesd/fbqstp/index.html</u> for EPA Region 4 protocols.

#### Walter Coke Response No. 25

Section 6.0 and the subsections has been modified to reference the USEPA Region 4 Field Branches Quality System and Technical Procedures which are found at <u>http://www.epa.gov/region4/sesd/fbqstp/index.html</u>.

#### **USEPA Comment No. 26**

#### p. 13-16 – Section 6.0:

Please ensure that the methods and equipment used for well purging and sampling are consistent with EPA approved procedures for low flow sampling. For instance, the use of peristaltic pumps (or bailers) for "low flow" VOC purging and/or sampling is not recommended as there is an extreme likelihood that their use will result in excessive volatilization of the samples. In addition, the typical depth at which a peristaltic pump may be effective is limited to approximately 25 to 30 feet below ground surface, which is expected to be shallower than several of the monitoring wells included in the IM GW SAP.

#### Walter Coke Response No. 26

Section 6.0 and its subsections have been revised to reference EPA Region 4 procedures for conducting low flow sampling.

#### **USEPA Comment No. 27**

#### p. 13-16 – Section 6.0:

Ensure that placement of the pump intake (for sampling and purging) allows for adjustments to the pump intake depth depending on the length of the applicable water column.



Section 6.0 and its subsections have been revised to reference EPA Region 4 procedures for conducting low flow sampling and pump placement.

#### **USEPA Comment No. 28**

p. 13-16 – Section 6.0: Please remove the discussion of the default 30-minute well purging procedures (p. 15).

#### Walter Coke Response No. 28

The discussion of the default 30-minute well purging procedure has been removed.

#### **USEPA Comment No. 29**

p. 13-16 – Section 6.0:

Please delete the use of a hexane rinse during decontamination procedures. All decontamination procedures should be implemented in accordance with the EPA-Region 4 standard operating procedures (see link provided above).

#### Walter Coke Response No. 29

The use of a Hexane Rinse has been deleted.

#### USEPA Comment No. 30

p. 17 – Section 7.1: Please add a discussion of the sample container requirements for SVOC and PAH analysis.

#### Walter Coke Response No. 30

Paragraph 2 of Section 7.1 has been added to discuss the sample container requirements for SVOC and PAH analysis.

#### USEPA Comment No. 31

p. 17 – Section 8.0:

Disposal of investigation derived waste (IDW) should be conducted in accordance with all appropriate requirements and protocols, including the EPA Region 4 standard operating procedures for the management of IDW (<u>http://www.epa.gov/region4/sesd/fbqstp/Management-of-IDW.pdf</u>).



Paragraph 2 of Section 8.0 has been added to reference that the disposal of the IDW will be conducted in accordance with the EPA Region 4 FBQS (<u>http://www.epa.gov</u> /region4/sesd/fbqstp/index.html).

#### **USEPA Comment No. 32**

#### p. 18-19 – Sections 9.1 and 9.2:

Please specify that field log books, photographs, sample labels, and chain of custody forms will also indicate the unique sample ID for each sample. A discussion of the unique sample identification method (consistent with the EDD requirements) that will be used to identify each sample should also be included.

#### Walter Coke Response No. 32

Section 9.1 and 9.2 have been updated to reference Unique Sample ID's for each sample and reference the USEPA Region 4 guidance for electronic deliverables (http://www.epa.gov/region4/superfund/allresource/edd/ edd.html).

#### **USEPA Comment No. 33**

#### p. 21-22 – Section 10:

Please revise the introduction to this section to indicate that QC samples are required, rather than the need for QC samples being "assessed" during each sampling event. Also, QC samples are necessary at specific intervals/ratios relative to the environmental samples and, therefore, should be clearly defined for this project. For instance, the list of QC samples is incomplete and should also include duplicate samples (one for every 10 environmental media samples collected), matrix spike/matrix spike duplicate, and equipment blanks (one for every 20 environmental samples or one per day of field activities). Trip blanks (complete set) are required for each cooler or shipping container which contains aqueous matrix samples for VOC analysis, not one set per shipment. A table displaying the necessary QC samples would be appropriate, or perhaps these samples can be added to Table 5-1 Sample Summary. Please refer to the EPA's guidance document on quality assurance project plans at <a href="http://www.epa.gov/QUALITY/qs-docs/r5-final.pdf">http://www.epa.gov/QUALITY/qs-docs/r5-final.pdf</a>.



Section 10.0 has been revised to include all of the QC samples that are required to be collected during each groundwater sampling event. In addition, the list of QC samples to be collected has been added to Table 5-1.

## **USEPA** Comment No. 34

p. 22 – Section 11: Any modifications to the sampling plan should also be reported to the EPA for approval.

### Walter Coke Response No. 34

Section 11.0 has been modified to include the reporting of modifications to USEPA for approval.

## **USEPA** Comment No. 35

Figure 3: Please add a legend to Figure 3 to define the symbols used.

## Walter Coke Response No. 35

A legend has been added to Figure 3.

#### CLOSING

If you should have any questions, please do not hesitate to contact us at (205) 942-1289.

Sincerely, Terracon Consultants, Inc.

full i

Terrell W. Rippstein, AL-PG#8 Principal Geologist

Cc: Ms. Meredith Anderson; USEPA Region 4

# Interim Measures (IM) Groundwater Sampling and Analysis Plan (Revision 1.0)

Walter Coke 3500 35<sup>th</sup> Avenue North Birmingham, Jefferson County, Alabama USEPA ID No. ALD 000 828 848 October 9, 2012 Terracon Project No. E1127095



Prepared for: Walter Coke Birmingham, Alabama

## **Prepared by:**

Terracon Consultants, Inc. Birmingham, Alabama

Offices Nationwide Employee-Owned Established in 1965 terracon.com Geotechnical Environmental Construction Materials Facilities October 9, 2012



Walter Coke 3500 35<sup>th</sup> Avenue North Birmingham, Alabama 35207

Attention: Mr. Don Wiggins

 Re: Interim Measures (IM) Groundwater Sampling and Analysis Plan (Revision 1.0) Walter Coke
 3500 35<sup>th</sup> Avenue North
 Birmingham, Jefferson County, Alabama
 USEPA ID No. ALD 000 828 848
 Terracon Project No. E1127095

Dear Mr. Wiggins:

Terracon Consultants, Inc. (Terracon) is pleased to provide this Interim Measures (IM) Groundwater Sampling and Analysis Plan for the above-referenced site. This plan has been developed in response to the April 16, 2012, USEPA approval of the Interim Measures Work Plan and Addendum. A Health and Safety Plan (HASP) will be submitted under separate cover in addition to this Plan.

Where applicable, this plan references EPA Region 4 guidance documents and web addresses.

If you should have any questions, please do not hesitate to contact us at (205) 942-1289.

Sincerely, Terracon Consultants, Inc.

Tendy to ty

Terrell W. Rippstein, AL-PG#8 Principal Geologist

Cc: Ms. Meredith Anderson; USEPA Region 4

Attachments



Terracon Consultants, Inc. 110 12th Street North Birmingham, Alabama 35203 P [205] 942 1289 F [205] 443 5302 terracon.com

## Interim Measures (IM) Groundwater Sampling and Analysis Plan (Revision 1.0) Walter Coke 3500 35<sup>th</sup> Avenue North Birmingham, Jefferson County, Alabama EPA ID No. ALD 000 828 848

Terracon Project No. E1127095 October 9, 2012

## A-1: DISTRIBUTION LIST

Project contact information are described below. The personnel listed below will receive hard copies of this IM Groundwater Sampling and Analysis Plan.

#### **USEPA**, Region 4

Project CoordinatorRCRA Corrective Action SectionRestoration and Underground Storage Tank Branch61 Forsyth St. SWAtlanta, Georgia 30303Phone:(404) 562-8608Fax:(404) 562-9964Contact:Ms. Meredith AndersonEmail:anderson.meredith@epamail.epa.gov

#### Alabama Department of Environmental Management

Chief, Engineering Services Section Industrial Hazardous Waste Branch P.O. Box 301463 Montgomery, Alabama 36130-1463 Phone: (334) 271-7700

#### Walter Coke

3500 35<sup>th</sup> Avenue North
Birmingham, Alabama 35207
Phone: (205) 808-7972
Contact: Mr. Don Wiggins
Email: <u>don.wiggins@walterenergy.com</u>



## TERRACON

110 12<sup>th</sup> Street NorthBirmingham, Alabama 35203Phone:(205) 942-1289Fax:(205) 443-5302Contact:Mr. Terrell W. Rippstein, P.G., Project ManagerEmail:twrippstein@terracon.com

## TERRACON

4521 Bonny Oaks Drive
Chattanooga, Tennessee 37416
Phone: (423) 499-6111
Fax: (423) 499-8099
Contact: Mr. Dallas Whitmill, QA/QC Reviewer
Email: <u>dewhitmill@terracon.com</u>

## A-2: TABLE OF CONTENTS

A-1:	DISTRIBUTION LISTi				
A-2:	TABLE OF CONTENTSiii				
1.0	Intro	Introduction			
	1.1	Site Name or Sampling Area	1		
	1.2	Site or Sampling Area Location	1		
	1.3	Responsible Agency	2		
	1.4	Project Organization			
		1.4.1 Terracon Project Manager			
		<ul><li>1.4.2 QA/QC Reviewer</li><li>1.4.3 Site Personnel</li></ul>			
	1.5	Statement of the Specific Problem			
2.0	Bacl	kground			
	2.1	Site or Sampling Area Description			
	2.2	Geological Information			
3.0	PRO	JECT DATA QUALITY OBJECTIVES	7		
	3.1	Project Task and Problem Definition	7		
	3.2	Data Quality Objectives (DQOs)	7		
	3.3	Data Review and Validation			
		<ul> <li>3.3.1 Qualitative Data – Level A</li> <li>3.3.2 Quantitative Data – Level B</li> </ul>			
		3.3.3 Unusable Data			
	3.4	Data Management			
4.0	SAM	IPLING RATIONALE	12		
	4.1	Preconstruction Monitoring	12		
	4.2	Water Sampling/System Performance Monitoring	13		
5.0	REQ	UEST FOR ANALYSES	13		
	5.1	Analyses Narrative	13		
	5.2	Analytical Laboratory	15		
6.0	FIEL	D METHODS AND PROCEDURES	15		
5.0	6.1	Field Equipment			
		<ul><li>6.1.1 List of Equipment Needed</li><li>6.1.2 Calibration of Field Equipment</li></ul>			
	6.2	Groundwater Water Sampling			
	0.2	6.2.1 Water-Level Measurements			
		6.2.2 Purging			
	6.0	6.2.3 Well Sampling			
7.0	6.3				
7.0		IPLE CONTAINERS, PRESERVATION AND STORAGE			
	7.1	Water Samples			
8.0	DISF	POSAL OF RESIDUAL MATERIALS	19		

## TABLE OF CONTENTS (CONT.)

9.0	SAMPLE DOCUMENTATION AND SHIPMENT		.20
	9.1	Field Notes	
		9.1.1 Field Logbooks	
		9.1.2 Photographs	.21
	9.2	Labeling	.21
	9.3	Sample Chain-Of-Custody Forms and Custody Seals	.22
	9.4	Packaging and Shipment	.23
10.0	QUALITY CONTROL		.24
	10.1	Field Blanks	.24
	10.2	Trip Blanks	.24
	10.3	Temperature Blanks	.25
	10.4	Equipment Blanks	.25
	10.5	Duplicate Samples	.25
	10.6	Matrix Spike/Matrix Spike Duplicates	.26
11.0	FIEL	D VARIANCES	.26
12.0	FIEL	D HEALTH AND SAFETY PROCEDURES	.26

## APPENDICES

Appendix A :	Figure 1 – Former Chemical Plant (FCP) Location		
	Figure 2 – Topographic Map		
	Figure 3 – Containment Well and Monitoring Well Locations for the FCP		
Appendix B:	Terracon's Standard Operating Procedures (TSOP) Manual		
Appendix C:	Terracon's Corporate Quality Program Manual		
Appendix D:	Laboratory QA Manuals		

### ACRONYMS AND ABBREVIATIONS

µmhos/cm ..... micromhos per centimeter ACM..... Asbestos-Containing Material ADEM ..... Alabama Department of Environmental Management AEIRG..... Alabama Environmental Investigation and Remediation Guidance AHERA..... Asbestos Hazard Emergency Response Act AIHA ...... American Industrial Hygiene Association AMSL ..... Above Mean Sea Level ARARs ...... Applicable or Relevant and Appropriate Requirements ARBCA..... Alabama Risk-Based Corrective Action AST..... Aboveground Storage Tank BTEX ...... Benzene, Toluene, Ethylbenzene, and Xylenes BTS.....Biological Treatment System CAD ..... Computer-Aided Drafting CAWP ...... Cooperative Agreement Work Plan CERCLIS ...... Comprehensive Environmental Response, Compensation, and Liability Information System CFR ..... Code of Federal Regulations CIH ..... Certified Industrial Hygienist CORRACTS ...... RCRA TSD Corrective Action Facilities CSM..... Conceptual Site Model CWM..... Clear ,Wide Mouth Glass Jar DQO..... Data Quality Objective EDR ..... Environmental Data Resources, Inc. EPA ..... Environmental Protection Agency ESA ..... Environmental Site Assessment ESC ..... Environmental Science Corporation eV ..... electron-volt f/cc.....fibers per cubic centimeter of air FOIA ..... Freedom of Information Act GC ..... Gas Chromatograph GIS ..... Geographical Information System GPS ..... Geographical Positioning System GRO..... Ground water Remediation Objective GSA ..... Geological Survey of Alabama HASP ..... Health and Safety Plan ..... Hydrochloric Acid HCI HNO<sub>3</sub> ..... Nitric Acid HUD...... United States Department of Housing and Urban Development IDW..... Investigation Derived Waste IM ..... Interim Measures IMPDMENT ...... Impoundment IMWP ...... Interim Measures Work Plan L ..... liter LBP..... Lead-Based Paint LCS..... Laboratory Control Sample LQAP ..... Laboratory Quality Assurance Programs LUST..... Leaking Underground Storage Tank mg/kg..... milligrams per kilogram mL..... milliliter MS/MSD...... Matrix Spike/Matrix Spike Duplicate MWSR ...... Monitoring Well Sampling Record NaOH.....Sodium Hydroxide NELAC ...... National Environmental Laboratory Accreditation Conference

#### ACRONYMS AND ABBREVIATIONS (CONT.)

NESHAP ...... National Emissions Standards for Hazardous Air Pollutants NFR ..... No Further Remediation NIST......National Institute of Standards and Technology NLLAP ...... National Lead Laboratory Accreditation Program NTU ..... Nephelometric Turbidity Units NVLAP ...... National Voluntary Laboratory Accreditation Program OSHA..... Occupational Health and Safety Administration OVM..... Organic Vapor Meter oz.....ounce PAH ..... Polycyclic Aromatic Hydrocarbon PARCCS ...... Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity PCB ..... Polychlorinated Biphenyl PE..... Professional Engineer PG ..... Professional Geologist PLM ..... Polarized Light Microscopy PQL ..... Practical Quantitation Limit PSV ..... Preliminary Screening Value PVC ..... Polyvinyl Chloride QA ..... Quality Assurance QAPP......Quality Assurance Project Plan QC ..... Quality Control RA..... Remedial Applicant RCRA..... Resource Conservation and Recovery Act REC ...... Recognized Environmental Condition RO ..... Remediation Objective RPD ..... Relative Percent Difference RSL.....Regional Screening Level SIR.....Site Investigation Report SPLP..... Synthetic Precipitation Leaching Procedure SQG..... Small Quantity Generator SRO..... Soil Remediation Objective SRP ..... Site Remediation Program Stat Analysis...... Stat Analysis Corporation SVOC..... Semivolatile Organic Compound SWMU ...... Solid Waste Management Unit TACO...... Tiered Approach to Corrective Action Objectives TAL ...... Target Analyte List TCLP...... Toxicity Characteristic Leaching Procedure TSD ...... Treatment, Storage, and Disposal TSOP...... Terracon Standard Operating Procedures for EPA Brownfields UAS ...... United Analytical Services, Inc. USEPA...... United States Environmental Protection Agency USGS..... United States Geological Survey UST ..... Underground Storage Tank VCP ..... Voluntary Cleanup Program VOC..... Volatile Organic Compound VSP ..... Visual Sample Plan XRF .....X-Ray Fluorescence

## Interim Measures (IM) Groundwater Sampling and Analysis Plan (Revision 1.0) Walter Coke 3500 35<sup>th</sup> Avenue North Birmingham, Jefferson County, Alabama USEPA ID No. ALD 000 828 848

Terracon Project No. E1127095 October 9, 2012

## **1.0 INTRODUCTION**

This IM Groundwater Sampling and Analysis Plan (Plan) was prepared by Terracon Consultants, Inc. (Terracon) for use in implementing the final, modified, and approved Groundwater Interim Measures Work Plan (IMWP), dated May 2012, prepared by CH2MHill for the Former Chemical Plant (FCP) at the Walter Coke, Inc. (Walter Coke) facility located at 3500 35<sup>th</sup> Avenue North in Birmingham, Jefferson County, Alabama (Figure 1, Appendix A). The Work Plan and Addendum was approved by the USEPA in a letter dated April 16, 2012. Project activities will focus on the area of the FCP located on the site and in the offsite area east of the FCP.

This document will serve as the QA/QC document for the groundwater sampling performed during the IM. As such, this Plan will be supplemented by a site-specific HASP, to be submitted under separate cover. This Plan describes the process for producing analytical data of sufficient quality and quantity to accurately evaluate site conditions and meet data quality requirements under USEPA requirements.

This Plan will be used in conjunction with the Terracon Standard Operating Procedures (TSOP) Manual, included as Appendix B of this Plan. In addition to the TSOP manual, project QC will be further maintained through assessment and reporting practices consistent with the Terracon Corporate Quality Program Manual. This manual outlines internal QC procedures, formal review procedures, and standard company practice. A copy of the Terracon Corporate Quality Program Manual is included as Appendix C.

## 1.1 Site Name or Sampling Area

The IM is being conducted at the Walter Coke facility in the area known as the former chemical plant (FCP).

## 1.2 Site or Sampling Area Location

The Walter Coke facility is located at 3500 35<sup>th</sup> Avenue North in Birmingham, Alabama.



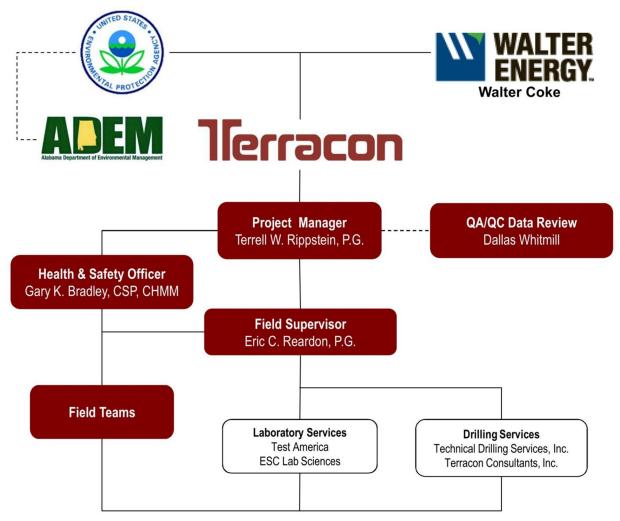
## **1.3 Responsible Agency**

Terracon will be implementing the IM Workplan for Walter Coke. The lead regulatory agency is the USEPA Region 4.

## 1.4 **Project Organization**

The *project organizational chart and table* included below illustrates the projected project team for the IM activities. However, subcontractors performing IM field activities (drilling, sampling, and laboratory analysis) may change. Any changes in subcontractors from those illustrated and discussed below will be included in a Plan Addendum. Subsequent sections discuss key personnel roles associated with each project.

## **Project Organizational Chart**





Title/Responsibility	Name	Phone Number
USEPA Project Manager	Meredith Anderson	(404) 562-8608
ADEM	Chief, Engineer Services Section	(334) 271-7700
Walter Coke Manager	Don Wiggins	(205) 808-7972
Terracon Project Manager	Terry Rippstein	(205) 443-5244
Quality Assurance Manager	Dallas Whitmill	(423) 499-6111
Field Supervisor	Eric Reardon	(205) 443-5218
ESC Lab Sciences (Analytical)	Jimmy Hunt	(800) 767-5859
TestAmerica (Analytical)	Pam Langford	(800) 765-0980
Terracon (Drilling)	Derek Hodnett	(423) 499-6111
Technical Drilling (Drilling)	Dette Lee	(205) 758-7454

## Table 1. Project Organization

Project activities will be organized and conducted in accordance with this Plan. Activities will be performed using various Terracon resources, relevant subcontractor resources, and management guidance and oversight from Walter Coke and the USEPA. The local project office for this assessment is the Terracon office in Birmingham, Alabama.

## 1.4.1 Terracon Project Manager

The Terracon Project Manager provides technical guidance, administration, and resources to direct project QA. A strong working knowledge of state and federal regulatory programs is essential to the position. *Mr. Terrell W. Rippstein, P.G., Birmingham Office* fills this role with more than 22 years of relevant experience. Mr. Rippstein draws local resources and staffing primarily from the Terracon office in Birmingham, Alabama. Mr. Rippstein will interact with the USEPA directly as needed. Mr. Rippstein is located less than ten miles from the Walter Coke facility. His oversight and technical duties beyond this project do not require more than short, regional travel.

## 1.4.2 QA/QC Reviewer

The QA/QC Reviewer (Terracon) provides documentation audits and technical review to assist in promoting, implementing, and documenting QA compliance. The QA/QC Reviewer is isolated from the implementation chain-of-command. This allows lateral support as a peer to the Project Manager without introducing unintentional biases from conducting the work. The QA/QC Reviewer must have extensive environmental and regulatory assessment experience at both the state and federal levels. **Mr. Dallas Whitmill** in our Chattanooga, Tennessee, office fills this roll. Mr. Whitmill has over fifteen years' experience in the environmental field. Mr. Whitmill is a senior member of the firm with extensive environmental and regulatory assessment and remediation experience.



## 1.4.3 Site Personnel

Terracon site personnel will have completed the Occupational Safety and Health Administration basic 40-hour health and safety training course, Hazardous Waste Operations and Emergency Response (HAZWOPER), including annual refreshers. Terracon field staff also complete inhouse training modules on-line through the *Terracon Learning System* (TLS), an online training system available via live webcasts, recorded webcasts, and self-paced online and offline options. Materials covered include: sample collection protocols, conventional and direct push drilling investigation techniques, decontamination procedures, and IDW management. TLS modules also include a "lessons learned" element designed to familiarize field staff with common problems encountered during field data collection and the appropriate corrective measures as a response to those problems.

All training records will be maintained in Terracon's Corporate Headquarters in Olathe, Kansas. Please refer to the Terracon Corporate Quality Program Manual included as Appendix C for more information related to training requirements.

## **1.5** Statement of the Specific Problem

Walter Coke located in Birmingham, Jefferson County, Alabama, has been conducting a Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) since 1990 in accordance with the regulations set forth by the RCRA Hazardous and Solid Waste Amendments (HSWA) to evaluate past waste management practices at its Birmingham, Alabama, facility. During the RFI, a groundwater plume was identified in the FCP located at the southeastern portion of the facility (Figures 1 and 2, Appendix A). Chemicals identified in groundwater beneath the FCP at concentrations above their respective maximum contaminant levels (MCLs) include benzene, toluene, chlorobenzene, and several chlorinated ethenes.

## 2.0 BACKGROUND

An IMWP, prepared and submitted to the USEPA in February 2002, included a detailed description of the conceptual geologic and hydrogeologic model for the site, which would affect groundwater flow from the FCP, along with an evaluation of several remedial options to reduce the chemical mass beneath the FCP and to prevent offsite migration of affected groundwater. An addendum to the 2002 IMWP was submitted to USEPA in February 2011 to address USEPA comments on the original submittal. On April 16, 2012, USEPA approved the IMWP, specifically approving Sections 2 and 5 of the original 2002 submittal and the 2011 Addendum, pending modification per USEPA comments. The final, modified, and approved Groundwater IMWP dated May 2012, prepared by CH2MHill was approved by USEPA on April 16, 2012.



## 2.1 Site or Sampling Area Description

The Walter Coke Facility is located in an industrial area in the northern portion of Birmingham, Alabama. A residential area lies south of the site across Shuttlesworth Drive. The FCP is located in the southeastern portion of the site (Appendix A, Figures 2 and 3). The monitoring wells and hydraulic control wells associated with the FCP are shown on Figure 3 (Appendix A). The monitoring wells which will be used to evaluate the effectiveness of the IM are listed on Table 2-1.

Well ID	Monitored Unit	Screened Interval	Depth to Bedrock
		(ft bgs)	(ft bgs)
MW-49S	SB	16-26	13.5
MW-50	SB	25-35	19
MW-51	SB	14-24	9
MW-52	SB	11.5-21.5	10.5
MW-53	SB	12-22	10
MW-54	SB	22-32	18
MW-55	SB	12-22	8
MW-56	SB	10-20	4
MW-77	SB	25-35	6.5
MW-78	SB	36-46	7.1
MW-80	SB	33-43	20
MW-81	SB	11-21	6.5
MW-70	SB	18.8-28.8	13.3
MW-71	SB	30.8-40.8	12
MW-72	SB	42.8-52.8	16
MW-90	SB	NYI	NYI
MW-49D	DB	159.5-169.5	13.5
MW-89	DB	280-300	6

Table 2-1. List of	Monitoring Wells	to Evaluate the IM

SB = Shallow Bedrock

DB = Deep Berock

NYI = Not Installed (MW-90 will be installed as part of the approved IM Work Plan)

## 2.2 Geological Information

The facility is underlain by sedimentary rocks that range in age from Cambrian to Pennsylvanian. The Opossum Valley Fault generally trends northeast to southwest, crossing through the Walter Coke property in the northern portion of the facility at the Polishing Pond (SWMU 22). The majority of the Walter Coke property lies on the hanging wall fault block to the east of the Opossum Valley Fault. The foot wall of the fault lies to the west and underlies Sand Mountain. The majority of the Walter Coke property is underlain by the Conasauga Formation. The Red Mountain Formation, Fort Payne Formation, Tuscumbia Limestone, Hartselle



Sandstone, Floyd Shale, and Pottsville Formation outcrop in a small area of the facility on the western side of the fault.

The Conasauga Formation is Cambrian-Aged and typically is medium gray, thin- to mediumbedded limestone. Locally, bedding thickness is reported to range from a few inches to as much as 5 feet or more in the massive sections. Locally, the Conasauga Formation dips to the southeast at 26 to 32 degrees, with a strike of approximately N45°E. An extensive network of faults and joints has developed in the Conasauga Limestone because of thrust faulting. The faults and joints typically trend northeast and northwest. The northeast trending joints (strike of N45°E) dip approximately 60°NW (approximately perpendicular to bedding), while the northwest trending joints strike 300°NW and have subvertical dips. The results of previous investigations indicate that the upper 2 feet of the Conasauga Formation underlying the Walter Coke facility are highly weathered. Below the weathered surface, the limestone is generally massive, with few fractures. The limestone is typically hard, with 1- to 2-foot-thick lenses of softer, darker gray shale and shaley limestone. Occasionally, fractures are present, ranging from a few inches to a few feet thick. Fracture zones typically contain limestone rubble that exhibits secondary healing by calcite crystals. Fracture zones typically are encountered in the upper 50 feet of the formation and are less frequent with increasing depth. On the western side of the Opossum Valley Fault (in the SWMU 23 area), outcrops of the Hartselle Sandstone, Tuscumbia Limestone, Fort Payne Chert, Red Mountain Formation, and Pottsville Formation have been mapped. Brief descriptions of these units are provided below:

- Hartselle Sandstone consists mainly of clean, well-sorted, light-colored, very fine- to medium-grained quartz sand;
- Tuscumbia Limestone consists of thick-bedded, medium-dark to medium-gray, crystalline, oolitic, sublithographic, and bioclastic limestone with minor amounts of chert;
- Fort Payne Chert consists of dark-gray sublithographic limestone and dense dark-gray chert;
- Red Mountain Formation consists of dark-reddish-brown to olive-gray siltstone, sandstone, and shale with hematite beds; and
- Pottsville Formation consists of alternating beds of sandstone and shale with numerous coal seams and associated underclays.

The topography of the bedrock underlying the Walter Coke facility generally slopes to the north toward Five Mile Creek. Top-of-bedrock elevations range from 583.1 feet amsl in the Coke Plant area to 498.6 feet amsl near Five Mile Creek. Weathering of the Conasauga Formation has produced undulations in the surface of the bedrock. Several feet of relief have developed on the bedrock surface. This relief is as much as several tens of feet in some areas of the property; however, karst features are not evident at the ground surface. Where exposed, enlargement of bedding planes and fractures appears to have occurred through solution of the bedrock. Solutionally enlarged fractures and joints primarily are limited to the upper few feet of bedrock and have been observed up to 1 foot wide.



The following text presents the current conceptual hydrogeologic flow mode. The conceptual hydrogeologic flow model is composed of residuum groundwater, shallow bedrock groundwater, and deep bedrock groundwater. Groundwater occurs within the residuum where the water table is higher than the bedrock surface. Groundwater flow through this material occurs in interstitial pore spaces between the clay particles at a low rate due to the relatively low permeability. Flow rates may be higher where a concentration of chert gravels at the bedrock surface has occurred, although based on borehole observations, such occurrence is limited. Within the shallow and deep bedrock aquifers, groundwater migrates along fractures and bedding planes both horizontally and vertically. Within the shallow bedrock aquifer, groundwater flow is primarily horizontal due to the interconnectivity of the fractures. Groundwater within the shallow bedrock discharges to surface water bodies such as the Lafarge and Southern Ready Mix Quarries, surface drainage ditches, and Five Mile Creek. Deep bedrock groundwater probably migrates toward discharge points such as the Lafarge and Southern Ready Mix Quarries. The groundwater flow in the area of Five Mile Creek is east towards Shuttlesworth Drive.

## 3.0 PROJECT DATA QUALITY OBJECTIVES

## 3.1 **Project Task and Problem Definition**

The performance objective of the hydraulic containment IM is to maintain an inward gradient at those locations along the downgradient property boundary where chemical concentrations have been detected above USEPA's tap water regional screening levels (RSLs). This Groundwater Sampling and Analysis Plan was prepared to address the groundwater sampling performed as part of the IM. VOCs were detected in the shallow groundwater at the Chemical Plant at concentrations exceeding the USEPA MCLs.

As specified in the IMWP, groundwater monitoring will be performed on a guarterly basis for a period of one year from the monitoring wells listed in Table 2-1 (Section 2.0), except for deep bedrock monitoring wells MW-49D and MW-89 which will be sampled prior to pumping and at the end of one year. After the one year period, we will reevaluate the sampling frequency. Groundwater samples will be analyzed for VOCs (USEPA Method 8260B), SVOCs (EPA Method 8270D), and PAHs (EPA Method 8270SIM) to assess the effectiveness of the groundwater control system. Low flow (minimal drawdown) sampling techniques will be used to sample wells in accordance with USEPA found the auidance at http://www.epa.gov/region4/sesd/fbgstp/index.html.

## 3.2 Data Quality Objectives (DQOs)

When performing the IM, measurements will be made so that results are reflective of the medium and conditions being measured. DQOs are applicable to all phases and aspects of the



data collection process during the IM. Groundwater samples will be collected for laboratory analysis as specified in the final, modified, and approved Groundwater IMWP, dated May 2012, prepared by CH2MHill. The DQOs will be consistent with the EPA guidance documents found at: <u>http://www.epa.gov/quality1/qs-docs/g4-final.pdf</u> and <u>http://www.epa.gov/QUALITY/qs-docs/r5-final.pdf</u>.

Groundwater data will be compared to the current USEPA drinking water maximum contaminant (MCLs) and USEPA Regional Screen Levels levels (RSLs) found at http://www.epa.gov/reg3hwmd/risk/human/rb-concentration table/Generic Tables/index.htm. A sub-group of VOCs/SVOCs of particular interest to the EPA at this time due to their presence in the FCP groundwater plume are: benzene, chlorobenzene, naphthalene, TCE, PCE, cis-1,2-dichloroethene, 1,2-dichloroethane, 1,2,4-trichlorobenzene, cresol. 1-methyl naphthalene, 2-methyl naphthalene, PAHs, 1,1-dichloroethene, and vinyl chloride. The comparison of the data collected verses the MCLs/RSLs, and the trends of the data will be evaluated after the first year to determine the effectiveness of the hydraulic control system and is modifications are necessary.

One common problem in comparison of the regulatory criteria with site data is overcoming matrix interferences or elevated detection limits. In many samples, a consortium of analytes and non-target analytes may be present. Weathered hydrocarbons, samples with one or more highly elevated metals, and free-phase organic liquids can create a situation in which analytes of interest cannot be detected at normal levels. This can be especially frustrating as guite often there may be no target analyte present in the sample. However, site management decisions cannot be supported because sample reporting limits had to be elevated to the point that they are above applicable action levels. Roughly 95% of such cases can be resolved. Solutions must be evaluated on a case by case basis, but alternative solutions can include: sample extract cleanup in the case of organics; the sample may be re-analyzed at lower dilutions if possible (in such a case both the diluted and undiluted results may be reported); or an alternate test method that offers a lower reporting limit value may be used. An example of an alternate test method would be re-analyzing a metals sample using graphite furnace atomic absorption spectroscopy or inductively coupled plasma (ICP) mass spectrometry as opposed to traditional spectra-scanning methods with an ICP alone. The laboratory analytical methods used during this groundwater sampling conducted during the IM will allow the resulting data to be reliably compared against the appropriate screening levels (i.e., method detection limits (MDLs) are equal to or less than the RSLs). If the laboratory is unable to achieve an MDL at or below the RSL for any constituent, then it will be assumed that the particular constituent exceeds the RSL.

There are, unfortunately, a small percentage of samples which cannot be reconciled against detection limits and regulatory criteria. In these cases, the result must be scrutinized versus the entire data set and project goals. The resultant opinion of data quality and usability will then be incorporated as an element of uncertainty in the report.

IM Groundwater Sampling and Analysis Plan (Revision 1.0) Walter Coke 
Birmingham, Alabama October 9, 2012 
Terracon Project No. E1127095



As discussed above, it is important to note that the level of detail and data quality needed will vary with the intended use of the data. Data are typically assessed by evaluating PARCC (Precision, Accuracy, Representativeness, Completeness, and Comparability) of all aspects of the data collection process. PARCC for this project will be defined as:

*Precision*: a measure of the reproducibility of analyses under a given set of conditions compared to the criteria of the individual laboratory's Quality Assurance Manual.

Matrix precision is calculated using equation (1).

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100, \tag{1}$$

RPD = Relative Percentage Difference

D1 = First sample value

- D2 = Second sample value (duplicate)
- Accuracy: a measure of the bias that exists in a measurement system compared to the criteria of the individual laboratory's Quality Assurance Manual.

For accuracy analysis; the percent recovery is calculated using equations (2) and (3).

$$LCS = \frac{Amount \ of \ Spike \ Analyte \ Detected}{Known \ Amount \ of \ Spike \ Analyte \ Added} \times 100,$$
(2)

LCS = Laboratory Control Sample

$$MS (or MSD) = \frac{Total Amount of Analyte Detected - Amount of Analyte Detected in Sample}{Known Amount of Spike Analyte Added} \times 100$$
(3)

MS (or MSD) = Matrix Spike (or Matrix Spike Duplicate)

- Representativeness: the degree sampling data accurately and precisely depict selected characteristics will rely highly on maintaining conformance to established protocols as established by TSOPs, laboratory QA/QC protocol, and/or USEPA/ADEM standard operating procedures.
- *Completeness*: the measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under "normal" conditions. This goal will be accomplished if 95% of



Walter Coke Birmingham, Alabama October 9, 2012 Terracon Project No. E1127095

design samples are taken and found to be qualified for precision and accuracy.

Comparability: the degree of confidence with which one data set can be compared to another. This goal will be achieved by compliance with very specific requirements established by Terracon Standard Operating Procedures for USEPA Brownfields (TSOPs), laboratory standard Operating Procedures (SOPs) and/or USEPA/ADEM standard operating procedures.

## 3.3 Data Review and Validation

Analytical data review and validation will be accomplished according to the laboratory QA manuals included in Appendix D; however, data meeting *analytical* validity requirements set by the analytical method and the fixed-laboratory need additional validation against the project-specific DQOs outlined within this document. Terracon will conduct an internal data review and verification. This data validation will be performed by a qualified Terracon professional outside of the project implementation chain-of-command, in accordance with in the Terracon Corporate Quality Program Manual provided as Appendix C, and this project's DQOs.

The data collected will be reviewed per Tier 1A/1B level of validation for the following components (where applicable):

- Completeness Check.
- Chain of Custody (signatures, sample conditions, preservatives, sampling handling/filtering).
- Holding Times.
- Random check (10-20%) of Initial and Continuing Calibration.
- Review of Quality Control Summaries including negative control (blanks) and positive control (LCS).
- along with Sample Specific Controls (replicates, matrix spikes, surrogates, tracers/ yields).
- Overall PARCC + S assessment.

Data usability determination is also a part of data evaluation. After data validity has been assessed, that is, the analytical data has been reviewed and qualifier codes have been applied, these data must be individually identified and assessed for usability. Within any matrix it is likely certain samples may have parameters that require qualifier codes; sample data (both with and without qualifier codes) may be generally spoken of as qualitative (Level A), quantitative (Level B), or unusable.



## 3.3.1 Qualitative Data – Level A

Qualitative data can include "J" coded data. These data are considered to be an estimated quantity, i.e., presence or absence type value. Any sample data receiving an unexplained qualifier code will be considered unusable for making site decisions. Data that have been given a "J" code are not automatically classified as qualitative data only; these data may be considered as quantitative data depending on bias, and must be evaluated on a case by case basis. Data reported with a "U" code (undetected) may also be classified as qualitative data provided the detection limit is not above an applicable regulatory action level for that analyte.

### 3.3.2 Quantitative Data – Level B

Only data meeting all field and analytical data usability requirements may be classified as quantitative data. This means all quality assurance parameters have been satisfied, including quality control and quality assessment. Only data that were found to be analytically valid and passed all criteria for qualitative data may be considered for classification as quantitative data. These data are definitive and may be used for any purpose.

## 3.3.3 Unusable Data

If a significant data failure occurs (i.e. critical Level B data classified as "unusable"), the data will not be used or may be considered as solely qualitative in nature. Data failures will be given more extensive consideration and result in a written opinion by the Terracon Project Manager & Terracon Data Validator as to usability relative to project-specific DQOs. Often, individual analytes within a larger, multi-analyte data set for a particular sample may be classified as "unusable"; however, the balance of data for a particular sample may meet Level B criteria. Key to the reconciliation will be the following considerations;

- Is invalid, qualified, or tentatively identified compound (TIC) data the sole determinant of "clean"? In other words, are there other analytes present within the sample or in other comparable samples which support this data?
- Does inclusion of the flawed method or qualified data point/set skew the overall site characterization as related to the null hypothesis?
- What is the relative risk of being "wrong" with respect to known redevelopment goals should qualified, invalid, or TIC data be used?

The resultant opinion, as well as any relevant comment by other project stakeholders, will be incorporated as an element of uncertainty in the assessment reports, revisions, and addenda deliverables. Limitations on the use of the data produced during the each assessment will be included in each site-specific final report.

IM Groundwater Sampling and Analysis Plan (Revision 1.0) Walter Coke 
Birmingham, Alabama October 9, 2012 
Terracon Project No. E1127095



## 3.4 Data Management

Raw data from the laboratory will be provided to Terracon electronically. The electronic data will be used to directly populate tables for reporting purposes. In addition, field data may be entered onto tables or into forms for use in reporting. Terracon personnel not directly involved in the data input, perform a data check on all manually entered data to ensure data is not transposed or incorrectly typed.

Terracon maintains electronic files on the local office server. This information is backed up daily from Terracon's Corporate Data Center. Once the final report is generated, all electronic files except the final report are deleted. At initial project closing, hard copy files are purged of all documents not provided to the client or other third party. Final reports, site photos, internal memos, permits, and laboratory data are retained. Project closing is 30 days after the last project activity has ceased. A second purge is conducted at three years after project closing. All documents are removed from the file except final reports and documents provided to the client or third parties. These documents are retained at the local office indefinitely.

Terracon will submit the data to the USEPA in the USEPA Region 4 electronic data delivery (EDD) format (<u>http://www.epa.gov/region4/superfund/allresource/edd/edd.html</u>).

## 4.0 SAMPLING RATIONALE

## 4.1 **Preconstruction Monitoring**

Monthly water levels will be collected from the wells summarized in Table 2-1, and shown in Figure 3 (Appendix A), for 3 months before installing the recovery wells to establish a baseline for hydraulic gradient. The selected Shallow bedrock wells are screened within the shallow bedrock at depths ranging from 10 to 50 feet below ground surface (ft bgs). The two deep bedrock wells are screened approximately 160 to 300 ft bgs.

Once the entire IM is operational, monthly water levels will be collected manually for 6 months in the wells listed in Table 2-1, followed by quarterly monitoring for the remainder of the year.

In addition, a transducer study will be conducted along the property boundary at MWs-49S, 50, and 51. The transducers will be installed in the wells one week before system startup and continue for 1 month past system startup. These data will be used to demonstrate hydraulic connectivity at the monitoring well locations and demonstrate the inward gradient.



## 4.2 Water Sampling/System Performance Monitoring

Based on the approved IMWP and Addendum, groundwater samples will be collected from the 18 monitoring wells listed in Table 2-1 prior to system startup. Groundwater samples from the 16 shallow bedrock wells and a groundwater sample will be collected from the combined flow of groundwater being recovered from the containment wells on a quarterly schedule for a period of one year. Groundwater samples from the two deep bedrock monitoring wells will also be collected at the end of one year. The groundwater samples and QA/QC samples (discussed in Section 10.0) will be analyzed for VOCs by USEPA Method 8260B, SVOCs by USEPA Method 8270D, and PAHs by USEPA Method 8270SIM.

Groundwater levels will be collected from all 18 monitoring wells listed in Table 2-1 prior to system startup and then quarterly for the first year after system startup.

The concentrations of the chemicals detected in the combined flow sample will be multiplied by the volume of water extracted from the system during the monitoring period to calculate the total mass extracted in pounds. In addition to the mass (pounds) removed, the total volume of groundwater recovered will be reported in gallons. These data will be reported quarterly to USEPA and the Alabama Department of Environmental Management (ADEM). At the end of the initial (1- year) period, an evaluation will be performed to investigate whether there is additional value to be provided by continued quarterly sampling. Walter Coke will provide the results of this evaluation to USEPA.

## 5.0 REQUEST FOR ANALYSES

## 5.1 Analyses Narrative

Groundwater samples will be collected prior to system start up and quarterly from the 16 shallow bedrock monitoring wells listed in Table 2-1. A groundwater sample will be collected quarterly from the combined flow of groundwater being recovered from the containment wells, and a groundwater sample will be collected prior to system start up and at one year after start up for the two deep bedrock monitoring wells listed in Table 2-1. The results from this sampling will be used to determine the effectiveness of the IM (Table 5-1). The samples will be analyzed for VOCs by USEPA Method 8260B, SVOCs by USEPA Method 8270D, and PAHs by USEPA Method 8270SIM (Table5-2).



October 9, 2012 
Terracon Project No. E1127095

## Table 5-1. Sample Summary.

Sample Media	Minimum Number of Samples	Analyses (Method)	Rationale
Groundwater	16 shallow bedrock monitoring wells, 2 deep bedrock monitoring wells, and one combined flow sample from the pumping of the containment wells	VOCs (8260B), SVOCs (8270D), and PAHs (8270SIM)	Determine effectiveness of the IM containment wells
Field blanks	1 per 20 samples or 1 per day of field activities	VOCs (8260B)	Evaluate the effects of ambient conditions and sample containers on accuracy
Trip Blanks	1 per cooler containing samples for VOC analysis	VOCs (8260B)	Evaluate how shipping and handling procedures are affecting accuracy by introducing contaminants into the samples
Temperature Blanks	1 per cooler	Thermometer	Evaluate sample temperature effects on accuracy
Equipment blanks	1 per 20 samples or 1 per day of field activities	VOCs (8260B), SVOCs (8270D), and PAHs (8270SIM)	Evaluate sample equipment and/or field decontamination effects on accuracy
Duplicate Samples	1 for every 10 samples collected	VOCs (8260B), SVOCs (8270D), and PAHs (8270SIM)	Assess the effects of sample collection technique on sample precision
Matrix spike/matrix spike duplicate	1 per 20 samples or 1 per day of field activities	VOCs (8260B), SVOCs (8270D), and PAHs (8270SIM)	Evaluate the matrix effects on sample precision

Note: For more information on QA/QC samples such as Matrix Spikes or Duplicates. See Section 10.

## Table 5-2. Standard Analytical Methods for Aqueous Samples.

Contaminant of Concern	Analytical Method	Packaging and Preservation	Maximum Holding Times
VOCs	USEPA 8260B	(3) 40-mL vials preserved with HCl	14 days
SVOCs	USEPA 8270D	(2) 1-L amber glass unpreserved	7 days
PAHs	USEPA 8270SIM	(2) 1-L amber glass unpreserved	7 days



## 5.2 Analytical Laboratory

Laboratory reporting quality will be enhanced through a formalized process and management system applied to ensure data quality within standard method requirements. Laboratory reporting will occur consistent with the laboratory QA Manuals (Appendix D). In addition, laboratory reports will include a final data quality documentation package for all analyses.

A list of information to be supplied with laboratory data deliverables is as follows:

- Standard QC Data Package Provided by the analytical laboratory, with final analytical report with qualifiers (where necessary)
- Chain of Custody Form
- Method Blank
- Matrix Spike/Spike Duplicate Summary (MS/MSD)-with control limits
- Laboratory Control Sample Summary (LCS)-with control limits
- Reporting Limits listed on all reports
- Surrogate Recoveries for GC and GC/MS analyses (on final report)
- Method Detection Limits (MDLs)

After validation of each laboratory package is completed, laboratory results will be summarized in tabular form. The data summary report will include tabular summaries of analytical testing results, laboratory reports, and a summary of data validation conclusions. In addition to the data summary report, data collected during the project will be summarized in the final report.

Laboratory audit and associated corrective action records will be maintained within laboratory QC records. Individual records may be reviewed as determined relevant to ensure QC on a project-by-project basis. Corrective actions taken in response to audit or QC data review findings will be evaluated by the Terracon Project Manager and/or the QA/QC reviewer and discussed in the final report.

## 6.0 FIELD METHODS AND PROCEDURES

## 6.1 Field Equipment

Testing, inspection, and maintenance of all sampling equipment, field instrumentation, and health and safety monitoring instrumentation will be performed by Terracon personnel prior to any deployment for field activities. Testing, inspection, and maintenance will be performed in accordance with referenced SOPs, manufacturers' recommendations, and the EPA Region 4 Field Branches Quality System and Technical Procedures (<u>http://www.epa.gov/region4/sesd/fbqstp/index.html</u>).

Walter Coke Birmingham, Alabama October 9, 2012 Terracon Project No. E1127095



## 6.1.1 List of Equipment Needed

The equipment needed during the IM include:

- Pumps
- Dedicated disposable tubing
- Water level meter
- pH/specific conductance meter
- water level meter
- Water level transducers
- Safety equipment

## 6.1.2 Calibration of Field Equipment

Field instrumentation and health and safety monitoring instrumentation will be calibrated daily or in accordance with manufacturers' recommendations by Terracon personnel. These tasks will be conducted in accordance with the SOPs referenced in Appendix B, the EPA Region 4 Field Branches Quality System (FBQS) and Technical Procedures found at http://www.epa.gov/region4/sesd/fbqstp/index.html. manufacturers' recommendations. and Calibration activities will be recorded in the field notes.

## 6.2 Groundwater Water Sampling

## 6.2.1 Water-Level Measurements

All field meters will be calibrated according to manufacturer's guidelines and specifications before and after every day of field use. Field meter probes will be decontaminated before and after use at each well. Decontamination and water level measurement procedures will follow the FBQS (http://www.epa.gov/region4/sesd/fbqstp/index.html).

If well heads are accessible, all wells will be sounded for depth to water from top of casing and total well depth prior to purging. An electronic sounder, accurate to the nearest +/- 0.01 feet, will be used to measure depth to water in each well. When using an electronic sounder, the probe is lowered down the casing to the top of the water column, the graduated markings on the probe wire or tape are used to measure the depth to water from the surveyed point on the rim of the well casing. Typically, the measuring device emits a constant tone when the probe is submerged in standing water and most electronic water level sounders have a visual indicator consisting of a small light bulb or diode that turns on when the probe encounters water. Total well depth will be sounded from the surveyed top of casing by lowering the weighted probe to the bottom of the well. The weighted probe will sink into silt, if present, at the bottom of the well



screen. Total well depths will be measured by lowering the weighted probe to the bottom of the well and recording the depth to the nearest 0.1 feet.

Water-level sounding equipment will be decontaminated before and after use in each well. Water levels will be measured in wells which have the least amount of known contamination first. Wells with known or suspected contamination will be measured last.

# 6.2.2 Purging

The wells will be purged accordance with the FBQS found at in http://www.epa.gov/region4/sesd/fbgstp/index.html using pumps and tubing specified in the FBQS. If the well casing volume is known, a minimum of three casing volumes of water will be purged. When a submersible pump is used for purging, clean flexible, disposable, dedicated tubing will be used for groundwater extraction. Pumps will be placed as described in the FBQS.

Water will be collected into a measured bucket to record the purge volume. Casing volumes will be calculated based on total well depth, standing water level, and casing diameter. One casing volume will be calculated as:

$$V = \pi r^2 h$$

where:

V is the volume of one well casing of water (in ft<sup>3</sup>);
r is the inner radius of the well casing (in feet);
h is the total depth of water in the well (in feet).

It is most important to obtain a representative sample from the well. Stable water quality parameter (pH and specific conductance) measurements indicate representative sampling is obtainable. Water quality is considered stable if for three consecutive readings:

- PH varies by no more than 0.1 pH units
- specific conductance readings are within 5% of the average; and
- turbidity is less than 10 NTUs.

The water in which measurements were taken will not be used to fill sample bottles.

If the well casing volume is known, measurements will be taken before the start of purging, in the middle of purging, and at the end of purging each casing volume. If water quality parameters are not stable after 5 casing volumes, purging may cease as described in the FBQS, which will be noted in the logbook, and ground water samples will be taken. The depth to water, water quality measurements and purge volumes will be entered in the logbook.



The "Low Flow" purging method may also be used. If used the method described in the FBQS will be used and as with the traditional purging method all chemical parameters will be stable, as described above, prior to sampling.

If a well dewaters during purging and three casing volumes are not purged, that well will be allowed to recharge up to 80% of the static water column and dewatered once more. After water levels have recharged to 80% of the static water column, groundwater samples will be collected.

# 6.2.3 Well Sampling

At each sampling location, all bottles designated for a particular analysis (e.g., volatile organic compounds) will be filled sequentially before bottles designated for the next analysis are filled (e.g., semivolatile organic compounds). If a duplicate sample is to be collected at this location, all bottles designated for a particular analysis for both sample designations will be filled sequentially before bottles for another analysis are filled. In the filling sequence for duplicate sample bottles with the two different sample designations will alternate (e.g., volatile organic compounds designation GW-2, volatile organic compounds designation GW-2, netals designation GW-4 (duplicate of GW-2), metals designation GW-2, metals designation GW-4 (duplicate of GW-2). Groundwater samples will be transferred from the pump tap directly into the appropriate sample containers with preservative, if required, chilled if appropriate, and processed for shipment to the laboratory. When transferring samples, care will be taken not to touch the tap to the sample container.

Samples for volatile organic compound analyses will be collected as described in the FBQS (http://www.epa.gov/region4/sesd/fbqstp/index.html). Vials for volatile organic compound analysis will be filled first to minimize the effect of aeration on the water sample. The test vials will come from the lab with hydrochloric acid (HCI) for preservation. The vials will be filled directly from the pump tap and capped. The vial will be inverted and checked for air bubbles to ensure zero headspace. If a bubble appears, the vial will be discarded and a new sample will be collected.

## 6.3 Decontamination Procedures

The decontamination procedures that will be followed are in accordance with the EPA Region 4 FBQS (http://www.epa.gov/region4/sesd/fbqstp/index.html). Decontamination of sampling equipment must be conducted consistently as to assure the quality of samples collected. All equipment that comes into contact with potentially contaminated soil or water will be decontaminated. Disposable equipment intended for one-time use will not be decontaminated, but will be packaged for appropriate disposal. Decontamination will occur prior to and after each use of a piece of equipment.



The following, to be carried out in sequence, is an USEPA recommended procedure for the decontamination of sampling equipment:

- Non-phosphate detergent and tap water wash, using a brush if necessary
- Tap-water rinse
- Deionized/distilled water rinse
- Deionized/distilled water rinse (twice)

Equipment will be decontaminated in a pre-designated area on pallets or plastic sheeting, and clean bulky equipment will be stored on plastic sheeting in uncontaminated areas. Cleaned small equipment will be stored in plastic bags. Materials to be stored more than a few hours will also be covered.

# 7.0 SAMPLE CONTAINERS, PRESERVATION AND STORAGE

Sample containers and other dedicated consumables will meet USEPA criteria for cleaning procedures required for low-level chemical analysis. Sample containers will have Level II certification provided by the manufacturer, in accordance with pre-cleaning criteria established by USEPA in "Specifications and Guidelines for Obtaining Contaminant-Free Sample Containers." The certificates of cleanliness are maintained by the container suppliers, and can be obtained upon request using the container batch and lot numbers. Preservatives, if required, will be added by the laboratory to the containers prior to shipment to Terracon.

## 7.1 Water Samples

Water samples to be analyzed for VOCs will be collected in 40-mL glass vials. 1:1 hydrochloric acid (HCl) will be added to the vial at the laboratory prior to shipment and sample collection. The vials will be filled so that there is no headspace. The samples will be chilled to 4°C immediately upon collection. Three vials of each water sample are required.

Water sample to be analyzed for SVOCs and PAHs will be collected in two 1-L amber glass jars with no preservatives. The samples will be chilled to 4°C immediately upon collection. Two jars for each analysis of each water sample are required.

# 8.0 DISPOSAL OF RESIDUAL MATERIALS

In the process of collecting environmental samples at the Walter Coke facility during the IM, the Terracon sampling team will generate the following types of IDW:



Walter Coke Birmingham, Alabama October 9, 2012 Terracon Project No. E1127095

- Purged groundwater and excess groundwater collected for sample container filling.
- Used PPE and disposable equipment.
- Decontamination fluids (i.e., deionized water, residual contaminants, and water with non-phosphate detergent. The volume and concentration of the decontamination fluid will be sufficiently low to allow disposal).

The disposal of the IDW will be conducted in accordance with the EPA Region 4 FBQS (http://www.epa.gov/region4/sesd/fbqstp/index.html).

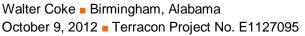
# 9.0 SAMPLE DOCUMENTATION AND SHIPMENT

## 9.1 Field Notes

## 9.1.1 Field Logbooks

At a minimum, the following information will be recorded during the collection of each sample:

- Sample location and description
- A unique sample ID for each sample collected in accordance with the USEPA guidance for electronic deliverables (<u>http://www.epa.gov/region4/superfund/allresource/edd/</u> edd.html)
- Site or sampling area sketch showing sample location and measured distances
- Sampler's name(s)
- Date and time of sample collection
- Designation of sample as composite or grab
- Type of sample (soil, sediment or water)
- Type of sampling equipment used
- Field instrument readings and calibration
- Field observations and details related to analysis or integrity of samples (e.g., weather conditions, noticeable odors, colors, etc.)
- Preliminary sample descriptions (e.g., for soils: clay loam, very wet; for water: clear water with strong ammonia-like odor)
- Sample preservation
- Lot numbers of the sample containers, sample identification numbers and any explanatory codes, and chain-of-custody form numbers
- Shipping arrangements (overnight air bill number)
- Name(s) of recipient laboratory(ies)





In addition to the sampling information, the following specific information will also be recorded in the field logbook for each day of sampling:

- Team members and their responsibilities
- Time of arrival/entry on site and time of site departure
- Other personnel on site
- Summary of any meetings or discussions with contractor or federal agency personnel
- Deviations from sampling plans or site safety plans
- Changes in personnel and responsibilities with reasons for the changes
- Levels of safety protection
- Calibration readings for any equipment used and equipment model and serial number

Field measurements performed by Terracon or subcontractor personnel will be documented through development of field analytical reports and/or field analysis reporting documents. These reports will document field methods, results, and any necessary deviations, in addition to any corrective measures taken in response to field audit findings. All information provided through field analysis reports will be incorporated accordingly into the report. The significance of deviations and actions requiring corrective action will be evaluated by the Terracon Project Manager and/or the QA/QC reviewer before report development.

## 9.1.2 Photographs

Photographs will be taken at the sampling locations and at other areas of interest on site or sampling area. They will serve to verify information entered in the field logbook. For each photograph taken, the following information will be written in the logbook or recorded in a separate field photography log:

- Time, date, location, and weather conditions
- Description of the subject photographed
- Name of person taking the photograph
- The unique sample ID (if applicable)

## 9.2 Labeling

All samples collected will be labeled in a clear and precise way for proper identification in the field and for tracking in the laboratory. The samples will have pre-assigned, identifiable, and unique sample ID numbers as presented in the USEPA guidance for electronic deliverables (<u>http://www.epa.gov/region4/superfund/allresource/edd/edd.html</u>). At a minimum, the sample labels will contain the following information:

IM Groundwater Sampling and Analysis Plan (Revision 1.0) Walter Coke Birmingham, Alabama

October 9, 2012 - Terracon Project No. E1127095



- Station ID and location
- Unique sample ID number
- Date and time of collection
- Analytical parameter(s), and
- Method of preservation.

Every sample, including samples collected from a single station location but going to separate laboratories, will be assigned a unique sample number. A station ID is not a sample ID. Sample IDs will be distinct from station IDs to prevent confusion.

In accordance with the USEPA guidance for electronic deliverables (http://www.epa.gov/region4/superfund/allresource/edd/edd.html), station ID designations will be less than 8 characters with no spaces dashed or underscores. The station ID will be used as part of the unique sample ID. For example if a unique groundwater sample is designated as MW12-0810 (monitoring well number, August 2010), the monitoring wells station ID is MW12. The monitoring well station ID will remain the same each time it is sampled; however, the month/year designation will give it a unique number.

## 9.3 Sample Chain-Of-Custody Forms and Custody Seals

Chain-of-custody record forms are used to document sample collection and shipment to laboratories for analysis.

All sample shipments for analyses will be accompanied by a chain-of-custody (COC) record. A COC will be completed and sent with the samples for each laboratory and each shipment (i.e., each day). If multiple coolers are sent to a single laboratory on a single day, form(s) will be completed and sent with the samples for each cooler.

The chain-of-custody form will identify the contents of each shipment and maintain the custodial integrity of the samples. Generally, a sample is considered to be in someone's custody if it is either in someone's physical possession, in someone's view, locked up, or kept in a secured area that is restricted to authorized personnel. Until the samples are shipped, the custody of the samples will be the responsibility of Terracon. The sampling team leader or designee will sign the chain-of-custody form in the "relinquished by" box and note date, time, and air bill number.

The sample numbers for all rinseate samples, reference samples, laboratory QC samples, and duplicates will be documented on this form. A photocopy or duplicate form will be placed in Terracon's masterfiles.

A self-adhesive custody seal will be placed across the lid of each sample. For VOC samples, the seal will be wrapped around the cap. The shipping containers in which samples are stored



(usually a sturdy cooler or ice chest) will be sealed with self-adhesive custody seals any time they are not in someone's possession or view before shipping. All custody seals will be signed and dated.

## 9.4 Packaging and Shipment

Sample containers will be placed in a hard sided shipping container (sturdy cooler). The following outlines the packaging procedures that will be followed for low concentration samples.

- 1. When ice is used, pack it in zip-locked, double plastic bags. Seal the drain plug of the cooler with fiberglass tape to prevent melting ice from leaking out of the cooler.
- 2. The bottom of the cooler should be lined with bubble wrap to prevent breakage during shipment.
- 3. Check screw caps for tightness and, if not full, mark the sample volume level of liquid samples on the outside of the sample bottles with indelible ink.
- 4. Secure bottle/container tops with clear tape and custody seal all container tops.
- 5. Affix sample labels onto the containers with clear tape.
- 6. Wrap all glass sample containers in bubble wrap to prevent breakage.
- 7. Seal all sample containers in heavy duty plastic zip-lock bags. Write the sample numbers on the outside of the plastic bags with indelible ink.
- 8. Place samples in a sturdy cooler(s) lined with a large plastic trash bag. Enclose the appropriate COC(s) in a zip-lock plastic bag affixed to the underside of the cooler lid.
- 9. Fill empty space in the cooler with bubble wrap or Styrofoam peanuts to prevent movement and breakage during shipment.
- 10. Ice used to cool samples will be double sealed in two ziplock plastic bags and placed on top and around the samples to chill them to the correct temperature.
- 11. Each ice chest will be securely taped shut with fiberglass strapping tape, and custody seals will be affixed to the front, right and back of each cooler.

Records will be maintained by Terracon's sample custodian of the following information:

- Name and location of the site or sampling area
- Total number(s) by estimated concentration and matrix of samples shipped to each laboratory
- Carrier, air bill number(s), method of shipment (priority next day)
- Shipment date and when it should be received by lab
- Irregularities or anticipated problems associated with the samples
- Whether additional samples will be shipped or if this is the last shipment.



# **10.0 QUALITY CONTROL**

In order to assess sample technique, field conditions, decontamination, and sample transport effects on precision and accuracy, the following QC samples will be required during each sampling event:

- Field blanks (1 per 20 samples or 1 per day of field activities)
- Trip Blanks (1 per cooler containing samples for VOC analysis)
- Temperature Blanks (1 per cooler)
- Equipment blanks (1 per 20 samples or 1 per day of field activities)
- Duplicate Samples (1 for every 10 samples collected)
- Matrix spike/matrix spike duplicate (1 per 20 samples or 1 per day of field activities)

These QC samples are also shown on Table 5-1. Please refer to the EPA's guidance document on quality assurance project plans at <u>http://www.epa.gov/QUALITY/qs-docs/r5-final.pdf.</u>

## 10.1 Field Blanks

Field blanks will be collected to evaluate the effects of ambient conditions and sample containers on accuracy. Field blank samples will be obtained by pouring HPLC organic-free water (for organics) and/or deionized water (for inorganics) into a sampling container at the sampling point. The field blanks that are collected will be analyzed for VOCs.

The field blanks will be preserved, packaged, and sealed in the manner described for the environmental samples. A separate sample number and station number will be assigned to each sample, and it will be submitted to the laboratory.

## 10.2 Trip Blanks

Trip blanks will be prepared to evaluate how shipping and handling procedures are affecting accuracy by introducing contaminants into the samples. A minimum of one trip blank per cooler containing VOCs samples will be submitted to the laboratory for VOC analysis. Trip blanks are laboratory-prepped 40mL vials that have been filled with HPLC-grade water that has been purged so it is VOC free. The blank is shipped with the empty sampling containers to the site or sampling area prior to sampling. The sealed trip blanks are not opened in the field and are shipped to the laboratory in the same cooler with the samples collected for VOCs analyses. The trip blanks will be preserved, packaged, and sealed in the manner described for the environmental samples. A separate sample number and station number will be assigned to each trip blank, and the blank will be submitted to the laboratory.



## **10.3 Temperature Blanks**

In order to evaluate sample temperature effects on accuracy, sample temperature will be checked using a temperature blank. For each cooler that is shipped or transported to an analytical laboratory, a single laboratory-prepped container of HPLC-grade water will be included that is marked "temperature blank." This blank will be used by the sample custodian to check the temperature of samples upon receipt.

## 10.4 Equipment Blanks

Equipment blanks are used to evaluate sample equipment and/or field decontamination effects on accuracy. Equipment blanks are prepared in-field in the same manner as a field blank with the added stipulation that they be collected through the sampling equipment. In the case of dedicated sampling equipment which contacts a sample (meters, pumps, etc.), the blank will be collected by pouring HPLC-grade water over, through, or around the sample device in order to evaluate field decontamination and point-to-point cross contamination. In the case of dedicated sampling equipment (tubing, bailers, etc.), the sample equipment will be set up in as similar a manner as possible to actual conditions, and a sample of HPLC-grade water will be collected through the dedicated sampling equipment. The equipment blanks will be preserved, packaged, and sealed in the manner described for the environmental samples. A separate sample number and station number will be assigned to each equipment blank, and the blank will be submitted to the laboratory for analysis of VOCs, SVOCs, and PAHs.

## **10.5 Duplicate Samples**

Duplicate samples for this project will be collected to assess the effects of sample collection technique on sample precision. A duplicate sample is a sample of a particular matrix, collected at the same time and in the same manner as the sample to which it corresponds. The duplicate is labeled as a separate sample and is analyzed by the laboratory as a separate sample. Subsequent to analytical reporting, the sample duplicate pair will be assessed for sample technique effects on precision by evaluating relative percent difference (RPD) between the sample result and the duplicate result for each analyte. The duplicate samples will be submitted to the laboratory for analysis of VOCs, SVOCs, and PAHs.

RPD is the absolute value of the difference between the sample and duplicate, divided by the average of the sample and duplicate results, times 100. RPD is used to evaluate precision when both the sample and duplicate for a given analyte are greater than five times the practical quantitation limit (PQL). For this project, the results will be considered acceptable if the RPD is less than 30 %. If the PQLs differ for the sample and duplicate for a given analyte, the higher RDL will be used for the evaluation. RPD calculation is sensitive to the magnitude of the results being evaluated; as such, results within five times the PQL will not be subject to the 30% RPD goal.



## 10.6 Matrix Spike/Matrix Spike Duplicates

Matrix Spike/ Matrix Spike Duplicate (MS/MSD) pairs are used for evaluation of matrix effects on sample precision. MS/MSD aliquots will be collected at the frequency described above. The MS/MSD will be spiked with known concentrations of analytes by the laboratory; MS/MSD controls are prescribed in the contract laboratory QAPP (attached). The MS/MSD aliquots are collected in order to insure adequate sample volume is available for assessment of matrix effects. In order to assess a meaningful evaluation of matrix effects, MS/MSD aliquots will neither be collected from background locations nor from "source area" sample locations. The MS/MSD will be submitted to the laboratory for analysis of VOCs, SVOCs, and PAHs.

# **11.0 FIELD VARIANCES**

As conditions in the field may vary, it may become necessary to implement minor modifications to sampling as presented in this plan. When appropriate, the QA Officer will be notified and a verbal approval will be obtained before implementing the changes. In addition, any modifications to the sampling plan will be reported to the USEPA for approval. Modifications to the approved plan will be documented in the sampling project report.

# **12.0 FIELD HEALTH AND SAFETY PROCEDURES**

In addition to this Plan, Terracon will provide a site specific HASP. The HASP will be prepared in accordance with the requirements set forth in the Occupational Safety and Health Administration Regulation 29 CFR 1910.120, where applicable, and applicable state, city, or local safety codes. The HASP will be reviewed and signed daily by all field personnel prior to field work indicating that they understand the plan and its requirements. Copies of the plan will be maintained on-site and made available to all personnel throughout the investigation activities. A need for special personal protective equipment (PPE) beyond standard Level D is not anticipated. However, should site conditions warrant, all onsite personnel will withdraw to a predesignated rally point per the site HASP. Further information regarding health and safety considerations is included in the HASP.



## APPENDIX A

Figure 1 – Former Chemical Plant (FCP) Location Figure 2 – Topographic Map Figure 3 – Containment Well and Monitoring Well Locations for the FCP

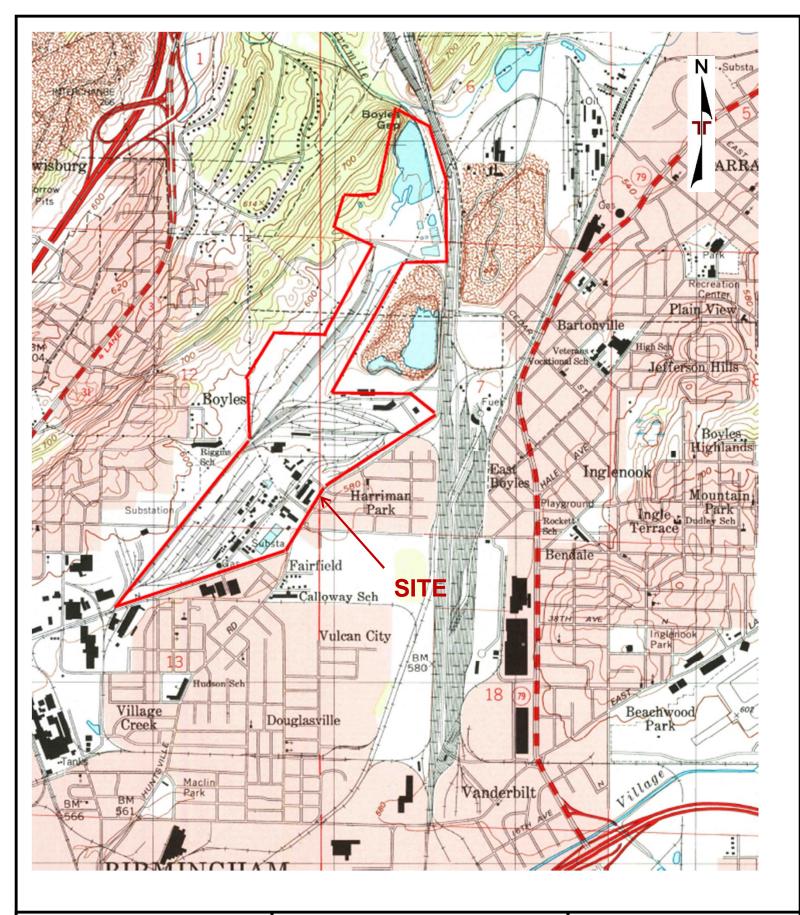


**Ferrac** <u>110 12<sup>TH</sup> STREET NORTH</u> P [205] 942-1289 BIRMINGHAM, AL 35203 F [205] 443-5302

IM WALTER COKE 3500 35<sup>TH</sup> AVENUE NORTH BIRMINGHAM, JEFFERSON COUNTY, ALABAMA **TERRACON PROJECT NO. E1127095** 

FORMER CHEMICAL PLANT LOCATION

Scale approximate 1"=420'





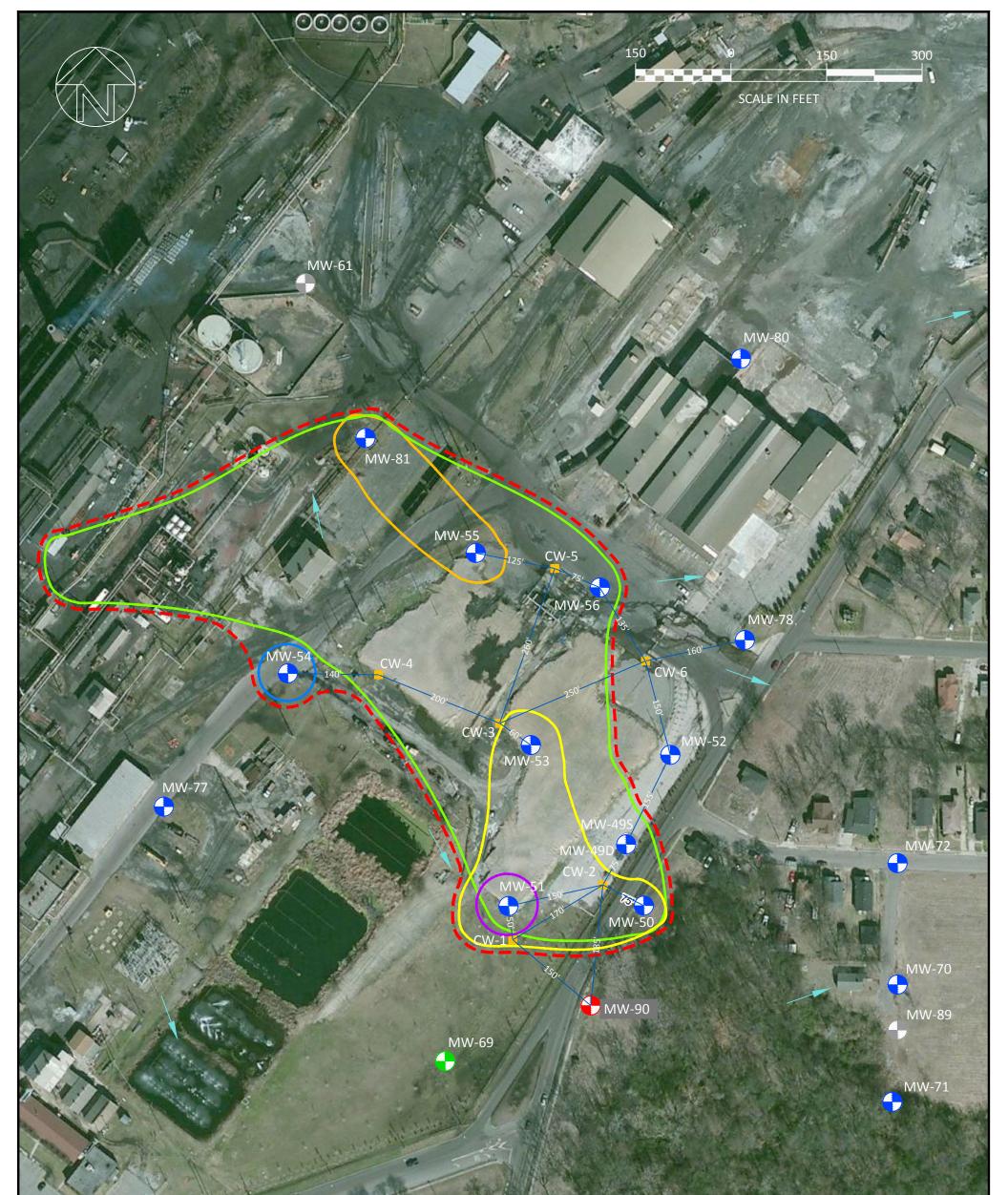
PROJECT

IM WALTER COKE 3500 35<sup>TH</sup> AVENUE NORTH BIRMINGHAM, JEFFERSON COUNTY, ALABAMA TERRACON PROJECT NO. E1127095

#### FIGURE 2

#### TOPOGRAPHIC MAP

North Birmingham, AL 7.5 Minute Quadrangle 1997 Scale 1"=2,000'



# LEGEND

SHALLOW BEDROCK MONITORING WELL BENZENE PLUME (EPA MCL - 5 μg/L) PROPOSED SHALLOW BEDROCK MONITORING WELL TOLUENE PLUME (EPA MCL - 100 µg/L) DEEP BEDROCK MONITORING WELL DCE PLUME (EPA MCL - 70 µg/L) MIXED MONITORING WELL PCE PLUME (EPA MCL - 5 µg/L) CONTAINMENT WELL LOCATIONS VC PLUME (EPA MCL - 2 µg/L) GENERAL DIRECTION OF GROUNDWATER FLOW EXTENT OF COMBINED CONTAMINANTS PLUME roject Mngr: roject No CONTAINMENT WELL & MONITORING WELL LOCATIONS FOR THE FCP TWR E1127096 llerracon )rawn By: Scale: INTERIM MEASURES AS SHOWN LJK 1) DISTANCES SHOWN BETWEEN WALTER COKE

ngham, AL 35203 205.443.5302

FIG. No.

3

3500 35th AVENUE NORTH

BIRMINGHAM, JEFFERSON COUNTY, ALABAMA

Last Saved By: LJKEARLEY Date: 10/9/2012 8:21 AM File Path: N:\PROJECTS\2012\E1127096\WORKING FILES\DIAGRAMS-DRAWINGS-FIGURES\DWG\_BHAM\FIGURE 3\_CW AND MW LOCATIONS FOR THE FCP.DWG

NOTES:

WELLS IS APPROXIMATE.

hecked By:

roved By

File No

10.09.2012

TWR

TWR



APPENDIX B

Terracon's Standard Operating Procedures (TSOP) Manual

	RENCE 10.	TITLE	LAST REVISED Or REVIEWED
E.10	0.	Project Mobilization	June 2010
E.20		Standard Safe Operating Procedures for Hazardous Waste Operations	June 2010
E.30		Chain of Custody Documentation	June 2010
E.50		Sampling – Environmental Representativeness	June 2010
E.100		Surface & Near Surface Soil Sampling – Grab	June 2010
E.150		Soil Sampling – Low Level Volatile By TerraCore™	June 2010
E.155		Soil Sampling – High Level Volatile By TerraCore™	June 2010
E.200		Surface Soil Sampling – Oakfield	June 2010
E.300		Sampling & Drilling Platforms	June 2010
L.000	E.310	Auger Drilling and Sampling	June 2010
	E.320	Hollow-stem Auger Drilling	June 2010
	E.325	Casing Advance Drilling	June 2010
	E.330	Fluid Rotary Drilling and Sampling	June 2010
	E.340	Air Rotary Drilling and Sampling	June 2010
E.400		Subsurface Sampling – Geoprobe© Platform	June 2010
E.410		Subsurface Sampling – General Push-Probe Technology	June 2010
E.450		Subsurface Soil Sampling – Xitech Sampler	June 2010
E.460		Subsurface Sampling – Shelby Tube	June 2010
E.465		Subsurface Sampling – Split Barrel	June 2010
E.468		Sample Handling – Šoil (Level D)	June 2010
E.470		Sample Handling – Groundwater (Non-Hazardous)	June 2010
E.480		Surface Water Sampling	June 2010
E.5XX		Field Screening	June 2010
	E.500	pH Field Screening – Soil	June 2010
	E.530	pH Field Screening – Water	June 2010
	E.540	Conductivity Field Screening – Water	June 2010
	E.550	Field Surface Screening – Soil / Photoionization Detector	June 2010
	E.552	Field Headspace Screening – Soil / Photoionization Detector	June 2010
	E.554	Field Screening – Air / Photoionization Detector	June 2010
	E.560	SVOC Field Screening – Soil /Ultraviolet	June 2010
	E.570	Temperature Field Screening	June 2010
	E.580	Turbidity Field Screening	June 2010
E.600	E.590	Airborne Lead and Particulate Matter Monitoring	June 2010 June 2010
E.605		H <sub>2</sub> S Field Screening – Field Detector Methane – Field Detector	June 2010
E.605 E.610		Radioactivity – Field Detector	June 2010
E.620		Polychlorinated Biphenyl Field Screening: Clor-N-Oil Field Detector	June 2010
E.623		Polychlorinated Biphenyl Field Screening: Clor-N-Soil Field Detector	June 2010
E.630		X-Ray Fluorescence (XRF) Screening – Airborne Dust	June 2010
E.634		X-Ray Fluorescence (XRF) Screening – Lead Paint	June 2010
E.638		X-Ray Fluorescence (XRF) Screening – Soil/Fills	June 2010
E.700		Well Construction – Temporary	June 2010
E.800		Well Construction – Permanent	June 2010
E.900		Well Security – Type A (Simple Cap)	June 2010
E.905		Well Security – Type B (Locking Expansion)	June 2010
E.910		Well Security – Type B (Protective Casing)	June 2010
E.920		Well Security – Type C (Flush Mount)	June 2010
E.1300		Well Development – Volumetric	June 2010
E.1400		Well Development – Parametric	June 2010
E.1500		Boring Abandonment – Commercial Sealant	June 2010
E.1600		Boring Abandonment – <i>Tremie</i> ' Grout	June 2010
E.1700		Well Abandonment – Iowa IAC39 Criteria	June 2010
E.18XX	E 4000	Physical Field Measurements	June 2010
	E.1800	Field Measurement – Surface Layout	June 2010
	E.1805	Field Measurement – Elevations	June 2010
<u> </u>	E.1808	Field Measurement – Licensed Survey	June 2010

1[erracon

REFERENCE NO.		TITLE	LAST REVISED Or REVIEWED
	E.1810	Field Measurement – Subsurface Soils	June 2010
	E.1820	Field Measurement – Groundwater	June 2010
	E.1830	Field Measurement - Free-Phase Product	June 2010
	E.1840	Field Measurement – Hydraulic Conductivity Testing (Slug)	June 2010
	E.1870	Field Measurement – Electromagnetic Survey	June 2010
E.1900		Groundwater Sampling – Bailer	June 2010
E.2000		Groundwater Sampling – Low Flow Pumping	June 2010
E.2100		Soil Vapor Sampling – Iowa IAC135	June 2010
E.22XX		Site Housekeeping	June 2010
	E.2210	General	June 2010
	E.2220	Disposal of Spent Supplies	June 2010
	E.2230	Handling and Storage of Drill Cuttings (Non-Hazardous)	June 2010
	E.2235	Handling and Storage of Drill Cuttings (Hazardous)	June 2010
	E.2240	Site Security Procedures	June 2010
E.24XX		Cleaning & Decontamination	June 2010
	E.2405	Cleaning - General	June 2010
	E.2410	Cleaning - Manual Washing	June 2010
	E.2420	Cleaning - High-Pressure, Hot-water Washing	June 2010
	E.3000	Bulk Sampling of Suspect Asbestos-Containing Material (ACM)	June 2010
	E.4000	Sampling of Potential Lead-Based Paint	June 2010

#### STANDARD OPERATING PROCEDURE

# E.10 PROJECT MOBILIZATION

#### Last Review or Revision: June 2010

#### OBJECTIVE

Allow field personnel an opportunity to review the requirements and objectives for performing the necessary field tasks and discuss with the project manager concerns associated with the project, safety, and methodologies.

#### PROCEDURE

- Field personnel should discuss the proposed field activities with the project manager <u>prior</u> to initiating the site work. This will be a formal "sit down, face-to-face" transfer of project information and objectives from the Project Manager to the designated field or task manager. This should include, but not be limited to, discussions on:
  - a) Project Objectives
  - Assure that personnel have an understanding of the objectives <u>prior</u> to initiating the field activities. This will provide a greater insight and allow field personnel to make appropriate decisions based on site specific conditions. This briefing provides background information which allows the field personnel to think about field operations in a way which will optimize performance and data gathering efficiency.
- Discuss the regulatory framework within which the report and results of field work will be used. Identify special requirements of specific regulatory programs.
  - a) Site Safety Plan (SSP)
  - Review the SSP with the project manager and sign the acknowledgement. Confirm that you have the proper monitoring equipment, protective clothing and respiratory device and confirm that you are aware of the monitoring requirements and safety level.
  - b) Permits/Right of Entry (R.O.E.)/Utility Clearance
  - Make sure that the proper permits, R.O.E. and utility clearance have been secured. This will avoid delays at the job-site. Determine that all parties, including the client, property owner or tenant, and regulatory official have been notified, if necessary.
  - c) Drilling/Development/Sampling Requirements
  - Review the drilling/development/sampling requirements to allow for satisfactory completion. Determine location of all borings/monitoring wells and the soil and groundwater sampling sequence for chemical analysis and definition of the stratigraphy.
  - d) Decontamination

Review cleaning procedures for the drilling equipment and/or sampling equipment. This is imperative to minimize cross-contamination and maintain safe site conditions.

e) Communications

Establish a communications link in the event that field personnel must consult with the project manager or designee in instances when the project manager cannot be reached.

f) Chain-of-Custody

Review the procedures to provide for proper Chain-of-Custody maintenance from sample collection through analysis.

g) Equipment

Confirm that the proper equipment is available, functional, cleaned, and calibrated. The project manager should provide a checklist of all equipment required, especially when the equipment needs to vary from the norm.

#### DOCUMENTATION

The project manager is responsible for providing the following documentation to the field crews prior to field activities:

- a) Site Safety Plan (completed)
- b) Mobilization Sheet
- c) Site Location Map
- d) Site Diagram
- e) Field manuals and references as appropriate

## E.20

## STANDARD SAFE OPERATING PROCEDURES FOR HAZARDOUS WASTE OPERATIONS

## Last Review or Revision: June 2010

#### I. PURPOSE

This document defines standard safe operating procedures for use on project sites where intrusive activities will be preformed and where soil/groundwater contaminants are known or are reasonably expected to exist. These procedures will be incorporated in the project safety and health plan which is mandatory for intrusive activities conducted at all such project sites.

## II. SAFETY AND HEALTH RESPONSIBILITY

A. The Project Manager is ultimately responsible for ensuring that work on environmental investigation/remediation projects are performed in accordance with provisions outlined in the project safety and health plan. A Site Safety and Health Officer (SSO) will be designated at each project site to monitor compliance with this safety-related practices during field activities. The SSO will ensure that a copy of the safety and health plan is available on site for the duration of project activities.

- B. The Corporate Safety and Health Manager will be consulted for each intrusive environmental investigation/remediation project. The Corporate Safety and Health Manager will develop the site safety and health plan, and will be available to consult with Project Manager/SSO in the event of questions, concerns or changed site conditions. The Corporate Safety and Health Manager will specify air monitoring and personal protective equipment requirements for the project, and will assist in obtaining specialized equipment required for the project.
- C. If hazardous conditions develop during the course of project activity, the SSO in conjunction with the Terracon Corporate Safety and Health Manager, will coordinate actions required to safeguard site personnel and members of the general public. Additional safety measures will be verbally communicated to all project personnel, recorded in writing and appended to the site safety and health plan.
  - D. Terracon and subcontractor task leaders will be responsible for:
  - Presenting the contents of the site safety and health plan to all subordinate site personnel.
  - Monitoring compliance with applicable provisions of the safety and health plan.
  - Periodically inspecting heavy equipment and other machinery and maintaining such equipment in compliance with applicable federal, state or local safety regulations.

• Enforcement of corrective actions.

#### III. MEDICAL SURVEILLANCE

A. All Terracon personnel participating in intrusive projects with known or reasonably anticipated contaminants must be enrolled in the Terracon Medical Surveillance Program. Each project participant will be certified by a licensed physician as fit for respirator and semi-permeable/impermeable protective equipment use. Medical clearance will be current to within one year of the project start date.

B. Certificates of medical examination for all project personnel will be maintained by the Corporate Safety and Health Manager and/or by the SSO in the project command center or support vehicle.

C. At the discretion of the Terracon consulting physician, an "exit" physical examination will be conducted at the completion of project activities or upon termination of a project participant. Follow-up medical examinations will also be provided in the event of job site injury or unprotected exposure to contaminants in excess of eight-hour time weighted average permissible exposure limits.

#### **IV. TRAINING REQUIREMENTS**

A. Terracon personnel participating in hazardous waste operations will have completed 40 hour Hazardous Waste Operations Training and at least three days supervised field activity. A current 8-hour annual refresher training certificate will be required for all personnel. Training certificates for all project personnel will be maintained by the Corporate Safety and Health Manager.

B. The SSO will conduct a pre-project safety and health briefing for all project participants. The personnel responsible for project safety and health will be addressed, as will site history, scope of work, site control measures, emergency procedures and site communications. The briefing will address site contaminants, air monitoring protocols, action levels for upgrade/downgrade of personal protective equipment and level of personal protective equipment to be employed for each project task.

C. Project participants will sign the Acknowledgment of Instruction form contained in each safety and health plan following the initial site briefing.

## V. CHEMICAL HAZARDS

A. The Project Manager is responsible for obtaining available information regarding site contaminants, including analytical information obtained from samples previously collected at the project site, and for forwarding the information to the Corporate Safety and Health Manager for research and hazard analysis. A site specific safety and health plan will be developed notifying personnel of the potential chemical contaminants, their health hazards, routes of entry, warning properties and symptoms of exposure.

B. Evaluation of chemical contaminants/concentrations will be used in the development of the air monitoring and personal protective equipment requirements contained in the site safety and health plan.

## VI. PHYSICAL HAZARDS

## A. Drilling Projects

- All personnel working around drill rigs will be familiarized with emergency shut-down procedures and the position of "kill" switches.
- No loose fitting clothing, jewelry or unsecured long hair is permitted near the rig.
- Keep hands and feet away from all moving parts while drilling is in progress. Shovel auger cuttings with long handled shovel. *DO NOT* use hands or feet.
- Daily inspection of all ropes, cables and moving parts is mandatory.
- A first aid kit and fire extinguisher will be immediately available at all times.
- All drill crews shall consist of at least two persons.
- No drilling is permitted during impending electrical storms, tornadoes or when rain creates a hazardous work environment.
- A minimum horizontal and vertical clearance distance of **10 feet** must be maintained between the drill rig and overhead power lines; use spotters to help rig operator position the vehicle when near overhead power lines.

#### **B. Excavation Project Sites**

- Wherever possible, soil samples will be collected from backhoe buckets. Personnel will enter excavations only as a final option and only in accordance with the regulatory requirements outlined above.
- AT NO TIME WILL TERRACON PERSONNEL ENTER EXCAVATIONS TO COLLECT SOIL SAMPLES UNTIL A PROPER MEANS OF EGRESS/EXIT IS PROVIDED AND THE EXCAVATION HAS BEEN INSPECTED BY A COMPETENT PERSON AND APPROPRIATELY SHORED OR SLOPED IN ACCORDANCE WITH THE OSHA EXCAVATIONS STANDARD (29 CFR 1926, SUBPART P).
- Personnel will remain at least 3 feet from the sides of excavations during sample collection and excavation observation.
- Personnel will remain outside the swing radius of backhoe buckets during excavation, and will stand behind the backhoe or within line-of-sight contact with the backhoe operator at all times.

#### VII. ACCIDENT PREVENTION

- The Site Safety Officer will hold daily safety briefings at the beginning of each day of site activity.
- Traffic control measures will be arranged for all projects conducted within or immediately adjacent to active roadways. Signage, warning and/or channelizing devices will conform to the Manual on Uniform Traffic Control Devices. Flagging operations will be conducted only by personnel who have received training in proper traffic flagging procedures. The preferred method of traffic control will be to contract these services to a reputable traffic control service knowledgable in local traffic control regulations.
- Safety orange work vests will be worn by personnel working within 10 feet of any active roadway.
- The Site Safety Officer will ensure that unauthorized personnel do not enter the work zone. Authorized visitors will be briefed on site contaminants, personal protective equipment requirements and the decontamination provisions of the site safety and health plan.
- The Site Safety Officer will continually inspect the work area for infractions of safety and health requirements as contained in the site specific safety and health plan.
- The Site Safety Officer will investigate and immediately report all accidents to the Corporate Safety and Health Manager.
- Site activities will be conducted only during daylight hours unless adequate portable lighting • is mobilized to the project site.
- The "buddy system" will be observed at all times during intrusive site investigations. A minimum of two people will work together and remain within eye sight or not greater than 100 ft. apart.
- Teamwork and the use of mechanical lifting devices will be employed where practical to ease lifting tasks and reduce the potential for muculoskeletal injury.

#### VIII. SITE CONTROL

A. An Exclusion Zone, Contaminant Reduction Zone and a Support Zone will be established on hazardous waste operations sites requiring Level C or Level Bpersonal protective equipment. Defined access and egress points will be

established and personnel will enter only through those points.

B. As permitted by site topography, the area within a 50 foot radius of a drill rig and 100 foot radius of UST removal excavations will be considered the Exclusion Zone. Only those personnel designated by the Project Manager/SSO are allowed to enter the Exclusion Zone. Where practical, or where their use will prevent public injury, temporary signs or barricade fencing will be established to define the Exclusion Zone. ABSOLUTELY NO SMOKING WILL BE PERMITTED WITHIN THE EXCLUSION OR CONTAMINANT REDUCTION ZONES ON ANY PETROLEUM CONTAMINATED SITE.

#### **TSOP E.205**

C. If unauthorized personnel attempt to enter the exclusion zone, the SSO will verbally inform the individual(s) to leave the project site. If unauthorized individuals refuse to leave the Exclusion Zone or are considered in danger or pose danger to project personnel, the SSO will cease project activities (i.e., shut down drill rigs, excavation equipment, etc.) and notify the client representative or the local police of the situation.

## IX. AIR MONITORING

A. Air monitoring protocols will be designed to prevent personnel exposure to airborne contaminants in excess of established permissible exposure limits. The results of field air monitoring will be used to determine the continued adequacy of initial personal protective equipment.

B. Task Leader(s) will be knowledgeable in the operation and troubleshooting of air monitoring equipment. A manual on the operation of each air monitoring instrument and an appropriate calibration kit will be mobilized to the project site with the instrument. Air monitoring instruments will be calibrated under field conditions each day prior to use. Task Leaders are instructed to consult the operator's manual for appropriate calibration gas and calibration techniques.

## X. PERSONAL PROTECTIVE EQUIPMENT

Personal protective equipment requirements for each hazardous waste operations site and task will be specified in the site safety and health plan. Personal protective equipment selection will be based upon the site contaminants and tasks to be performed. Personal protective equipment ensembles will be selected in general accordance with standard EPA levels of protection as follows:

A. **Level A** - To be selected when the greatest level of skin, respiratory, and eye protection is required. Level A personal protective equipment ensembles will consist of:

1. Positive pressure, full face-piece self-contained breathing apparatus (SCBA), or positive pressure supplied air respirator with escape SCBA, approved NIOSH.

- 2. Totally-encapsulating chemical-protective suit.
- 3. Coveralls.(optional, as appropriate)
- 4. Long underwear.(optional, as appropriate)
- 5. Gloves, outer, chemical-resistant.
- 6. Gloves, inner, chemical-resistant.
- 7. Boots, chemical-resistant, steel toe and shank.
- 8. Hard hat (under suit).(1)
- 9. Disposable protective suit, gloves and boots

B. Level B - To be selected when the highest level of respiratory protection is necessary but a lesser level of skin protection is required. Level B personal protective equipment ensembles will consist of:

1. Positive pressure, full-facepiece self-contained breathing apparatus (SCBA), or positive pressure supplied air respirator with escape SCBA (NIOSH approved).

2. Hooded chemical-resistant clothing (overalls and long-sleeved jacket; coveralls; one or two-piece chemical-splash suit; disposable chemical-resistant overalls).

- 3. Coveralls (optional, as appropriate)
- 4. Gloves, outer, chemical-resistant.
- 5. Gloves, inner, chemical-resistant.
- 6. Boots, outer, chemical-resistant steel toe and shank.
- 7. Hard hat.

C. Level C - To be specified when the identity and approximate concentration of airborne contaminants is known and the criteria for using air purifying respirators are met. Level C personal protective equipment ensembles shall consist of the following:

1. Full-face (typically) air purifying respirators (NIOSH approved).

2. Chemical-resistant clothing (coveralls; two-piece chemical-splash suit; disposable chemical- resistant overalls, as appropriate).

- 3. Gloves, outer, chemical-resistant.
- 4. Gloves, inner, chemical-resistant.
- 5. Boots (outer), chemical-resistant steel toe and shank.
- 6. Disposable boot-covers, outer, chemical-resistant (optional)
- 7. Hard hat.(1)

D. **Level D** - A work uniform affording minimal protection. Level D or modified Level D personal protective equipment will be used when contaminant concentrations are low and not readily absorbable through the skin and where atmospheric monitoring indicates no need for respiratory protection. The following constitute Level D equipment; it may be used as appropriate:

- 1. Coveralls.
- 2. Chemical-resistant gloves.(style selected based on contaminants)
- 3. Boots/shoes, chemical-resistant steel toe and shank.
- 4. Boots, outer, chemical-resistant (disposable).
- 5. Safety glasses or chemical splash goggles (where appropriate).
- 6. Hard hat.

## XI. DECONTAMINATION

A. Equipment decontamination is necessary on all contaminated project sites. Personnel decontamination for projects below personal protective Level C will consist of washing off safety footwear, proper cleaning or disposal of outer and inner gloves and thorough washing of face, arms and hands. A full body shower will be required as soon as possible upon leaving the project site.

B. For projects involving personal protective equipment at Level C or above, a decontamination station will be established on the interface of the Exclusion Zone. A Contaminant Reduction Zone will be established and will extend 10 feet beyond the decontamination station.

- Two Wash Tubs
- Scrub Brush
- Plastic Bags
- Water and Alconox Detergent

The wash tub on the exclusion zone side of the site will contain a solution of and Alconox detergent; the second wash tub will contain clean rinse water. Personnel decontamination will consist primarily of detergent washing and rinsing of reusable exterior protective gear. Coveralls will be removed by turning the clothing inside out.

Personnel may not leave the contaminant reduction zone without proceeding through the decontamination sequence described below.

- Wash work gloves, boots and polylaminated protective coveralls,
- Rinse work gloves, boots and coveralls,
- Remove tape at wrists and ankles,
- Remove protective coveralls,
- Remove respirator
- Dispose of spent cartridges; wash and rinse respirator
- Remove outer gloves
- Remove inner gloves

Expendable personal protective equipment will be placed in plastic trash bags, sealed and disposed of per client agreement. Decontamination solutions will be containerized or disposed of as arranged by Project Manager.

C. Decontamination of equipment will be performed to limit the migration of contaminants off-site. All equipment will be cleaned prior to site entry to remove grease, oil and encrusted soil. Decontamination of large equipment will consist of physically removing gross contamination with shovels, brushes etc. followed by detergent and water high pressure wash with a clean water rinse. The Project Manager is responsible for determining if decontamination solutions must be containerized. If so, a decontamination sump or polyethylene sheeting and fluid containers will be mobilized and established in the decontamination area. Decontamination of hand samplers and similar small equipment will be performed at a designated location within the Contaminant Reduction Zone. Decontamination of such equipment will consist of detergent solution wash and clean water rinse. Specialized decontamination solutions (acids, solvents, biocidal, etc.) may be specified in site specific site safety and health plans.

## XII. SITE COMMUNICATIONS

Communication between personnel on most hazardous waste project sites will be via verbal communication or hand signals. Visual contact between members of task teams should be possible throughout the course of project activities. Contact with the SSO will be through direct verbal communication. The following hand signals will be used whenever verbal communication is limited:

<u>Signal</u> Thumbs Up Grab throat with both hands Shake head, thumbs down Point right (when facing equipment operator) Point left when facing equipment operator) Grab partner's wrist Meaning OK, all is well Can't breathe NO, negative Move/steer left Move/steer right Leave area immediately

## XIII. EMERGENCY RESPONSE PROCEDURES

A. The Project Manager is responsible for obtaining and recording emergency telephone contacts in the appropriate section of the site safety and health plan to site mobilization: A mobile telephone will typically be available on hazardous waste operations project sites.

prior

B. In the case of personal injury, appropriately trained personnel will be requested to provide first aid and emergency rescue. For minor injuries, such as cuts, burns, exhaustion, heat cramps, insect stings, etc., the affected employee will be removed to an uncontaminated area. The SSO or other designated employee trained in first aid procedures will administer appropriate first aid. If the injury requires additional medical attention, the injured employee will be cleaned and transported to the nearest hospital or emergency medical facility.

C. For more serious injuries the SSO or designee will summon an ambulance to the project site. No attempt will be made by Terracon personnel to move the victim, without the aid and/or instructions of qualified medical personnel.

D. If the victim cannot be safely moved without a stretcher or other specialized equipment, the victim will be removed at the earliest possible moment by appropriately attired Terracon personnel with the direction and/or assistance of qualified medical response personnel. The injured employee will be immediately decontaminated and transported to the nearest medical facility. A crew member designated by the SSO will inform the ambulance crew of contaminants of concern and provide assistance with additional decontamination if required.

## **XIV. EVACUATION AND SHUTDOWN PROCEDURES**

- A. On project sites posing a significant risk of chemical or physical hazard exposure, the site safety and health plan will instruct the SSO to establish and notify site personnel of emergency "rally" points. In the event of a site emergency, personnel will immediately exit the site and assemble at the designated rally point. Evacuation routes will be dependent on site topography and wind conditions. The routes will be selected and presented by the SSO daily prior to site activity.
- B. If emergency evacuation becomes necessary, the SSO will sound the emergency alarm (e.g. support vehicle horn or compressed air horn). Personnel will safely shutdown all electrical and mechanical equipment and quickly proceed to closest designated rally point. The SSO will then account for each crew member on site.

C. In the event that a Terracon employee does not report to the designated rally point within 5 minutes of the evacuation alarm, the SSO will perform an immediate assessment of site conditions. If site conditions do not pose an immediate hazard to life or health, the SSO will initiate search and rescue efforts utilizing two crew members attired in appropriate personal protective equipment.

## E.30

## CHAIN OF CUSTODY DOCUMENTATION

Last Revision: June 2010

## **OBJECTIVE AND APPLICATION**

This document defines standard operating procedures for documenting sample collection using proper chain-of-custody techniques. The purpose of proper chain-of-custody techniques is to provide accountability for and documentation of sample integrity from the time samples are collected until sample disposal.

This procedure is intended to document sample possession during each stage of a sample's life cycle, that is, during collection, shipment, storage, and the process of analysis.

## EQUIPMENT

- Terracon chain-of-custody record(s) or laboratory-specific chain-of-custody forms (typically supplied with sample containers),
- If samples are being shipped via courier, custody seals for coolers,
- Indelible ink marker, and
- Zip top bag.

## PROCEDURE

Sample containers will be labeled in advance of sampling with the sample date, location (well identifier), sampler's initials, and project name. Written sample custody procedures will be followed whenever samples are collected, transferred, stored, analyzed, or destroyed, in order to trace possession and handling of a sample from collection to disposal. Accountability for a sample begins when the sample is collected. Each sample will be accounted for with the use of sample labels, chain-of-custody forms, a record of sample collection, and field data notebooks.

The following chain-of-custody procedures will be implemented by the field staff:

- Entries in the field notebook and chain-of-custody form will be made in ink. Documentation of each sample must be completed at the time of sampling.
- The chain-of-custody should include at a minimum:
  - Project name and/or number
  - Name and contact information for the sampler collector
  - Collector's signature
  - Sample designation
  - Date sampled
  - Time sampled

- Sample media
- Number and size of containers for each sample
- Types of sample preservatives used
- Analyses requested
- The original chain-of-custody must accompany the samples at all times after collection, until receipt at the analytical laboratory. A copy of the chain-of-custody form will be kept by the field staff until filing at the office.
- The original chain-of-custody form should be sealed in a Ziplock bag if shipping samples on ice via courier. The sealed Ziplock bag will protect the document from moisture that may be present due to sample preservation. The chain-of-custody should be the last item packed in a sample cooler, so that it is easily accessible if the cooler is misplaced by the courier or shipped to an incorrect address.
- If shipping samples, a chain-of-custody specific to the contents of each cooler will be
  packaged with the respective samples. Chain-of-custody forms should not be shipped in
  separate containers than the samples they document. At least one custody seal should be
  completed by the collector and applied to each cooler sent to the laboratory. The custody
  seal should be affixed to the cooler in such a manner as to ensure breakage of the seal
  upon opening of the cooler (e.g., across the cooler lid opening).
- When the possession of samples is transferred, the individuals relinquishing and receiving the samples will sign, date, and note the time on the chain-of-custody form.
- If samples are shipped, strict chain-of-custody is violated. However, at the discretion of the project manager the procedures can still be followed.

## ATTACHED REFERENCES

Terracon Form COC-7/92 Chain-of-Custody Record, revised 4/93

Quality Environmental Containers Custody Seal

## OTHER SUPPORTING DOCUMENTS

ASTM D4840-99 Standard Guide for Sampling Chain-of-Custody Procedures

# E.50 Sampling – Environmental Representativeness

## LAST REVIEW OR REVISION: June 2010

## **OBJECTIVE AND INTENT:**

The information value of data depends heavily upon the interaction among sampling and analytical designs in relation to the intended use of the data, the site-specific context surrounding that intended use, and the associated quality control. The environmental condition of the site will be determined by the chemical data from samples collected in the field.

## A Quick Look - USEPA CLU-IN 2001

Data quality is the function of the data's information content and its ability to represent the true state of a site.

Data representativeness is the measure of the degree to which samples can be used to estimate the characteristics of the true state of a hazardous waste site.

Brownfields are considered an application in which data quality and representativness will play an important role.

The term "representative data" means that there is some stability in the samples and assurance of a reasonable data density for the site being sampled. "Reasonable" data density varies depending on the intended use of the information. Project decisions which are very general in nature or used only for preliminary decision-making may have a very limited data density to represent the site, but the decisions which can be made from the data are also limited. Project decisions made from data for enforcement or litigation are much more concerned with sampling density and how well the information represents the site.

The procedures, handling and documentation by Terracon staff should routinely and consistently be as uniform as is practicable so that any sample from any media best represents the environmental condition.

This procedure is provided as supporting guidance and direction to Terracon field and design staff to provide quality samples representative of the intended project decision.

## APPLICATION

Sampling is the selection of a representative portion of a larger population, universe or body. Through examination of a sample, the characteristics of the larger body can be inferred. The characteristics to be inferred will directly affect the method and procedure to select a representative sample.

Technical information derived from soil and groundwater samples differ greatly for purposes of geotechnical and environmental engineering, although the physical procedures of sample collection or measurement are often the same. The proper application of any one or more physical procedures to collect the sample will be dependent on the characteristic condition the sample is intended to represent. Environmental and geotechnical characteristics can often be determined

#### TSOPE.50

from the same sample, other times they cannot. Combined use must be carefully considered by the Terracon Project Manager before application.

In general, any representative environmental sample is intended to reflect the in-situ, or undisturbed, chemically-impacted condition measured relative to the project decision and must consider;

- The media of the sample and it's physical properties
- The contaminant of concern and it's physical properties
- The contaminant of concern and it's chemical properties
- The spatial boundaries to be represented by the sample

#### EQUIPMENT

Equipment will be as specified by the Project Manager, specific to the requisite Terracon Standard Operating Procedure (TSOP).

#### PROCEDURES

Procedures will be as specified by the Project Manager, specific to the requisite Terracon Standard Operating Procedure (TSOP).

However, Terracon personnel shall be cognizant of and maintain the following general "rules of thumb" when reviewing TSOPs provided by Managers and when field procedures raise changed conditions which require communication of the new conditions back to the Project Manager;

- Volatile chemicals of concern dictate the least amount of disturbance and handling to preserve the representative characteristic; the lower the concentration of concern in the sampled body the more important the issue of disturbance becomes.
- Non-volatile chemicals of concern dictate a greater amount of disturbance and handling to
  preserve the representative characteristic; this may allow more extensive methods to physically
  select a representative sample and may allow the construction of composite samples in some
  regulatory programs.
- A decreasing sensitivity to disturbance is generally associated with sampled media as follows;
  - Water
  - Granular soils or other materials
  - Bedrock or other consolidated materials
  - Clay soils or other materials with high clay or organic contents
- Samples taken by mechanical samplers should represent both the vertical and horizontal spatial boundaries of the media collected.

 Samples taken by mechanical samplers should represent the media that is least influenced by the sampling method. For example, in a split spoon sampler the interior of the sample recovered is more representative than the exterior edge which contacted and is disturbed by the steel split spoon shell during penetration.

#### DOCUMENTATION

Documentation will be as specified by the Project Manager, specific to the requisite TSOP.

#### ATTACHED SUPPORTING DOCUMENTS

SOP 2001, Rev 0.0 EPA ERT GENERAL FIELD SAMPLING GUIDELINES

## **OTHER SUPPORTING DOCUMENTS**

TSOP E.30	Chain-of-Custody Documentation
TSOP E.100	Surface & Near Surface Soil Sampling – Grab
TSOP E.100 Series	Surface/Near Surface Sampling Terracon Procedures
TSOP E.300 Series	Drilling Terracon Procedures
TSOP E.400 Series	Subsurface Sampling Terracon Procedures

#### STANDARD OPERATING PROCEDURE

# E.100 SURFACE & NEAR-SURFACE SOIL SAMPLING - GRAB

## LAST REVIEW OR REVISION: June 2010

## **OBJECTIVE AND APPLICATION**

To provide standard procedure for collecting surface and near-surface soil samples appropriate to project conditions.

Surface soil/tailings/spoils samples are collected from the surface to a depth of approximately four (~4) inches. Near-surface soil/tailings/spoils samples are collected from the surface to a depth of approximately four to twelve (~4-12) inches from original surface. The original surface may be ground surface or the exposed horizontal or vertical surface of material excavated in mass from the subsurface.

Grab sampling is appropriate to conditions and projects where the end use of the sample is not overly sensitive to disturbance and handling in the course of collection. Grab sampling should not be applied where the sample is used for field or laboratory measurements of low levels of readily volatile organic compounds. Grab sampling is appropriate for general field screening and as a field guide to directing environmental excavation, if compatible with the physical properties of the chemical(s) to be measured. Grab sampling should not be used for samples intended to represent in-situ, undisturbed subsurface conditions.

Sufficient sample will be collected for the analysis that will be performed as prescribed by the project documents. Soil descriptions will be completed for each collected soil sample using the general terminology of the unified soil classification system (ASTM D2487). Descriptions shall be recorded in field books.

#### EQUIPMENT

- Rigid sampling equipment such as a trowel or shovel of inert material relative to the chemical(s) of concern and capable of reaching the depths prescribed for surface and near-surface soils.
- Disposable gloves.
- Chemical-resistant work gloves.
- Laboratory prepared sample containers.
- Roll of plastic sheeting.
- Plastic trash bag for collecting expended supplies.
- Field documentation forms or project logbook.

- Chain-of-Custody forms for samples intended for laboratory analysis.
- Marking pencils or indelible markers that will not leave residues which can cause interference with laboratory testing procedures.

## PROCEDURES

Select the location. Identify it with a unique designation for the project. Diagram or otherwise describe the location on forms or in the logbook relative to a fixed benchmark that will allow the specific location to be re-visited in the future, if necessary. If appropriate, estimate the vertical elevation of the sample and record.

If the grab sample is constructed of multiple aliquots to represent averaging of conditions in soils/tailings/spoils, diagram or otherwise describe the area represented by the constructed sample. Diagram or otherwise accurately describe sub-sample locations on forms or in the logbook relative to a fixed benchmark that will allow the specific sub-sample locations to be revisited in the future, if necessary. If appropriate, estimate the vertical elevation of the sub-samples and record.

At each location before collecting the soil sample, put on a clean pair of disposable chemicalresistant gloves.

Collect each sample by hand using procedures and equipment/tools specified by the project manager.

Place the sample directly into the laboratory prepared sample container(s) and complete the sample label and Chain-of-Custody as instructed by the project manager.

If intended for laboratory analysis, preserve the sample as required by project plans.

## ATTACHED REFERENCES

• **ASTM E1903-97** Standard Guide for Environmental Site Assessments: Phase II Environmental Site Assessment Process.

## **OTHER SUPPORTING DOCUMENTS**

• **ASTM D2487-00** Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System).

#### STANDARD OPERATING PROCEDURE

# E.150 SOIL SAMPLING Low-Level Volatile / Terra Core™

#### Last Review or Revision: June 2010

#### **Objective and Application:**

To provide standard procedure for sample collection in conditions in which low concentrations of volatile organic compounds are anticipated in soils which minimizes handling and volatilization from the samples. The methods will provide representative samples for laboratory analysis using EPA Standard Method SW-846 5035.

This application uses proprietary commercial equipment and materials which will be purchased from an authorized vendor.

## Equipment:

- Commercial Low-Level Terra Core™ Kits, each to include:
  - 8 disposable Terra Core<sup>™</sup> Samplers
  - 16 tared 40-milliliter (ml) Volatile Organic Analysis (VOA) vials containing 5 mls of sodium bisulfate solution and integral stir bars
  - 8 tared 40-ml VOA vials containing methanol
  - 8 two ounce dry weight jars with lids
  - 8 zippered plastic bags
- Disposable chemical resistant gloves.
- Chain of Custody.
- Stable field platform to support test kit, screened from wind and elements.

#### **Procedures:**

Set up the working platform on a stable surface or in the field vehicle remote from fuel, exhaust or other contaminant sources. Cover the platform and secure with disposable plastic sheeting.

At each location prior to collecting the soil sample, put on a clean pair of disposable chemical resistant gloves.

Have ready a tared 40ml glass VOA vial containing the appropriate preservative. With the plunger seated in the handle, push the Terra Core<sup>™</sup> into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 grams of soil.



Wipe all soil or debris from the outside of the Terra Core<sup>™</sup> sampler. Immediately cap the end of the plunger with clean cap provided. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.

Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the tared 40ml VOA vial containing the appropriate preservative, and extrude the sample by pushing the plunger down. Quickly place the lid back on the 40ml VOA vial. When capping the 40ml VOA vial, be sure to remove any soil or debris from the threads of the vial.

Label with a non-solvent-based permanent marker. Place in the zippered bags provided.

Pack well in the kit box provided by the manufacturer. Place the kit box in a large capacity Ziploc® freezer bag and seal. Avoid use of tape and a plastic bag as this method deals specifically with low-level volatile testing.



Place in the cooler filled with ice and pack suitable for overnight courier shipping.

#### **Other Supporting Documents :**

- TerraCore<sup>™</sup> Internet Website at http://www.ennovativetech.com/TC\_sampler\_kit.htm En Novative Technologies, Inc. Phone:(920)465-3960
   Fax: (920)465-3963
   Toll Free: 888-411-0757
   Procedures by En Novative Technologies, Inc. 1999-2000, Modified: March 13, 2001
- Alternative EPA Method 5035 Sampling as EnCore
- USEPA SW-846 Method 5035 For Organic Analytes Using Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
   Test methods for Evaluating Solid Waste – Volume I, EPA Document #SW-846, United States Environmental Protection Agency, Office of Solid Waste and Emergency Response, Section B, Chapter Four.

#### Terracon

# E.155 SOIL SAMPLING High-Level Volatile / Terra Core™

#### Last Review or Revision: June 2010

**Objective:** To provide standard procedure for sample collection in conditions in which higher concentrations of volatile organic compounds are anticipated in soils but for which maximum reduction in volatilization during sampling is required. This special procedure minimizes handling and volatilization from the samples in the field and in the laboratory. This procedure will provide representative samples of soil for laboratory analysis using EPA Standard Method SW-846 5035.

**Application:** The procedure applies to fine grained soils, fills and sands, but may have limited applicability in soils/fills with gravel or debris greater than 1/4-inch in diameter. This application uses proprietary commercial equipment and materials which will be purchased from an authorized vendor.

# Equipment:

- Commercial High-Level Terra Core™ Kits, each to include:
  - 8 Disposable Terra Core Samplers
  - 8 tared 40-ml VOA vials containing methanol
  - 8 two ounce dry weight jars with lids
  - 8 zipper bags
- Additional Items If Needed: Deionized Water Vials for use in case of effervescence as described in low level sampling in EPA Method 5035.
  - 16 Tared, 40-ml vials containing 5mls of deionized water and stir bar.
- Disposable chemical-resistant gloves.
- Chain of Custody.
- Stable field platform to support test kit, screened from wind and elements.

#### **Procedures:**

Set up the working platform on a stable surface or in the field vehicle remote from fuel, exhaust or other contaminant sources. Cover the platform and secure with disposable plastic sheeting.

At each location prior to collecting the soil sample, put on a clean pair of disposable chemicalresistant gloves. Check the field kit for presence of all components listed on the box.

#### TSOP Page E.155.2

Terracon



Have ready a tared 40-ml glass VOA vial containing the appropriate preservative. With the plunger seated in the handle, push the Terra Core<sup>™</sup> into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 grams of soil.

Wipe all soil or debris from the outside of the Terra Core<sup>™</sup> sampler. Immediately cap the end of the plunger with clean cap provided. The soil

plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.

Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the 40ml VOA vial containing the appropriate preservative, and extrude the sample by pushing the plunger down. Quickly place the lid back on the 40ml VOA vial. When capping the 40ml VOA vial, be sure to remove any soil or debris from the threads of the vial.

Label with a non-solvent-based permanent marker. Place in the zippered bags provided.

Pack well in the kit box provided by the manufacturer. Place the kit box in a large capacity Ziploc® freezer bag and seal. Avoid use of tape and a plastic bag to avoid potential cross-contamination.

Place in the cooler filled with ice and pack suitable for overnight courier shipping.

#### **Other Supporting Documents :**

- TerraCore™ Internet Website at http://www.ennovativetech.com/TC\_sampler\_kit.htm En Novative Technologies, Inc.
   Phone:(920)465-3960
   Fax: (920)465-3963
   Toll Free: 888-411-0757
   Procedures by En Novative Technologies, Inc. 1999-2000, Modified: March 13, 2001
- Alternative EPA Method 5035 Sampling as EnCore
- USEPA SW-846 Method 5035 For Organic Analytes Using Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples Test methods for Evaluating Solid Waste – Volume I, EPA Document #SW-846, United States Environmental Protection Agency, Office of Solid Waste and Emergency Response, Section B, Chapter Four.





# E.200 SURFACE SOIL SAMPLING - OAKFIELD

# LAST REVIEW OR REVISION: June 2010

# **OBJECTIVE AND APPLICATION**

To provide standard procedure for collecting surface and near-surface soil samples appropriate to project conditions using an Oakfield Sampler.

Surface and near-surface soil/tailings/spoils samples are collected from the surface to a depth of approximately eighteen (~18) inches. The original surface may be ground surface or the exposed horizontal or vertical surface of material excavated in mass from the subsurface.

Sampling using the Oakfield apparatus is appropriate to conditions and projects where the end use of the sample is sensitive to disturbance and handling in the course of collection. The sampler also allows the user to observe in-situ lithology over the interval sampled. The Oakfield sampler can be applied where the sample is used for field or laboratory measurements of low levels of readily volatile organic compounds.

Sufficient sample will be collected for the analysis that will be performed as prescribed by the project documents. Soil descriptions will be completed for each collected soil sample using the general terminology of the unified soil classification system (ASTM D2487). Descriptions shall be recorded in field books.

# EQUIPMENT

- Oakfield sampler.
- Disposable chemical-resistant gloves.
- Work gloves, as appropriate.
- Laboratory prepared sample containers.
- Roll of plastic sheeting.
- Plastic trash bag for collecting expended supplies.
- Field documentation forms or project logbook.
- Chain-of-Custody forms for samples intended for laboratory analysis.
- Marking pencils or indelible markers that will not leave residues which can cause interference with laboratory testing procedures.



# PROCEDURES

Select the location. Identify it with a unique designation for the project. Diagram or otherwise describe the location on forms or in the logbook relative to a fixed benchmark that will allow the specific location to be re-visited in the future, if necessary. If appropriate, estimate the vertical elevation of the sample and record.

If the sample is constructed of multiple aliquots to represent averaging of conditions in soils/tailings/spoils, diagram or otherwise describe the area represented by the constructed sample. Diagram or otherwise accurately describe sub-sample locations on forms or in the logbook relative to a fixed benchmark that will allow the specific sub-sample locations to be revisited in the future, if necessary. If appropriate, estimate the vertical elevation of the sub-samples and record.

At each location before collecting the soil sample, clean the sampling equipment using the appropriate procedure as required by the project. Use a clean pair of disposable gloves before handling soil samples.

Collect each sample by manually pushing the Oakfield sampler into the material to be sampled. Push the sampler to the desired sampling depth, or to the full extent of the sampling equipment (18 inches). Retrieve the sampler from the ground. The sample can be collected by hand from the opening on the side of the Oakfield sampler.

Place the sample directly into the laboratory prepared sample container(s) and complete the sample label and Chain-of-Custody as instructed by the project manager.

If intended for laboratory analysis, preserve the sample as required by project plans.

# E.300 SAMPLING & DRILLING PLATFORMS

#### Last Review or Revision: June 2010

## **Objective and Application**

To provide standard procedure for the mechanical and working support of soil sampling collection by providing equipment platforms for the advancement of borings using traditional drilling equipment and techniques of the current industry. This procedure is applicable to drilling and sampling unconsolidated soils and highly weathered bedrock.

## Equipment

The platform to carry the self-powered drill may be either truck-mounted or adapted to an allterrain vehicle capable of leveling the platform for perpendicular alignment of the drill unit. The drill rig to be used for the field exploration employs a hydraulic head for drilling and sampling.

Drilling equipment may be solid stem augers, commercially manufactured, ranging in flight diameter from 6 to 8 inches. Commercially manufactured friction safety clips will be used to connect drill sections.

Drilling equipment may be three and one-quarter (3.25)-inch or four and one-quarter (4.25) nominal inside diameter hollow stem augers. Nominal outside diameter of flights will be 8 to 10 inches, depending on manufacturer's construction. Commercially manufactured connecting bolts will be used to connect drill sections.

Supporting tools and equipment capable of implementing standard sampling methods for soil and weathered bedrock.

# Training

The drill operator and drill helper will have entered and be current in the following;

- Industry drilling experience no less than 3 years for operator, 1 year for helper.
- Enrolled in Terracon medical monitoring program for environmental operations
- Completed 40-Hour OSHA 1910.120 HAZWOPER training, with respiratory protection.
- Current certificate of annual 8-Hour OSHA 1910.120 HAZWOPER Refresher Training
- Knowledge and experience in drilling both vadose and saturated soils.
- Knowledge and experience in the operation and maintenance of the specified drill platform.

#### **TSOP E.300**

# Procedures

The proper platform will be selected using as guidance ASTM D4700. Consistent with Section 1.7 of the standard, the guidance is not intended to replace education or experience and will be used in conjunction with professional judgment.

The drill platform will be as specified by the designated Drilling Coordinator or project manager. The field conditions as known and as recognized from historical working experience for a specific project will be reviewed. The appropriate drill platform will be assigned.

The Project Manager or Task Manager assigned will coordinate with Drilling to provide during mobilization the appropriate equipment and tools to support the specified sampling procedures presented for the field work.

# Supporting Documents

• **ASTM D4700** Standard Guide for Soil Sampling from the Vadose Zone.

# **Other References**

- 1. A Well Drilling Methods Primer An Overview of the Various Major Well Construction Methods, Water Well Journal, November 1981.
- 2. Central Mine Equipment Manuals:CME550/CME750, specific to limits, use and supporting equipment of the drilling platform, most current.
- 3. *Drilling At Hazardous Waste Sites*, Drill Bits Newsletter of the National Drilling Foundation, Columbia, SC, December 1984.
- 4. *Drilling* Safety Guide, National Drilling Federation, Diamond Core Drill Manufacturers Association, and National Drilling Contractors Association, 1989.
- 5. *Sling* Safety, Bulletin 3072, U.S. Department of Labor, Occupational Safety and Health Administration, 1986.
- 6. *TERRACON Employee Job Descriptions*, Terranet, Terracon, Inc. Human Resources, 2000.

# E.310 AUGER DRILLING AND SAMPLING

## Last Review or Revision: June 2010

## **Objective and Application**

To provide standard procedure for auger drilling and soil sampling collection using traditional drilling equipment and techniques of the current industry. This procedure is applicable to drilling and sampling unconsolidated soils and highly weathered bedrock.

## Equipment

Drilling equipment will consist of commercially-made augers with drill flights sufficient to the needs of the project. Supporting tools and accessories will be utilized as specified by the manufacturer or a generic equivalent approved by the Drilling Coordinator or Equipment Manager.

## Training

The drill operator and drill helper will have entered and be current in the following;

- Industry drilling experience no less than 3 years for operator, 1 year for helper.
- Enrolled in Terracon's medical monitoring program for environmental operations (if employed by Terracon).
- Completed 40-Hour OSHA 1910.120 HAZWOPER training, with respiratory protection.
- Current certificate of annual 8-Hour OSHA 1910.120 HAZWOPER Refresher Training.
- Knowledge and experience in drilling both vadose and saturated soils.
- Knowledge and experience in the operation and maintenance of the specified drill platform.

If the drill operator and drill helper(s) are subcontractor employees, equivalent training and medical monitoring will be required.

# Procedures

The proper platform will be selected using as guidance ASTM D4700-91. Consistent with 1.7 of the standard the guidance is not intended to replace education or experience and will be used in conjunction with professional judgment.

The drill platform will be as specified by the designated Drilling Coordinator or the project manager. The field conditions as known and as recognized from historical working experience for a specific project will be reviewed. The appropriate drill platform will be assigned.

## TSOP E.310

The Project Manager or Task Manager assigned will coordinate with the drilling crew to provide the appropriate equipment and tools to support the specified sampling procedures presented for the fieldwork.

# Attached Supporting Documents

- **ASTM D1452-80** Standard Practice for Soil Investigation and Sampling by Auger Borings.
- **E.310.A** Auger Tools, Inc. manufacturer information

# Other References

- 1. A Well Drilling Methods Primer An Overview of the Various Major Well Construction Methods, Water Well Journal, November 1981.
- 2. Central Mine Equipment Manuals:CME550/CME750, specific to limits, use, and supporting equipment of the drilling platform, most current.
- 3. *Drilling At Hazardous Waste Sites*, Drill Bits Newsletter of the National Drilling Foundation, Columbia, SC, December 1984.
- 4. *Drilling* Safety Guide, National Drilling Federation, Diamond Core Drill Manufacturers Association, and National Drilling Contractors Association, 1989.
- 5. *Sling* Safety, Bulletin 3072, U.S. Department of Labor, Occupational Safety and Health Administration, 1986.
- 6. *TERRACON Employee Job Descriptions*, Terranet, Terracon, Inc. Human Resources, 2000.

# E.320 HOLLOW-STEM AUGER DRILLING

#### Last Review or Revision: June 2010

#### **Objective and Application**

To provide standard procedure to maintain the integrity of the borehole during drilling operations in soils or fills whereby the side walls of the boring cannot do so naturally, sufficient to prevent sloughing or caving of materials to depth. The use of hollow-stem augers allows the sampling of soils and access for well construction through their hollow centers.

The hollow stem auger procedure can be used in lieu of driven casing in conjunction with joint seals for fluid drilling.

This procedure is applicable to drilling and sampling unconsolidated soils and highly weathered bedrock.

## Equipment

Drilling equipment will be commercially-made hollow stem augers with drill flights sufficient to the needs of the project. Supporting tools and accessories will be as specified by the manufacturer or a generic equivalent approved by the Drilling Coordinator or Equipment Manager.

Four and one-quarter (4.25)-inch or eight and one-quarter (8.25)-inch nominal inside diameter hollow stem augers may be used for standard split spoon and Shelby tube sampling, special coring or rotary washboring operations and for the construction of monitoring wells. Nominal outside diameter of flights will be 9 to 11 inches, depending on manufacturer's construction. Commercially manufactured connecting bolts will be used to connect drill sections.

Joint seals will be of rubber or neoprene as recommended by individual manufacturer. For environmental sites with high solvent concentrations, special Teflon® seals may be required by the Project Manager and will be specified at mobilization.

Disposable end plugs will be of materials suitable to the task and as specified by Project manager or Equipment Manager. Environmental sites will use steel or stainless steel end plugs.

# Training

The drill operator and drill helper will have entered and be current in the following;

• Industry drilling experience no less than 3 years for operator, 1 year for helper.

#### **TSOP E.320**

#### Terracon

- Enrolled in Terracon's medical monitoring program for environmental operations operations (if employed by Terracon).
- Completed 40-Hour OSHA 1910.120 HAZWOPER training, inclusive of respiratory protection.
- Current certificate of annual 8-Hour OSHA 1910.120 HAZWOPER Refresher Training
- Knowledge and experience in drilling both vadose and saturated soils.

Knowledge and experience in the operation and maintenance of the specified equipment.

If the drill operator and drill helper(s) are subcontractor employees, equivalent training and medical monitoring will be required.

# Procedures

The field procedures will be as specified by the manufacturer for the equipment.

The application of the procedure for design will be by the Project Manager using as guidance ASTM D5784. Consistent with Section 1.5 of the standard the guidance is not intended to replace education or experience and will be used in conjunction with professional judgment.

# Attached Supporting Documents

• **ASTM D5784** Standard Guide for Use of Hollow-Stem Augers for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices.

# Other References

- 1. A Well Drilling Methods Primer An Overview of the Various Major Well Construction Methods, Water Well Journal, November 1981.
- 2. Central Mine Equipment Manuals:CME550/CME750, specific to limits, use and supporting equipment of the drilling platform, most current.
- 3. *Drilling* Safety Guide, National Drilling Federation, Diamond Core Drill Manufacturers Association, and National Drilling Contractors Association, 1989.

# E.325 CASING ADVANCE DRILLING AND SAMPLING

#### Last Revision or Review: June 2010

## **Objective and Application**

To provide standard procedure for drilling and soil sampling collection using equipment and techniques of the current industry. This procedure is applicable to drilling and sampling unconsolidated soils and highly weathered bedrock. It may be used in conjunction with rotary rock bit or coring attachments in consolidated bedrock.

## Equipment

Drilling equipment will be commercially-made sufficient to the needs of the project. Supporting tools and accessories will be as specified by the manufacturer or a generic equivalent approved by the Drilling Coordinator or Equipment Manager.

# Training

The drill operator and drill helper will have entered and be current in the following;

- Industry drilling experience no less than 3 years for operator, 1 year for helper.
- Enrolled in Terracon medical monitoring program for environmental operations (if employed by Terracon).
- Completed 40-Hour OSHA 1910.120 HAZWOPER training, with respiratory protection.
- Current certificate of annual 8-Hour OSHA 1910.120 HAZWOPER Refresher Training
- Knowledge and experience in drilling both vadose and saturated soils.
- Knowledge and experience in the operation and maintenance of the specified drill platform.

If the drill operator and drill helper(s) are subcontractor employees, equivalent training and medical monitoring will be required.

#### Procedures

The proper platform will be selected using as guidance ASTM D5872-95. Consistent with Sections 1.5 and 1.6 of the standard the guidance is not intended to replace education or experience and will be used in conjunction with professional judgment.

The drill platform will be as specified by the designated Drilling Coordinator of the Division. The field conditions as known and as recognized from historical working experience for a specific project will be reviewed. The appropriate drill platform will be assigned.

#### SOP E.325

#### Terracon

The Project Manager or Task Manager assigned will coordinate with the drilling crew to provide the appropriate equipment and tools to support the specified sampling procedures presented for the field work.

# **Attached Supporting Documents:**

• **ASTM D5872-95** Standard Guide for Use of Casing Advancement Drilling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices.

# Other References:

- 1. A Well Drilling Methods Primer An Overview of the Various Major Well Construction Methods, Water Well Journal, November 1981.
- 2. Central Mine Equipment Manuals:CME550/CME750, specific to limits, use and supporting equipment of the drilling platform, most current.
- 3. *Drilling At Hazardous Waste Sites*, Drill Bits Newsletter of the National Drilling Foundation, Columbia, SC, December 1984.
- 4. *Drilling Safety Guide*, National Drilling Federation, Diamond Core Drill Manufacturers Association, and National Drilling Contractors Association, 1989.
- 5. *Sling Safety*, Bulletin 3072, U.S. Department of Labor, Occupational Safety and Health Administration, 1986.
- 6. *TERRACON Employee Job Descriptions*, Terranet, Terracon, Inc. Human Resources, 2001 or most current.

# E.330 FLUID ROTARY DRILLING AND SAMPLING

#### Last Revision or Review: June 2010

#### **Objective and Application**

To provide standard procedure for drilling and soil sampling collection using equipment and techniques of the current industry. This procedure is applicable to drilling and sampling unconsolidated soils when subsurface conditions require the introduction of drilling fluids into the borehole to minimize cave-in. The drilling fluids are typically a water solution of a thixotropic clay (such as bentonite), with or without other admixtures. The drilling fluid, when pumped into the borehole during drilling, provides sufficient hydrostatic pressure against the borehole walls to prevent cave-in.

## Equipment

Drilling equipment will be commercially-made sufficient to the needs of the project. Supporting tools and accessories will be as specified by the manufacturer or a generic equivalent approved by the Drilling Coordinator or Equipment Manager.

## Training

The drill operator and drill helper will have entered and be current in the following;

- Industry drilling experience no less than 3 years for operator, 1 year for helper.
- Enrolled in Terracon medical monitoring program for environmental operations (if employed by Terracon)
- Completed 40-Hour OSHA 1910.120 HAZWOPER training, with respiratory protection.
- Current certificate of annual 8-Hour OSHA 1910.120 HAZWOPER Refresher Training
- Knowledge and experience in drilling both vadose and saturated soils.
- Knowledge and experience in the operation and maintenance of the specified drill platform.

If the drill operator and drill helper(s) are subcontractor employees, equivalent training and medical monitoring will be required.

#### Procedures

The proper platform will be selected using as guidance ASTM D5872-95. Consistent with Sections 1.5 and 1.6 of the standard the guidance is not intended to replace education or experience and will be used in conjunction with professional judgment.

The drill platform will be as specified by the designated Drilling Coordinator of the Division. The field conditions as known and as recognized from historical working experience for a specific project will be reviewed. The appropriate drill platform will be assigned.

The Project Manager or Task Manager assigned will coordinate with Drilling to provide during mobilization the appropriate equipment and tools to support the specified sampling procedures presented for the field work.

# Attached Supporting Documents:

• **ASTM D5872-95** Standard Guide for Use of Casing Advancement Drilling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices.

# Other References:

- 1. A Well Drilling Methods Primer An Overview of the Various Major Well Construction Methods, Water Well Journal, November 1981.
- 2. Central Mine Equipment Manuals:CME550/CME750, specific to limits, use and supporting equipment of the drilling platform, most current.
- 3. *Drilling At Hazardous Waste Sites*, Drill Bits Newsletter of the National Drilling Foundation, Columbia, SC, December 1984.
- 4. *Drilling Safety Guide*, National Drilling Federation, Diamond Core Drill Manufacturers Association, and National Drilling Contractors Association, 1989.
- 5. *Sling Safety*, Bulletin 3072, U.S. Department of Labor, Occupational Safety and Health Administration, 1986.
- 6. *TERRACON Employee Job Descriptions*, Terranet, Terracon, Inc. Human Resources, 2001 or most current.

# E.340 AIR ROTARY DRILLING AND SAMPLING

#### Last Revision or Review: June 2010

## **Objective and Application**

To provide standard procedure for drilling and soil sampling collection using equipment and techniques of the current industry. This procedure is applicable to drilling into consolidated bedrock. During this type of drilling, high pressure air is forced into the borehole during advancement. The high pressure air returns bedrock chips to the surface.

## Equipment

Drilling equipment will be commercially-made sufficient to the needs of the project. Supporting tools and accessories will be as specified by the manufacturer or a generic equivalent approved by the Drilling Coordinator or Equipment Manager.

# Training

The drill operator and drill helper will have entered and be current in the following;

- Industry drilling experience no less than 3 years for operator, 1 year for helper.
- Enrolled in Terracon medical monitoring program for environmental operations (if a Terracon employee).
- Completed 40-Hour OSHA 1910.120 HAZWOPER training, with respiratory protection.
- Current certificate of annual 8-Hour OSHA 1910.120 HAZWOPER Refresher Training
- Knowledge and experience in drilling both vadose and saturated soils.
- Knowledge and experience in the operation and maintenance of the specified drill platform.

If the drill operator and drill helper(s) are subcontractor employees, equivalent training and medical monitoring will be required.

#### Procedures

The proper platform will be selected using as guidance ASTM D5872-95. Consistent with Sections 1.5 and 1.6 of the standard the guidance is not intended to replace education or experience and will be used in conjunction with professional judgment.

The drill platform will be as specified by the designated Drilling Coordinator of the Division. The field conditions as known and as recognized from historical working experience for a specific project will be reviewed. The appropriate drill platform will be assigned.

## TSOP E.340

#### Terracon

The Project Manager or Task Manager assigned will coordinate with Drilling to provide during mobilization the appropriate equipment and tools to support the specified sampling procedures presented for the field work.

#### Terracon

# **Attached Supporting Documents:**

• **ASTM D5872-95** Standard Guide for Use of Casing Advancement Drilling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices.

# Other References:

- 1. A Well Drilling Methods Primer An Overview of the Various Major Well Construction Methods, Water Well Journal, November 1981.
- 2. Central Mine Equipment Manuals:CME550/CME750, specific to limits, use and supporting equipment of the drilling platform, most current.
- 3. *Drilling At Hazardous Waste Sites*, Drill Bits Newsletter of the National Drilling Foundation, Columbia, SC, December 1984.
- 4. *Drilling Safety Guide*, National Drilling Federation, Diamond Core Drill Manufacturers Association, and National Drilling Contractors Association, 1989.
- 5. *Sling Safety*, Bulletin 3072, U.S. Department of Labor, Occupational Safety and Health Administration, 1986.
- 6. *TERRACON Employee Job Descriptions*, Terranet, Terracon, Inc. Human Resources, 2001 or most current.

# E.400 SUBSURFACE SAMPLING - GEOPROBE® PLATFORM

Last Revision or Review: June 2010

# 1. OBJECTIVE

Geoprobe® equipment is a proprietary system capable of performing both sampling and onsite analysis functions. The proprietary name Geoprobe® has become synonymous with pushprobe technology, where the samplers or monitoring technology are advanced to depth by hydraulic push. The push may be assisted by inducing a vibration to the sampler.

Generally, sampling is accomplished using hydraulically pushed probes and analysis is accomplished using a gas chromatograph (GC). This equipment was originally developed to screen for the subsurface presence of volatile organic compounds (VOCs) in unsaturated zone soil gas. It may also be used to obtain soil and ground water samples and to screen such samples for VOCs or semivolatile organic compounds (SVOCs). However, the analytes of primary interest in Geoprobe® work are generally VOCs.

Geoprobe® equipment offers a relatively high degree of mobility for sampling and the ability to produce screening level analytical results while still mobilized to a site. The cost-effectiveness of this approach is reduced when soil or ground water samples are of interest, the analytes involved are SVOCs, or a higher degree of analytical quality is necessary.

The purpose of this document is to provide a standard field procedure for general application. It may be supplemented by the generation of written site-specific sampling and analysis plans (SAPs) prepared prior to field work. Project Managers are generally responsible for providing Geoprobe® operators with a SAP sufficiently in advance of proposed site work to allow for proper mobilization (including procurement of any necessary sampling equipment, sample containers, analytical standards, reagents, personal protective equipment, or other necessary equipment) and are required to consult with Geoprobe® operators in the development of such SAPs.

# 2. SAFETY AND HEALTH

All Terracon field work is carried out under the provisions of a safety and health plan. Most Geoprobe® work is covered by generic safety and health plans pertinent to intrusive work where petroleum hydrocarbons are anticipated or where there is a potential to encounter low concentrations of either petroleum hydrocarbons or chlorinated compounds. For any project involving greater potential hazard (e.g., work inside spaces with restricted ventilation and the potential to encounter high concentrations of volatile compounds, work involving the potential to encounter high concentrations of chlorinated compounds, or solvent extraction prior to analysis), the Project Manager will ensure that a site-specific safety and health plan that includes proper safety procedures for the Project-specific hazards is obtained from the Corporate Safety and Health Manager prior to mobilization.

## 3. EQUIPMENT

Geoprobe® equipment may vary from unit to unit. But should carry the equivalent of the following equipment mounted on a mobile one ton van or truck:

#### 1. Geoprobe® components -

- a. GW-40 hydraulically powered probe.
- b. 1 inch nominal diameter 2 to 4 feet length probe rods.
- c. Soil piston samplers and acetate liners.

# 2. GC components<sup>1</sup> -

- a. Shimadzu GC-14A (or equivalent) laboratory quality GC.
- b. Shimadzu (or equivalent) electron capture detector (ECD).
- c. Shimadzu (or equivalent) flame ionization detector (FID).
- d. Photoionization detector (PID).
- e. 30 m x 0.53 mm ID Supelco 3 mm Vocol (or equivalent) capillary column.
- f. Shimadzu (or equivalent) gas flow controller.
- g. Compressed air (service to FID).
- h. Compressed hydrogen (service to FID).
- i. Ultrapure compressed nitrogen or helium (carrier gas).
- j. Regulators for compressed gas bottles.
- k. Various size glass syringes (1 uL to 5 mL).
- I. Appropriate analytical standards.
- m. 40 mL nominal volatile organic analysis (VOA) vials.

#### 3. Data system components -

- a. APEX CSI (or equivalent) dual channel A to D converter.
- b. Laptop computer.
- c. APEX data system program (Version 2.1) (or equivalent).

#### 4. Miscellaneous equipment -

- a. Scale.
- b. Oven.
- c. Fire extinguisher.
- d. Various tools.
- e. Clean distilled and/or deionized water (hereinafter referred to as distilled) water.

<sup>&</sup>lt;sup>1</sup>Current equipment is listed. Equipment may change as appropriate for the analytes of concern and to reflect new technological developments. For example, the PID is currently out of service and is expected to be replaced in the future. Therefore, specific information regarding it is not listed. The standard column currently in use is listed. However, other columns may be more appropriate for the analyte(s) of concern in a specific project.

# 4. METHODS

#### A. Sample Collection and Holding

The Geoprobe® van, the working end of the hydraulic system (i.e., those portions of the system in contact with probe rods), all used probe rods, and all used sample contacting equipment will be cleaned prior to coming onsite for the first time in a project. Hydraulic lines will also be checked and tightened, if necessary, to ensure that no leaks are occurring. Clean probe rods will be used for each probe and used probe rods will be kept segregated from clean probe rods from the time they have been used until they have been cleaned. The working end of the hydraulic system will also be checked between probes and cleaned as necessary to reduce potential cross-contamination (see Section 4.D below regarding cleaning procedures).

# 1. Soil Gas Samples (Onsite Analysis)

a. Soil gas samples provide semiquantitative information concerning concentrations of VOCs in soil gas at the time of sampling. These may be influenced by the proximity of sources of VOC contamination (e.g., vertical contamination in the unsaturated zone soil profile or contaminated ground water) as well as a variety of other factors (e.g., soil and VOC characteristics and ambient temperature).

b. Soil gas samples will be taken at a depth of at least 6 feet below ground level (BGL). When attempting to characterize contaminated ground water plumes, soil gas samples will be taken within 3 feet of ground water.

c. Soil gas probes will be placed into the ground in a manner that will maintain a seal between the probe and the surrounding soil.

d. The suitability of each sampling location will be determined by testing the air permeability of the soil from which the sample is to be taken. An amount of air equal to five to 10 times the internal volume of the probe will be extracted prior to sampling. If this volume cannot be drawn within 10 minutes, soil gas cannot be used to test for VOC contamination at that location.

outside air.

e.

The sample will be collected for analysis without opening the system to

f. The sampling stream will be completely free of elastomers and each component of the sampling stream shall be new and clean or have been cleaned prior to each use. The cleanliness of the sampling stream shall be verified by running analytical blanks as specified in this document.

g. Soil gas samples will be analyzed immediately after the sample has been obtained.

# 2. Soil Samples (On/Offsite Analysis)

a. Currently accepted standard procedures for sample collection, sample preparation, and analysis of VOC contaminated soil samples necessarily involve substantial losses. Therefore, results for such samples should only be considered semiquantitative. Additionally, quantitative comparison of results should take into account whether they are based on dry or wet sample weight. Generally, commercial analytical laboratories report on a wet weight basis. However, results may be reported on a dry weight basis under certain circumstances.

b. Soil samples will be obtained using any standard Geoprobe® sampler. Typically these consist of various size piston samplers. Preference is given to the use of large bore samplers with removable acetate liners. These are capable of recovering cores 24 to 48 inches long and 1-1/8 inches in diameter. Standard thin-walled samplers (Shelby tubes) may also be utilized.

c. A variety of containers are appropriate for the collection and storage of soil samples. These include the acetate liners noted above. When such liners are used, the ends must be sealed during storage prior to analysis and aliquots to be analyzed should be obtained from as near the center of the sample as possible. Soil samples to be sent offsite for analysis should be collected in clean, 4 ounce glass containers with Teflon-lined lids. Soil should be placed in these containers rapidly, with as little matrix disturbance as possible, and in a manner that minimizes headspace. The container should be tightly sealed immediately after the sample is placed in it. All soil samples should be preserved by cooling to 4 °C if they are not analyzed immediately after collection. Geoprobe® analysis of soil samples for VOCs must be completed within 24 hours of sample collection.

d. Soil samplers will be cleaned prior to initial use at a site. They will also be cleaned prior to each subsequent use. The cleanliness of soil samplers shall be verified by running analytical blanks as specified in this document.

# 3. Ground Water Samples (On/Offsite Analysis)

a. Ground water samples obtained using Geoprobe® equipment can be expected to contain substantial concentrations of sediments. Therefore, they should be considered equivalent in character to borehole water samples.

b. Ground water samples will normally be collected by vacuum extraction using the same probe rods and tubing utilized for soil gas sample collection. The Geoprobe® may also be used to install small diameter slotted well points or push screen point samplers. Additionally, ground water samples may be obtained through probe rods or from slotted well points using stainless steel mini-bailers.

c. Ground water samples will be placed in clean, glass 40 mL VOA vials with open caps and Teflon septums. The sample shall be placed into these vials rapidly, with as little turbulence as possible, and in a manner that eliminates headspace. The container should be tightly sealed immediately after the sample is placed in it. All ground water samples should be preserved by cooling to 4 °C if they are not analyzed immediately after collection. Geoprobe® analysis of ground water samples for VOCs must be completed within 24 hours of sample collection.

d. The sampling stream will be completely free of elastomers and each component of the sampling stream shall be new and clean or have been cleaned in accordance with the procedures specified in this document prior to each use. When reusable equipment is used (e.g., stainless steel mini-bailers), it will be cleaned prior to initial use and each subsequent use. The cleanliness of the sampling stream shall be verified by running analytical blanks as specified in this document.

Probe holes will generally be abandoned by backfilling with bentonite pellets immediately after rods and samplers have been withdrawn. In the event more restrictive project, local, or state plugging requirements exist, they will be identified prior to initiation of field work and complied with.

# B. Sample Preparation and Analysis (Onsite Analysis)

Manufacturer instructions will be complied with in the operation of the GC and ancillary equipment for the analysis of environmental samples. Information from the scientific literature will also be relied on for guidance. Selection of columns, GC operating parameters, and detectors will be consistent with and appropriate for the types of analytes of concern.

# 1. Soil Gas Samples

Soil gas samples are obtained with a glass syringe and run by direct injection into the GC. No other sample preparation is required. The volume of the injection can be varied to keep the response within the range of the calibration curve and bracketed by standards. Soil gas samples must be run at the time they are obtained. They may not be held for subsequent analysis.

# 2. Soil and Ground Water Samples

# a. VOCs

1) VOCs must be extracted from the soil or ground water matrix for analysis by GC. This may be accomplished by purge and trap or headspace procedures. The normal Geoprobe® procedure will be heated headspace. Research indicates that results by either approach can be expected to correlate well in the case of water samples that do not contain substantial concentrations of sediments. Data pertaining to the effect of sediments is lacking; however, purge and trap would be expected to be a more effective extraction procedure than headspace in the case of VOCs that are more likely to be adsorbed (i.e., having higher log octanol/water partition coefficients). Differences in the efficiency of extraction procedures for specific VOCs should be considered when comparing Geoprobe® and analytical laboratory results for split samples.

2) Soil Heated Headspace Analysis

a) Weigh a clean, dry, 40 mL VOA vial (with the top off). Record this weight to the nearest 0.1 g. The approximate mean weight of such vials is 24.1 g.

b) Place approximately 5 g of the soil sample in the vial and obtain their combined weight. Record this weight to the nearest 0.1 g and proceed to the next step (the combined weight minus the empty vial's weight is the weight of the sample). In the case of heavily contaminated samples, the mass of sample used can be reduced to keep the response within the range of the calibration curve and bracketed by standards.

c) Using a graduated cylinder, add 20 mL of clean, distilled water into the vial with the sample and cover it snugly by screwing on the vial's open top cap and Teflon seal.

d) Shake the sample to attempt to break the soil up.

e) Heat the sample in an oven at 60 °C for 15 minutes to facilitate volatilization of VOCs from the sample into the vial headspace.

f) Using a clean glass syringe, obtain a headspace gas sample through the Teflon seal for direct injection into the GC. The volume of the injection can be varied to keep the response within the range of the calibration curve and bracketed by standards.

3) Ground Water Heated Headspace Analysis

a) Using a clean graduated cylinder, place 20 mL<sup>2</sup> of the ground water sample into a clean 40 mL VOA vial and cover it snugly by screwing on the vial's open top cap and Teflon seal.

b) Heat the sample in an oven at 60 °C for 15 minutes to facilitate volatilization of VOCs from the sample into the vial headspace.

c) Using a clean glass syringe, obtain a headspace gas sample through the Teflon seal for direct injection into the GC. The volume of the injection can be varied to keep the response within the range of the calibration curve and bracketed by standards.

b. Various chemical extraction methods are available which may be used for SVOCs. Selection of an appropriate method will be made on a case-by-case basis. Whenever such a method is used, quality assurance (QA) measures will be utilized to evaluate its effectiveness (e.g., submittal of duplicate samples for laboratory confirmation or analysis of soil media certified performance evaluation samples) and appropriate provisions will be incorporated within the project safety and health plan.

<sup>&</sup>lt;sup>2</sup>The intention is to fill the vial to a volume exactly the same as that used for standards and approximately half full. Since the actual volume of these vials is approximately 44 mL, this will make the volumetric concentration of headspace gas slightly less than that of the ground water sample, if all VOCs can be driven from the aqueous to the gaseous phase. However, aqueous phase samples and standards having the same contaminant concentrations should produce equal gas phase concentrations.

# 3. Temperature Programs

The temperature program utilized must be appropriate for the analyte(s) of concern. Standard programs are as follows:

- a. Chlorinated Solvents
  - 1) Injector 225 °C 2) Initial column<sup>3</sup> 35 °C for 2 minutes
    - 3) Column ramp rate 10 °C/minute
    - 4) Final column 100 °C for 2 minutes
    - 5) ECD and PID 250 °C
  - b. Petroleum Hydrocarbons
  - 1) Injector 225 °C
    - 2) Initial column 60 °C for 2 minutes
    - 3) Column ramp rate 10 °C/minute
    - 4) Final column
      - 1) BTEX/Gas TPH 150 °C for 2 minutes
      - 2) Diesel TPH 200 °C
    - 5) FID and PID 250 °C

# 4. Compressed Gas Flow Rates

The standard column nitrogen gas flow rate is 10 mL/minute. The standard FID gas flow rates are 20 mL/minute for air and 4 mL/minute for hydrogen.

# C. Analytical Quality Control and Assurance

Quality control (QC) and assurance (QA) terms have been variously defined. In this procedure they are defined as follows: QC consists of those activities performed for the purpose of controlling analytical quality; and QA consists of those activities performed for the purpose of providing assurance that analytical quality is in fact being achieved. By these definitions, QC activities include training of personnel, utilization of standard procedures, maintaining clean conditions through the use of new or properly cleaned equipment and reagents, maintenance of equipment, calibration activities, and documentation. QA activities include analysis of blanks, analysis of known concentrations or spikes (including surrogates), analysis of replicate samples, and audits. QA activities provide evidence that the analytical process is under control and capable of producing suitably unbiased, accurate, and precise results.

# 1. Quality Control

<sup>&</sup>lt;sup>3</sup>When ambient air temperature exceeds 75 °F the initial column temperature will be 45 °C.

A primary QC requirement is that GC operators be properly trained and knowledgeable. However, operator training will not be otherwise addressed in this document. The purpose of this procedure is to help ensure that standard methods are available and implemented. Equipment cleaning procedures are specified in Section 4.D of this document. In general, maintenance of all equipment will be performed as specified in manufacturer instructions. Documentation requirements are specified in Section 5 below. Minimum calibration requirements are as follows:

a. Standards for calibration will be prepared from pure standard materials or purchased as certified solutions. Standards will be prepared in methanol. Standards will be stored with minimal headspace, at 4 °C, and protected from light. All standards must be replaced after six months, or sooner if comparison with check standards or other QA measures indicate a problem.

b. Initial calibration. When new equipment has been placed in operation, an initial calibration curve will be generated. A minimum of three standard levels will be used for initial instrument calibration when either an FID or PID is in use. When an ECD is used, a minimum of five standard levels will be used.

c. Continuing calibration. The calibration curve will be verified each working day by the injection of one or more calibration standards prior to analysis of any samples. If the response for analytes is within the range of 80 to 120 percent of that predicted, sample analysis may continue with the same calibration curve. If recovery is outside of that range, a new calibration curve will be prepared for the analyte involved by running two additional standards. Calibration standards will be run at a frequency of at least one for every 10 samples. When an ECD is in use, this frequency will be increased to one for every five samples. When a new calibration curve is required, results for all samples which have been run since the last satisfactory continuing calibration will be appropriately qualified to indicate that circumstance.

d. The type of calibration standard will be appropriate to the type of sample being analyzed. Gas phase standards will be used when analyzing soil gas and aqueous phase standards will be used to produce headspace gas for injection when analyzing soil and ground water samples. The working range will generally be defined by initial calibration standards of the following concentrations -

1) Gas phase: 15, 30, and 60 ug/L for all detectors. These concentrations will be achieved by varying injection volumes from a single concentration standard.

2) Aqueous phase: 5, 25, and 100 ug/L for FID and PID and 1, 5, 10, 25, and 50 ug/L for ECD. These concentrations will be achieved by a combination of varying the mass of standard injected into distilled water and/or injection volumes.

e. The calibration curve will be linear and pass through the origin. Additionally, the calculated correlation coefficient for it shall be 0.95 or greater.

f. Other combinations of calibration standards may be used if information on site conditions indicates they would produce equivalent or better results.

Additional QC requirements are as follows:

a. Glass syringes may be used on one sample per day. After use, they must be cleaned prior to reuse. Cleaning is accomplished by washing as necessary, rinsing with hexane and methanol, and baking at a temperature of at least 60 °C for at least 15 minutes.

b. The GC sample injector port septum will be replaced regularly, depending on use, to prevent possible gas leakage.

c. When pulling gas or aqueous phase samples from probes, tubing will be connected to the vacuum pump via an adaptor which does not come into contact with the sample stream. The adaptor will be replaced periodically as necessary to ensure cleanliness and a good fit. At the end of each working day, adaptors which have been used will be cleaned with a detergent solution, rinsed with control water, and baked at a temperature of at least 60 °C for at least 15 minutes prior to being reused.

# 2. Quality Assurance

a. Method Blanks

Method blanks will be analyzed for the purpose of evaluating process bias. The following method blanks will be performed:

1) A method blank will be run at the beginning and, generally, near the end of each day of operation. However, the second method blank will not be run if cleaning of sample contacting equipment has not been performed or if results for a site are predominantly low or below detection limits. The method blank will include final rinse water from cleaning of probe rods and/or sample contacting equipment and aliquots of any reagents used in sample preparation. Distilled or "control water" will be substituted for final rinse water if the latter is unavailable.

2) If analytes are detected in a method blank, the source of the contamination will be identified and measures instituted to eliminate it. Results for any project samples which have already been run since the last clean method blank will also be evaluated to determine the impact of this circumstance. If the analytes involved were detected in them, they will be rerun. Otherwise, they will not be rerun.

b. Known Concentrations or Spikes

Continuing calibration standards will be utilized as known concentrations for the purpose of evaluating process accuracy and bias. Surrogates and matrix spikes will not normally be analyzed. If surrogates or matrix spikes are analyzed, the acceptance range is 80 to 120 percent recovery. Results within this range indicate an acceptable level of accuracy. If results are outside of this range, data must be appropriately qualified to indicate that circumstance and corrective action taken to regain process control.

c. Replicates

For every 10 samples (or at least once per day), a replicate shall be run for the purpose of evaluating process precision. Replicates shall be carefully prepared to minimize sampling variation as a source of error. All available site information shall be utilized to ensure that detectable concentrations of analytes are present in replicates. If detectable concentrations of analytes are present of at least one continuing calibration standard per day shall be run. Results of 25 percent or less relative percent difference (RPD) indicate an acceptable level of precision. The RPD for comparison with this criterion is calculated as the absolute difference between replicate results divided by their mean. If results exceed this RPD, data must be appropriately qualified to indicate that circumstance and corrective action taken to improve process control.

# d. Performance Evaluation Samples

The mobile laboratory QA Officer will ensure that at least one certified performance evaluation sample (water media) per quarter shall be run on a single blind basis. The nature of such samples shall take the general sample load into consideration. When target compounds are predominantly volatile petroleum hydrocarbons, most performance evaluation samples will consist of the compounds benzene, toluene, ethylbenzene, and xylenes (BTEX). At least once a year, a performance evaluation sample (water media) for selected volatile halocarbons will also be run.

# e. QA Audits

QA audits will be performed by the mobile laboratory QA Officer on an on-call and projectspecific basis. The file on at least one completed mobile laboratory project involving onsite GC analysis will be randomly selected for audit each quarter. At or about the same time, the mobile laboratory QA Officer will visually inspect the mobile laboratory and all onboard associated equipment.

The mobile laboratory QA Officer will prepare a report following each calendar year for submittal to Terracon. It will include the following information for that period:

- 1. Performance evaluation sample results.
- 2. Routine project file audit results.
- 3. On-call project-specific audit results.
- 4. Mobile laboratory van visual inspection results.
- D. Equipment Cleaning

Cleaning of field equipment will be performed in accordance with ASTM Standard D 5088-90. At a minimum, this means that probe rods and sample contacting equipment will be washed with a detergent solution and rinsed with "control water". Control water is defined as water having a known chemistry. Water from any public water supply operating in compliance with the Safe Drinking Water Act should meet this requirement. It will generally not be necessary to document the quality of control water unless there are unresolved method blank detections. Trihalomethanes are the volatile contaminants most likely to be encountered in control water. Where more rigorous cleaning procedures are necessary, they will be specified in the sitespecific SAP.

# 5. DOCUMENTATION

A separate project file (alpha identifier) will be maintained for Geoprobe® projects. This file will include the following minimum documents:

1. That portion of the project SAP covering Geoprobe® work.

- 2. The site-specific safety and health plan for all work requiring one.
- 3. A short narrative project report to include
  - a. A summary of field work performed.

b. A summary of field methods actually used if there were any substantial deviations from or changes to the SAP.

c. Reasons for any changes to the SAP.

d. A site diagram with sufficient detail or information to approximately identify the location of all probes performed and/or samples obtained.

e. The identity of onsite Terracon project personnel.

f. The identity of onsite client project personnel.

g. The identity of onsite regulatory or other significant personnel.

4. Results for each sample run (including calibration and QA samples). Results include the chromatogram obtained and output values determined for analytes (i.e., retention time, area, peak height, and concentration). The source of calibration should be identified for all samples.

5. The initial calibration curve, results of continuing calibration standards, and any subsequent calibration curves.

6. Results for all QA samples including calculated recovery and relative percent difference (RPD) values.

7. Other relevant project data.

# E.410 SUBSURFACE SAMPLING – GENERAL PUSH-PROBE TECHNOLOGY

Last Revision or Review: June 2010

# 1. OBJECTIVE

Geoprobe© equipment is a proprietary system capable of performing both sampling and onsite analysis functions. The proprietary name Geoprobe© has become synonymous with pushprobe technology, where the samplers or monitoring technology are advanced to depth by hydraulic push. The push may be assisted by inducing a vibration to the sampler.

Generally, sampling is accomplished using hydraulically pushed probes and analysis is accomplished using a gas chromatograph (GC). This equipment was originally developed to screen for the subsurface presence of volatile organic compounds (VOCs) in unsaturated zone soil gas. It may also be used to obtain soil and ground water samples and to screen such samples for VOCs or semivolatile organic compounds (SVOCs). However, the analytes of primary interest in Geoprobe work are generally VOCs.

Geoprobe equipment offers a relatively high degree of mobility for sampling and the ability to produce screening level analytical results while still mobilized to a site. The cost-effectiveness of this approach is reduced when soil or ground water samples are of interest, the analytes involved are SVOCs, or a higher degree of analytical quality is necessary.

The purpose of this document is to provide Terracon Geoprobe operators with a standard field procedure for general application. It may be supplemented by the generation of written site-specific sampling and analysis plans (SAPs) prepared prior to field work. Project Managers are generally responsible for providing Geoprobe operators with a SAP sufficiently in advance of proposed site work to allow for proper mobilization (including procurement of any necessary sampling equipment, sample containers, analytical standards, reagents, personal protective equipment, or other necessary equipment) and are required to consult with Geoprobe operators in the development of such SAPs.

# 2. SAFETY AND HEALTH

All Terracon field work is carried out under the provisions of a safety and health plan. Most Geoprobe work is covered by generic safety and health plans pertinent to intrusive work where petroleum hydrocarbons are anticipated or where there is a potential to encounter low concentrations of either petroleum hydrocarbons or chlorinated compounds. For any project involving greater potential hazard (e.g., work inside spaces with restricted ventilation and the potential to encounter high concentrations of volatile compounds, work involving the potential to encounter high concentrations of chlorinated compounds, or solvent extraction prior to analysis), the Project Manager will ensure that a site-specific safety and health plan is obtained from the Corporate Safety and Health Manager prior to mobilization.

# 3. EQUIPMENT

Geoprobe equipment may vary from unit to unit. But should carry the equivalent of the following equipment mounted in mobile one ton van:

## 1. Geoprobe components -

- a. GW-40 hydraulically powered probe.
- b. 1 inch nominal diameter 3 feet length probe rods.
- c. Soil piston samplers and acetate liners.

# 2. GC components<sup>1</sup> -

- a. Shimadzu GC-14A laboratory quality GC.
- b. Shimadzu electron capture detector (ECD).
- c. Shimadzu flame ionization detector (FID).
- d. Photoionization detector (PID).
- e. 30 m x 0.53 mm ID Supelco 3 mm Vocol capillary column.
- f. Shimadzu gas flow controller.
- g. Compressed air (service to FID).
- h. Compressed hydrogen (service to FID).
- i. Ultrapure compressed nitrogen or helium (carrier gas).
- j. Regulators for compressed gas bottles.
- k. Various size glass syringes (1 uL to 5 mL).
- I. Appropriate analytical standards.
- m. 40 mL nominal volatile organic analysis (VOA) vials.

#### 3. Data system components -

- a. APEX CSI dual channel A to D converter.
- b. Sager NP 700 486-66 laptop computer.
- c. APEX data system program (Version 2.1).

#### 4. Miscellaneous equipment -

- a. Scale.
- b. Oven.
- c. Fire extinguisher.
- d. Various tools.
- e. Clean distilled and/or deionized water (hereinafter referred to as distilled) water.

<sup>&</sup>lt;sup>1</sup>Current equipment is listed. Equipment may change as appropriate for the analytes of concern and to reflect new technological developments. For example, the PID is currently out of service and is expected to be replaced in the future. Therefore, specific information regarding it is not listed. The standard column currently in use is listed. However, other columns may be more appropriate for the analyte(s) of concern in a specific project.

# 4. METHODS

# A. Sample Collection and Holding

The Geoprobe van, the working end of the hydraulic system (i.e., those portions of the system in contact with probe rods), all used probe rods, and all used sample contacting equipment will be cleaned prior to coming onsite for the first time in a project. Hydraulic lines will also be checked and tightened, if necessary, to ensure that no leaks are occurring. Clean probe rods will be used for each probe and used probe rods will be kept segregated from clean probe rods from the time they have been used until they have been cleaned. The working end of the hydraulic system will also be checked between probes and cleaned as necessary to reduce potential cross-contamination (see Section 4.D below regarding cleaning procedures).

# 1. Soil Gas Samples (Onsite Analysis)

a. Soil gas samples provide semiquantitative information concerning concentrations of VOCs in soil gas at the time of sampling. These may be influenced by the proximity of sources of VOC contamination (e.g., vertical contamination in the unsaturated zone soil profile or contaminated ground water) as well as a variety of other factors (e.g., soil and VOC characteristics and ambient temperature).

b. Soil gas samples will be taken at a depth of at least 6 feet below ground level (BGL). When attempting to characterize contaminated ground water plumes, soil gas samples will be taken within 3 feet of ground water.

c. Soil gas probes will be placed into the ground in a manner that will maintain a seal between the probe and the surrounding soil.

d. The suitability of each sampling location will be determined by testing the air permeability of the soil from which the sample is to be taken. An amount of air equal to five to 10 times the internal volume of the probe will be extracted prior to sampling. If this volume cannot be drawn within 10 minutes, soil gas cannot be used to test for VOC contamination at that location.

e. The sample will be collected for analysis without opening the system to outside air.

f. The sampling stream will be completely free of elastomers and each component of the sampling stream shall be new and clean or have been cleaned prior to each use. The cleanliness of the sampling stream shall be verified by running analytical blanks as specified in this document.

g. Soil gas samples will be analyzed immediately after the sample has been obtained.

# 2. Soil Samples (On/Offsite Analysis)

a. Currently accepted standard procedures for sample collection, sample preparation, and analysis of VOC contaminated soil samples necessarily involve substantial losses. Therefore, results for such samples should only be considered semiquantitative. Additionally, quantitative comparison of results should take into account whether they are based on dry or wet sample weight. Generally, commercial analytical laboratories report on a wet weight basis. However, results may be reported on a dry weight basis under certain circumstances.

b. Soil samples will be obtained using any standard Geoprobe sampler. Typically these consist of various size piston samplers. Preference is given to the use of large bore samplers with removable acetate liners. These are capable of recovering cores 24 inches long and 1-1/8 inches in diameter. Standard thin-walled samplers (Shelby tubes) may also be utilized.

c. A variety of containers are appropriate for the collection and storage of soil samples. These include the acetate liners noted above. When such liners are used, the ends must be sealed during storage prior to analysis and aliquots to be analyzed should be obtained from as near the center of the sample as possible. Soil samples to be sent offsite for analysis should be collected in clean, 4 ounce glass containers with Teflon-lined lids. Soil should be placed in these containers rapidly, with as little matrix disturbance as possible, and in a manner that minimizes headspace. The container should be tightly sealed immediately after the sample is placed in it. All soil samples should be preserved by cooling to 4 °C if they are not analyzed immediately after collection. Geoprobe analysis of soil samples for VOCs must be completed within 24 hours of sample collection.

d. Soil samplers will be cleaned prior to initial use at a site. They will also be cleaned prior to each subsequent use. The cleanliness of soil samplers shall be verified by running analytical blanks as specified in this document.

# 3. Ground Water Samples (On/Offsite Analysis)

a. Ground water samples obtained using Geoprobe equipment can be expected to contain substantial concentrations of sediments. Therefore, they should be considered equivalent in character to borehole water samples.

b. Ground water samples will normally be collected by vacuum extraction using the same probe rods and tubing utilized for soil gas sample collection. The Geoprobe may also be used to install small diameter slotted well points or push screen point samplers. Additionally, ground water samples may be obtained through probe rods or from slotted well points using stainless steel mini-bailers.

c. Ground water samples will be placed in clean, glass 40 mL VOA vials with open caps and Teflon septums. The sample shall be placed into these vials rapidly, with as little turbulence as possible, and in a manner that eliminates headspace. The container should be tightly sealed immediately after the sample is placed in it. All ground water samples should be preserved by cooling to 4 °C if they are not analyzed immediately after collection. Geoprobe analysis of ground water samples for VOCs must be completed within 24 hours of sample collection.

d. The sampling stream will be completely free of elastomers and each component of the sampling stream shall be new and clean or have been cleaned in accordance with the procedures specified in this document prior to each use. When reusable equipment is used (e.g., stainless steel mini-bailers), it will be cleaned prior to initial use and each subsequent use. The cleanliness of the sampling stream shall be verified by running analytical blanks as specified in this document.

Probe holes will generally be abandoned by backfilling with bentonite pellets immediately after rods and samplers have been withdrawn. In the event more restrictive project, local, or state requirements exist, they will be identified prior to field work and complied with.

# B. Sample Preparation and Analysis (Onsite Analysis)

Manufacturer instructions will be complied with in the operation of the GC and ancillary equipment for the analysis of environmental samples. Information from the scientific literature will also be relied on for guidance. Selection of columns, GC operating parameters, and detectors will be consistent with and appropriate for the types of analytes of concern.

# 1. Soil Gas Samples

Soil gas samples are obtained with a glass syringe and run by direct injection into the GC. No other sample preparation is required. The volume of the injection can be varied to keep the response within the range of the calibration curve and bracketed by standards. Soil gas samples must be run at the time they are obtained. They may not be held for subsequent analysis.

# 2. Soil and Ground Water Samples

# a. VOCs

1) VOCs must be extracted from the soil or ground water matrix for analysis by GC. This may be accomplished by purge and trap or headspace procedures. The normal Geoprobe procedure will be heated headspace. Research indicates that results by either approach can be expected to correlate well in the case of water samples that do not contain substantial concentrations of sediments. Data pertaining to the effect of sediments is lacking; however, purge and trap would be expected to be a more effective extraction procedure than headspace in the case of VOCs that are more likely to be adsorbed (i.e., having higher log octanol/water partition coefficients). Differences in the efficiency of extraction procedures for specific VOCs should be considered when comparing Geo-probe and analytical laboratory results for split samples.

2) Soil Heated Headspace Analysis

a) Weigh a clean, dry, 40 mL VOA vial (with the top off). Record this weight to the nearest 0.1 g. The approximate mean weight of such vials is 24.1 g.

b) Place approximately 5 g of the soil sample in the vial and obtain their combined weight. Record this weight to the nearest 0.1 g and proceed to the next step (the combined weight minus the empty vial's weight is the weight of the sample). In the case of heavily contaminated samples, the mass of sample used can be reduced to keep the response within the range of the calibration curve and bracketed by standards.

c) Using a graduated cylinder, add 20 mL of clean, distilled water into the vial with the sample and cover it snugly by screwing on the vial's open top cap and Teflon seal.

d) Shake the sample to attempt to break the soil up.

e) Heat the sample in an oven at 60 °C for 15 minutes to facilitate volatilization of VOCs from the sample into the vial headspace.

f) Using a clean glass syringe, obtain a headspace gas sample through the Teflon seal for direct injection into the GC. The volume of the injection can be varied to keep the response within the range of the calibration curve and bracketed by standards.

3) Ground Water Heated Headspace Analysis

a) Using a clean graduated cylinder, place 20 mL<sup>2</sup> of the ground water sample into a clean 40 mL VOA vial and cover it snugly by screwing on the vial's open top cap and Teflon seal.

b) Heat the sample in an oven at 60 °C for 15 minutes to facilitate volatilization of VOCs from the sample into the vial headspace.

c) Using a clean glass syringe, obtain a headspace gas sample through the Teflon seal for direct injection into the GC. The volume of the injection can be varied to keep the response within the range of the calibration curve and bracketed by standards.

b. Various chemical extraction methods are available which may be used for SVOCs. Selection of an appropriate method will be made on a case-by-case basis. Whenever such a method is used, quality assurance (QA) measures will be utilized to evaluate its effectiveness (e.g., submittal of duplicate samples for laboratory confirmation or analysis of soil media certified performance evaluation samples) and appropriate provisions will be incorporated within the project safety and health plan.

<sup>&</sup>lt;sup>2</sup>The intention is to fill the vial to a volume exactly the same as that used for standards and approximately half full. Since the actual volume of these vials is approximately 44 mL, this will make the volumetric concentration of headspace gas slightly less than that of the ground water sample, if all VOCs can be driven from the aqueous to the gaseous phase. However, aqueous phase samples and standards having the same contaminant concentrations should produce equal gas phase concentrations.

## 3. Temperature Programs

The temperature program utilized must be appropriate for the analyte(s) of concern. Standard programs are as follows:

a. Chlorinated Solvents

1)	Inje	ctor 22	5 °C
	2)	Initial column <sup>3</sup>	35 °C for 2 minutes
	3)	Column ramp rate	e 10 °C/minute

- 4) Final column 100 °C for 2 minutes
- 5) ECD and PID 250 °C
- b. Petroleum Hydrocarbons
- 1) Injector 225 °C
  - 2) Initial column  $60 \,^{\circ}\text{C}$  for 2 minutes
  - 3) Column ramp rate 10 °C/minute
  - 4) Final column 1) BTEX/Gas TPH
    - 1) BTEX/Gas TPH 150 °C for 2 minutes
    - 2) Diesel TPH 200 °C
  - 5) FID and PID 250 °C

## 4. Compressed Gas Flow Rates

The standard column nitrogen gas flow rate is 10 mL/minute. The standard FID gas flow rates are 20 mL/minute for air and 4 mL/minute for hydrogen.

## C. Analytical Quality Control and Assurance

Quality control (QC) and assurance (QA) terms have been variously defined. In this procedure they are defined as follows: QC consists of those activities performed for the purpose of controlling analytical quality; and QA consists of those activities performed for the purpose of providing assurance that analytical quality is in fact being achieved. By these definitions, QC activities include training of personnel, utilization of standard procedures, maintaining clean conditions through the use of new or properly cleaned equipment and reagents, maintenance of equipment, calibration activities, and documentation. QA activities include analysis of blanks, analysis of known concentrations or spikes (including surrogates), analysis of replicate samples, analysis of evaluation samples, and audits. QA activities provide evidence that the analytical process is under control and capable of producing suitably unbiased, accurate, and precise results.

<sup>&</sup>lt;sup>3</sup>When ambient air temperature exceeds 75 °F the initial column temperature will be 45 °C.

## 1. Quality Control

A primary QC requirement is that GC operators be properly trained and knowledgeable. However, operator training will not be otherwise addressed in this document. The purpose of this procedure is to help ensure that standard methods are available and implemented. Equipment cleaning procedures are specified in Section 4.D of this document. In general, maintenance of all equipment will be performed as specified in manufacturer instructions. Documentation requirements are specified in Section 5 below. Minimum calibration requirements are as follows:

a. Standards for calibration will be prepared from pure standard materials or purchased as certified solutions. Standards will be prepared in methanol. Standards will be stored with minimal headspace, at 4 °C, and protected from light. All standards must be replaced after six months, or sooner if comparison with check standards or other QA measures indicate a problem.

b. Initial calibration. When new equipment has been placed in operation, an initial calibration curve will be generated. A minimum of three standard levels will be used for initial instrument calibration when either an FID or PID is in use. When an ECD is used, a minimum of five standard levels will be used.

c. Continuing calibration. The calibration curve will be verified each working day by the injection of one or more calibration standards prior to analysis of any samples. If the response for analytes is within the range of 80 to 120 percent of that predicted, sample analysis may continue with the same calibration curve. If recovery is outside of that range, a new calibration curve will be prepared for the analyte involved by running two additional standards. Calibration standards will be run at a frequency of at least one for every 10 samples. When an ECD is in use, this frequency will be increased to one for every five samples. When a new calibration curve is required, results for all samples which have been run since the last satisfactory continuing calibration will be appropriately qualified to indicate that circumstance.

d. The type of calibration standard will be appropriate to the type of sample being analyzed. Gas phase standards will be used when analyzing soil gas and aqueous phase standards will be used to produce headspace gas for injection when analyzing soil and ground water samples. The working range will generally be defined by initial calibration standards of the following concentrations -

1) Gas phase: 15, 30, and 60 ug/L for all detectors. These concentrations will be achieved by varying injection volumes from a single concentration standard.

2) Aqueous phase: 5, 25, and 100 ug/L for FID and PID and 1, 5, 10, 25, and 50 ug/L for ECD. These concentrations will be achieved by a combination of varying the mass of standard injected into distilled water and/or injection volumes.

e. The calibration curve will be linear and pass through the origin. Additionally, the calculated correlation coefficient for it shall be 0.95 or greater.

f. Other combinations of calibration standards may be used if information on site conditions indicates they would produce equivalent or better results.

Additional QC requirements are as follows:

a. Glass syringes may be used on one sample per day. After use, they must be cleaned prior to reuse. Cleaning is accomplished by washing as necessary, rinsing with hexane and methanol, and baking at a temperature of at least 60 °C for at least 15 minutes.

b. The GC sample injector port septum will be replaced regularly, depending on use, to prevent possible gas leakage.

c. When pulling gas or aqueous phase samples from probes, tubing will be connected to the vacuum pump via an adaptor which does not come into contact with the sample stream. The adaptor will be replaced periodically as necessary to ensure cleanliness and a good fit. At the end of each working day, adaptors which have been used will be cleaned with a detergent solution, rinsed with control water, and baked at a temperature of at least 60 °C for at least 15 minutes prior to being reused.

## 2. Quality Assurance

## a. Method Blanks

Method blanks will be run for the purpose of evaluating process bias. The following method blanks will be performed:

1) A method blank will be run at the beginning and, generally, near the end of each day of operation. However, the second method blank will not be run if cleaning of sample contacting equipment has not been performed or if results for a site are predominantly low or below detection limits. The method blank will include final rinse water from cleaning of probe rods and/or sample contacting equipment and aliquots of any reagents used in sample preparation. Distilled or "control water" will be substituted for final rinse water if the latter is unavailable.

2) If analytes are detected in a method blank, the source of the contamination will be identified and measures instituted to eliminate it. Results for any project samples which have already been run since the last clean method blank will also be evaluated to determine the impact of this circumstance. If the analytes involved were detected in them, they will be rerun. Otherwise, they will not be rerun.

## b. Known Concentrations or Spikes

Continuing calibration standards will be utilized as known concentrations for the purpose of evaluating process accuracy and bias. Surrogates and matrix spikes will not normally be analyzed. If surrogates or matrix spikes are analyzed, the acceptance range is 80 to 120 percent recovery. Results within this range indicate an acceptable level of accuracy. If results are outside of this range, data must be appropriately qualified to indicate that circumstance and corrective action taken to regain process control.

c. Replicates

For every 10 samples (or at least once per day), a replicate shall be run for the purpose of evaluating process precision. Replicates shall be carefully prepared to minimize sampling variation as a source of error. All available site information shall be utilized to ensure that detectable concentrations of analytes are present in replicates. If detectable concentrations of analytes are not present in samples, a replicate of at least one continuing calibration standard per day shall be run. Results of 25 percent or less relative percent difference (RPD) indicate an acceptable level of precision. The RPD for comparison with this criterion is calculated as the absolute difference between replicate results divided by their mean. If results exceed this RPD, data must be appropriately qualified to indicate that circumstance and corrective action taken to improve process control.

## d. Performance Evaluation Samples

The Geoprobe QA Officer will ensure that at least one certified performance evaluation sample (water media) per quarter shall be run on a single blind basis. The nature of such samples shall take the general sample load into consideration. When target compounds are predominantly volatile petroleum hydrocarbons, most performance evaluation samples will consist of the compounds benzene, toluene, ethylbenzene, and xylenes (BTEX). At least once a year, a performance evaluation sample (water media) for selected volatile halocarbons will also be run.

e. QA Audits

QA audits will be performed by the Geoprobe QA Officer on an on-call and project-specific basis. The file on at least one completed Geoprobe project involving onsite GC analysis will be randomly selected for audit each quarter. At or about the same time, the Geoprobe QA Officer will visually inspect the Geoprobe van and all onboard associated equipment.

The Geoprobe QA Officer will prepare a report following each calendar year for submittal to all Southern Division Office Managers. It will include the following information for that period:

- 1. Performance evaluation sample results.
- 2. Routine project file audit results.
- 3. On-call project-specific audit results.
- 4. Geoprobe van visual inspection results.
- D. Equipment Cleaning

Cleaning of field equipment will be performed in accordance with ASTM Standard D 5088-90. At a minimum, this means that probe rods and sample contacting equipment will be washed with a detergent solution and rinsed with "control water". Control water is defined as water having a known chemistry. Water from any public water supply operating in compliance with the Safe Drinking Water Act should meet this requirement. It will generally not be necessary to document the quality of control water unless there are unresolved method blank detections. Trihalomethanes are the volatile contaminants most likely to be encountered in control water. Where more rigorous cleaning procedures are necessary, they will be specified in the site-specific SAP.

## 5. DOCUMENTATION

A separate project file (alpha identifier) will be maintained for Geoprobe projects. This file will include the following minimum documents:

- 1. That portion of the project SAP covering Geoprobe work.
- 2. The site-specific safety and health plan for all work requiring one.
- 3. A short narrative project report to include
  - a. A summary of field work performed.

b. A summary of field methods actually used if there were any substantial deviations from or changes to the SAP.

c. Reasons for any changes to the SAP.

d. A site diagram with sufficient detail or information to approximately identify the location of all probes performed and/or samples obtained.

e. The identity of onsite Terracon project personnel.

- f. The identity of onsite client project personnel.
- g. The identity of onsite regulatory or other significant personnel.

4. Results for each sample run (including calibration and QA samples). Results include the chromatogram obtained and output values determined for analytes (i.e., retention time, area, peak height, and concentration). The source of calibration should be identified for all samples.

5. The initial calibration curve, results of continuing calibration standards, and any subsequent calibration curves.

6. Results for all QA samples including calculated recovery and relative percent difference (RPD) values.

7. Other relevant project data.

# E.450 SUBSURFACE SOIL SAMPLING – XITECH SAMPLER

#### Last Revision or Review: June 2010

## **Objective and Application**

To provide standard procedure to obtain a sample of soil or other materials *in-situ* that is reasonably representative of the physical and chemical nature of the materials as they reside in the subsurface.

This is done by pressing a thin-walled metal tube and liner into the soil/fill, removing the soil filled tube and removing the soil from the internal liner.

The application is appropriate to cohesive soils with high clay content sufficient to remain stable within the sampler. The method is not appropriate for unconsolidated materials or highly weathered bedrock.

## Equipment

- Xitech hand auger assembly
- Xitech slide hammer assembly
- Xitech probe extensions
- Internal sampler liners (plastic or brass)
- Disposable chemical-resistant gloves.
- Work gloves as appropriate.
- Laboratory prepared sample containers.
- Roll of plastic sheeting.
- Plastic trash bag for collecting expended supplies.
- Field documentation forms or project logbook.
- Chain-of-Custody forms for samples intended for laboratory analysis.
- Marking pencils or indelible markers that will not leave residues which can cause interference with laboratory testing procedures.

#### Procedures

Select the location. Identify it with a unique designation for the project. Diagram or otherwise describe the location on site diagrams or in the logbook relative to a fixed benchmark that will allow the specific location to be re-visited in the future, if necessary. If appropriate, estimate the vertical elevation of the sample and record.

If the sample is constructed of multiple aliquots to represent averaging of conditions in soils/tailings/spoils, diagram or otherwise describe the area represented by the constructed

#### TSOP E.450

sample. Diagram or otherwise accurately describe sub-sample locations on forms or in the logbook relative to a fixed benchmark that will allow the specific sub-sample locations to be revisited in the future, if necessary. If appropriate, estimate the vertical elevation of the sub-samples and record.

At each location before collecting the soil sample, clean the Xitech sampling equipment using the appropriate procedure as required by the project. If using brass liners, clean the liners before each use. If using plastic liners, use a clean liner prior to collecting each sample. Use a clean pair of disposable chemical-resistant gloves before handling soil samples.

Assemble the Xitech hand auger assembly by attaching the tee-handle onto the auger drill head. Rotate the auger drill head into the soil. When the containment chamber becomes full, remove the auger drill head and manually clear the chamber. Attach extensions to the hand auger assembly as necessary to reach the desired depth.

When the desired depth has been reached, remove the hand auger drill head and attach the probe head. Slip a liner (brass or plastic) into the sampler housing and secure using the locking pin. Remove the hand auger tee-handle and attach the slide hammer assembly. Lower the probe with liner into the augered hole and hammer the assembly six (6) inches into the soil. Hammer upward (if necessary) to retrieve the core.

Place the sample directly into the laboratory prepared sample container(s) and complete the sample label and Chain-of-Custody as instructed by the project manager.

If intended for laboratory analysis, preserve the sample as required by project plans.

## Attached Supporting Documents

• Soil Sampling Section from Owners Manual for Xitech Instruments, Inc. Soil Gas Sampler, Model Vista 4000.

# E.460 SUBSURFACE SOIL SAMPLING – SHELBY TUBE APPARATUS

Last Review or Revision: June 2010

#### **Objective and Application**

To provide standard procedure to obtain a sample of soil or other materials *in-situ* that is reasonably representative of the physical and chemical nature of the materials as they reside in the subsurface.

This is done by pressing a thin-walled metal tube into the soil/fill, removing the soil-filled tube and sealing the tube sampler against disturbance.

The application is appropriate to cohesive soils with high clay content sufficient to remain stable within the sampler. The method is not appropriate for unconsolidated materials or highly weathered bedrock.

## Equipment

The apparatus will be generally consistent with ASTM D1587 and will be commercially manufactured.

The materials of the apparatus will be compatible, physically and chemically with the materials to be sampled.

The equipment will be used with proper accessories and equipment compatible with the drilling/sampling platforms specified under TSOP E.300.

The samplers will be maintained consistent with the manufacturer's recommendations.

#### Procedures

The procedure for applying the equipment will be determined by the Project Manager using as guidance ASTM D1584. The guidance is not intended to replace education or experience and will be used in conjunction with professional judgment.

## Attached Supporting Documents

- **ASTM D6169-98** Standard Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations.
- ASTM D1587 Standard Practice for Thin-Walled Tube Geotechnical Sampling of Soils.
- **E.460.A** *Thin Wall Tube Sampler*, Soil Sampling, Core Rotary Tools and Accessories Catalog 747, Mobile Drilling Company, Inc.

## **Other References**

- 1. A Well Drilling Methods Primer An Overview of the Various Major Well Construction Methods, Water Well Journal, November 1981.
- 2. Central Mine Equipment Manuals:CME550/CME750, specific to limits, use and supporting equipment of the drilling platform, most current.
- 3. *Drilling Safety Guide*, National Drilling Federation, Diamond Core Drill Manufacturers Association, and National Drilling Contractors Association, 1989.

# E.465 SUBSURFACE SOIL SAMPLING – SPLIT BARREL

#### Last Review or Revision: June 2010

#### **Objective and Application**

To provide standard procedure to obtain a sample of soil or other materials *in-situ* that is reasonably representative of the physical and chemical nature of the materials as they reside in the subsurface.

This is done by drilling inside a hollow-stem auger, pressing or pounding a split-barreled metal tube into the soil/fill, removing the soil-filled tube from the ground, and obtaining the soil sample by removing the two halves of the barrel.

The application is appropriate to cohesive soils with high clay content sufficient to remain stable within the sampler. The method is not appropriate for consolidated materials or highly weathered bedrock. For unconsolidated materials, placement of a catcher within the shoe of the split barrel sampler is necessary to ensure retention of the unconsolidated material with the sampler.

#### Equipment

The apparatus will be generally consistent with ASTM D1586 and will be commercially manufactured.

The materials of the apparatus will be compatible, physically and chemically with the materials to be sampled.

The equipment will be used with proper accessories and equipment compatible with the drilling/sampling platforms specified under TSOP E.300.

The samplers will be maintained consistent with the manufacturer's recommendations.

## Procedures

The procedure for applying the equipment will be determined by the Project Manager using as guidance ASTM D1584. The guidance is not intended to replace education or experience and will be used in conjunction with professional judgment.

#### TSOP E.465

## Attached Supporting Documents

- **ASTM D1586** Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils.
- **E.465.A** Split Barrel Sampler, Soil Sampling, Core Rotary Tools and Accessories Catalog 747, Mobile Drilling Company, Inc.

## Other References

- 1. A Well Drilling Methods Primer An Overview of the Various Major Well Construction Methods, Water Well Journal, November 1981.
- 2. Central Mine Equipment Manuals:CME550/CME750, specific to limits, use and supporting equipment of the drilling platform, most current.
- 3. *Drilling Safety Guide*, National Drilling Federation, Diamond Core Drill Manufacturers Association, and National Drilling Contractors Association, 1989.
- 4. ASTM D6169-98 Standard Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations.

## E.468 SAMPLE HANDLING - SOIL (LEVEL D)

#### Last Review or Revision: June 2010

1. OBJECTIVE

To obtain a representative soil or sediment sample for chemical analysis. This includes the documentation of sampling methods, and protocols used for sample collection, processing, handling and shipment.

#### 2. EQUIPMENT

- Monitoring equipment (HNU, OVA, OVM, TGI, TIP, FID color metric detector tubes) as specified by Project Manager;
- Sampling Device (split barrel sampler, hand auger, hand trowel, shovel, posthole digger, tube sampler, or other appropriate sampling device);
- Decontamination Equipment;
- Laboratory prepared sample containers;
- Forms including "Soil Sampling Information Sheet", chain-of-custody, etc;
- Indelible ink pen;
- Stainless steel bowl;
- Plastic sheet;
- Site map;
- Measuring wheel;
- Engineers tape marked in units of feet, tenths of a foot (0.1 ft.), and hundredths of a foot (0.01 ft.);
- Tool box;
- Disposable chemical-resistant gloves; and
- Chem-wipes.

#### 3. PROCEDURES.

- a) Surficial soil/sampling
  - Determine sample location (set grid, if necessary)
  - Determine the proper sampling device based on soil type, depth, sample type, etc.
  - Collect each sample at the specified depth consistently for each sample.
- b) Direct Sampling
  - Transfer sample directly from the sampling device to the sample container.
  - If evaluating for organic vapors, transfer half of sample to glass mason jar or plastic bag (zip top) for field testing. The sample should be split so as to obtain a sample for screening that is representative of the sample for testing. This can be accomplished by slicing the sample (if cohesive) lengthwise or by using other mechanical means. Care should be taken so as not to over-agitate the sample, especially if volatile organic compound testing is required.
  - Document visual and physical characteristics
- c) Composite sampling (non-volatile only)
  - Decreases analytical cost but also decreases ability to detect low level contamination
  - Transfer equal volume/weight of sample from each location/depth to a stainless steel mixing bowl
  - Use a hand trowel or spoon to mix the soil sample
  - If the sample size is very large, composite on a large sheet of clean plastic or stainless steel cookie sheet pan, or mix equal volumes from numerous composite samples.
  - If soils are cohesive, break up clumps.
  - Spread soil uniformly on plastic sheet or in bottom of stainless steel bowl or stainless steel tray and divide into quarters.
  - Obtain equal quantity of soil from each sample for transfer to sample container (without mixing or break up).

- d) Decontamination
  - Decontamination procedures should be specified by the project manager.
  - Decontamination procedures for UST sites includes an Alconox® detergent scrub followed by a clean water rinse.
  - Decontamination fluids are to be replaced between sample locations (each boring) to reduce the potential for cross contamination.
- e) Sample preservation store in cooler with ice.
- f) Sample documentation
  - Complete the "Soil Sampling Information Sheet" and chain-of-custody form. Date to be recorded includes sampling location, methodology, depth, visual and physical characteristics, time and date.
- 4. ATTACHED SUPPORTING DOCUMENTATION
  - a) ASTM D4220 Practice For Preserving and Transporting Soil Samples
- 5. OTHER REFERENCES
  - a) Laboratory- or program-specific requirements for handling, preservation, and transport of samples for chemical analyses.

Terracon

٦

## EXAMPLE SOIL SAMPLING INFORMATION SHEET

PROJECT NAME \_\_\_\_\_\_ PROJECT NO. \_\_\_\_\_

PROJECT LOCATION \_\_\_\_\_

SAMPLE POINT DESCRIPTION _	 	-
SAMPLE INTERVAL SAMPLE DESCRIPTION		
SAMPLE APPEARANCE		-
ORGANIC VAPOR READING		_
SAMPLING PROBLEMS		-
CLEANING PERFORMED IN FIEL		_
COMMENTS	 	

SAMPLE POINT DATE TIME	
SAMPLE POINT DESCRIPTION	
SAMPLE METHOD	
SAMPLE INTERVAL	
SAMPLE DESCRIPTION	
SAMPLE APPEARANCE	
ORGANIC VAPOR READING	
SAMPLING PROBLEMS	
CLEANING PERFORMED IN FIELD	
COMMENTS	

FORM COMPLETED BY: \_\_\_\_\_ DATE \_\_\_\_\_

# E.470 SAMPLE HANDLING – GROUNDWATER (LEVEL D)

#### Last Review or Revision: June 2010

#### OBJECTIVE

To collect a representative groundwater sample from the sampling point for chemical analysis. This includes the documentation of sampling methods, sampling supplies, and protocol to reduce potential for alteration and or cross-contamination during the sampling event.

#### EQUIPMENT

- Monitoring equipment specified by project manager;
- Electronic water level indicator, phase level indicator, etc.;
- Decontamination equipment;
- Proper forms, labels and indelible ink pen;
- pH, temperature, and specific conductance meter;
- Sample containers and packing material, tape, and labels;
- Filtration device and filters (and fixing agents as appropriate);
- Cooler with ice pack and packing media;
- Bucket (calibrated in gallons or liters)
- Sampling device
- Bailers, pumps, etc.
- Keys for locking cap on well;
- Rope steel, nylon, teflon, or polypropylene;
- Deionized (DI) water;
- Chemical-resistant gloves
- Knife; and
- Site map.

## PROCEDURES

- a) Preparation
  - Meet with Project Manager;
  - Obtain the bottles, forms, and equipment necessary to complete the sampling event;
  - Calibrate all field equipment i.e., pH and conductivity meters;
  - Establish sampling well sequence (generally least impacted to most impacted).
- b) Field Activities
  - Water levels collect and record water levels (and well depth, if requested by P.M.)
- c) Contamination minimization
  - Use plastic sheet if necessary
  - Use proper bailing techniques (hand over hand) to prevent rope from touching the ground or low flow sampling techniques with dedicated disposable tubing



- d) Sample Collection
  - 1) Preservation
    - Use containers with proper preservative if necessary.
    - Routine preservatives are listed in the attached documentation (Check with Project Manager and/or lab).
  - 2) Filtration
    - Metals only (Do not filter samples for VOC analyses).
    - Field filter samples collected for dissolved metals analyses immediately after collecting the sample (if required and/or allowed by local or state regulations).
    - Filter the sample prior to adding preservative or transferring to sample container containing a preservative(if required and/or allowed by local or state regulations).
    - Discard the filter, tubing and transfer container.

#### Terracon

#### **TSOP E.470**

- 3) Collection
  - Minimize disturbances that may aerate the sample (i.e., lower bailer slowly into water, pour slowly into sample container, use low flow sampling procedures, etc.).
  - Pour water from the top of the bailer or insert the bottom emptying device for sample transfer.
  - Transfer the groundwater sample directly to the laboratory prepared sample container or the filter cup.
  - Samples collected for VOCs should always be collected from a recently filled bailer full of water as soon as it is brought to the surface.



- Collect samples for VOCs by forming a positive meniscus on the sample vial and capping immediately.
- VOC samples must be free of air bubbles.
- Do not over-fill sample containers which contain a preservative.
- Place samples in cooler with ice or blue-ice.
- 4) Filtration Procedures
  - Set up filtering apparatus according to the manufacturer's directions.
  - Use a 0.45 micron membrane filter (may need a pre-filter to prevent clogging if the sample is turbid).
  - Flush a minimum of 250 ml of D.I. water (or larger volume if recommended by manufacturer specifications) through the filtering apparatus and filter prior to filtering the sample.
  - Pump the sample through the filter and discard the initial 100-200 ml (if you have sufficient volume).
  - Collect and transfer the remaining sample volume to the sample container.
- 5) Data Documentation
  - Record all pertinent sampling information on the sampling container label, sampling information form, chain-of-custody, and shipping form.

- Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, sampling methodology, parameters to be analyzed, stabilization data, and general observations.
- Make all entries in the chain-of-custody form prior to leaving the site. Ensure that the chain-of-custody protocol required for the project is maintained.
- If samples must be shipped, the chain-of-custody form must be enclosed with the samples and the container sealed with Terracon security labels. Obtain a post office receipt, bill of lading or similar document from the shipper to be included as part of the chain-ofcustody documentation. Return one copy of the chain-of-custody documentation to the project manager.
- 6) Equipment Cleaning
  - Clean all equipment used as specified by the Project Manager and according to cleaning procedures prior to collecting a sample from the next sample location.
- 7) Sample Packaging and Shipment
  - Carefully package the samples in a cooler with ice. Take care to wrap the sample containers in packing materials and place in sealed Zip top bags.
  - Ship the samples via overnight courier as specified by the Project Manager. Be sure to secure all address labels with clear packing tape.

## **Attached Supporting Documents**

• Typical Chemical Analysis Methods and Preservatives.

#### **Other Supporting Documents**

• **ASTM D4448-85a** Standard Guide for Sampling Groundwater Monitoring Wells.

## **Typical Chemical Analysis Methods and Preservatives**

Analytical Test Method	Pilot Chemical Group	Sample Matrix			Sample Quantity (Minimum)														Holding	
					Field Recovery Quantity			Laboratory Quantity								Preservative				
		Soil	Water	Gas/Vapor	1500 grams	200 milliliters	2 Liters	400 milligrams	4 oz	16 oz.	40 milliliters	200 milliliters	250 milliliters	500 milliliters	1 Liter	Cool 4°C, dark	pH<2, w/HNO₃	pH<2 w/HCL	Time (Days)	Container Type
EPA 8260B	VOCs	Х			Х				Х							Х			14*	8 oz. CWM
ETA 0200D			Х				Х				Х							Х	14*	40 mL GV
EPA 8270C	SVOCs	х			Х				Х							Х			7**	8 oz. CW M
			Х												Х	Х			7**	2 Liter AWM
EPA 8082	PCBs	Х			Х				Х							X			7**	8 oz. CWM
			Х				Х								Х	X			7**	2 Liter AWM
EPA 6010	Metals	Х			х				Х							Х			180	4 oz. CWM
			Х				Х							Х			Х		180	500 mL HDPE
NIOSH 1501	Benzene, Toluene			Х		Х		Х								X			14	Carbon Filter
EPA 9040A	рН	Х			Х				Х							X			14*	4 oz. CWM
			Х				Х						X			X			14*	250 mL HDPE

\* = Preserve within 2 days then analyze within 14 days \*\* = Extraction to occur within 7 days and analysis within 40 days after extraction

CWM = clear wide mouth

HDPE = High-density polyethylene bottles

GV = Glass vial

AWM = Amber wide mouth

AJ = Amber Jug

# E.480 SURFACE WATER SAMPLING

#### Last Review or Revision: June 2010

#### OBJECTIVE

To collect a representative surface water sample from the sampling point for chemical analysis. This includes the documentation of sampling methods, sampling supplies, and protocol to reduce potential for alteration and or cross-contamination during the sampling event.

This TSOP describes the procedure and equipment for collecting surface samples of water or other liquids using a dipper or equivalent. A pond sampler or dipper with extension handle allows the operator to sample streams, ponds, waste pits, and lagoons as far as 15 ft from the bank or other secure footing. The dipper is useful in filling a sample bottle without contaminating the outside of the bottle. This TSOP can be used to describe the sampling procedures to be used from a boat or from within the stream using hip-waders, however specific safety precautions should be implemented in these situations. See the project manager if sampling will not be conducted from the shore or other stable surface (boat dock, etc.).

#### EQUIPMENT

- Sampling and monitoring equipment specified by project manager;
- Disposable chemical-resistant gloves;
- Cleaning equipment;
- Proper forms, labels and indelible ink pen;
- Sample containers and packing material, tape, and labels;
- Cooler with ice pack and packing media;
- Dipper or equivalent sampling device; and
- Site map.

#### PROCEDURES

- 1. Clean sampling equipment in accordance with E.2410.
- 2. Locate sampling site at the designated point in the stream.
- 3. Attach extensions to the dipper as required to reach the distance from the shore specified by the project manager.
- 4. Submerge the container end of the dipper at sampling point such that mouth of dipper is at the depth specified by the project manager. If no depth has been specified, submerge the dipper about 2 to 3 inches below the water surface, if possible.



- 5. Allow the dipper to fill; rinse the dipper by shaking and discharging this water. Repeat this procedure three times.
- 6. Collect sample and transfer into a holding container or directly to the laboratory sample container. If a holding container is used for ease of transfer or to create a composite sample from multiple sampling locations, transfer water from the holding container into sampling bottles.
- 7. Fill out appropriate field form(s) documenting sample location, time, and other pertinent information before leaving sampling site.

## **OTHER SUPPORTING DOCUMENTS**

**ASTM D5358-93** Standard Practice for Sampling with a Dipper or Pond Sampler.

## E.500 pH FIELD SCREENING - SOIL

#### Last Review or Revision: June 2010

#### **Objective and Application**

To provide a qualitative and limited quantitative field screening of soil samples to aid in the evaluation of soil for acidity or causticity. This procedure is applicable for sites where moderately low or high pH soils are expected, such as coal pile storage.

## Equipment

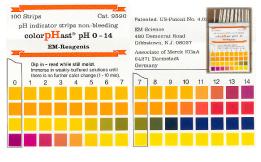
- Disposable chemical resistant gloves,
- Sterile container or clean zip top bag,
- ColorPHast® or equivalent litmus strips, and
- Deionized water.

Note regarding the ColorPHast® or equivalent litmus strips: These litmus strips are commonly available to cover a range of possible pHs. Typical ranges used for this procedure are 0-6, 5-10, and 7.5-14 standard units. Other ranges are also available. Before field mobilization, discuss the anticipated pH range of the soils to be tested and mobilize strips for the appropriate range. Strips should be stored in a dry location; it may be necessary to package the strips in a watertight zip top bag or equivalent before mobilization into the field.

#### Procedures

For each soil sample to be screened for pH, place a small (1oz.) amount of soil in a sterile container or clean zip top bag. Add an approximately equal amount of deionized water. Mix the solution until a slurry results or until all of the soil has been wetted. Place a litmus strip in the solution and allow the strip to become fully wetted. A test time of about 30 seconds is usually sufficient.

Compare the color of the test strip to the pH indicator chart on the front of the ColorPHast® package. The precision of the indicator chart will vary depending on the range of the strips used. The example shown indicates that for strips covering the 0-14 standard units range, a precision of 1 standard unit is available. Intermediate pH values may be estimated by interpolating between the colors shown on the chart.



#### **Documentation**

Record the pH of the sample on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

## **Other Supporting Documents**

ASTM D4972-01 Standard Test Method for pH of Soils.

# E.530 pH FIELD SCREENING - WATER

#### Last Revision or Review: June 2010

## **Objective and Application**

To provide a qualitative and limited quantitative field screening of water samples to aid in the evaluation of water for acidity or causticity. This procedure is applicable for sites where moderately low or high pH water is expected.

## Equipment

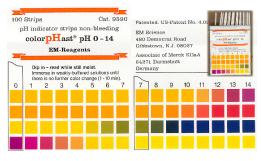
- Disposable chemical-resistant gloves,
- Sterile container or clean zip top bag,
- Corning Checkmate M90<sup>™</sup> equipped with pH sensor,
- Corning Checkmate M90<sup>™</sup> pH buffers (4, 7, and 10 standard units),
- ColorPHast® or equivalent litmus strips (to be used if pH meter is not being used or as a check of field equipment), and
- Deionized water.

Note regarding the ColorPHast® or equivalent litmus strips: These litmus strips are commonly available to cover a range of possible pHs. Typical ranges used for this procedure are 0-6, 5-10, and 7.5-14 standard units. Other ranges are also available. Before field mobilization, discuss the anticipated pH range of the water to be tested and mobilize strips for the appropriate range. Strips should be stored in a dry location; it may be necessary to package the strips in a watertight zip top bag or equivalent before mobilization into the field.

## Procedures

- Install the pH sensor onto the Corning Checkmate M90<sup>TM</sup> meter.
  - 1. Locate the pins of the pH sensor and push firmly into the meter.
  - 2. Remove the sensor wetting cap and slide the vent sleeve to expose the fill hole.
- Calibrate the pH meter at least daily in accordance with the manufacturer's specifications. The following procedure will calibrate the Corning Checkmate M90<sup>™</sup> for pH measurements.
  - 1. Place the sensor in the calibrating medium (pH 7 buffer).
  - 2. Press cal cal 1 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.

- 3. Place the sensor in the second calibrating medium (pH 4 or 10 buffer). If samples are anticipated to be acidic, use the pH 4 buffer. If samples are anticipated to be basic, or caustic, use the pH 10 buffer.
- 4. Press cal cal 2 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.
- For each water sample to be screened for pH, place a small amount of sample in a sterile container or clean zip top bag. Place the sensor into the sample. Automatic endpoint detection freezes the display when plateau is reached.
- ColorPHast® or equivalent litmus strips can be used to verify the results of the Corning Checkmate M90<sup>™</sup> meter and to check if the meter requires recalibration. Place a litmus strip in the sample and allow the strip to become fully wetted. A test time of about 30 seconds is usually sufficient. Compare the color of the test strip to the pH indicator chart on the front of the ColorPHast® package. The



precision of the indicator chart will vary depending on the range of the strips used. The example shown indicates that for strips covering the 0-14 standard units range, a precision of 1 standard unit is available. Intermediate pH values may be estimated by interpolating between the colors shown on the chart.

## Documentation

Record the pH of the sample on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

## **Attached Supporting Documentation**

Corning Checkmate M90<sup>TM</sup> (or equivalent) Operating Instructions

Corning Checkmate M90<sup>™</sup> (or equivalent) Sensor Information

# E.540 CONDUCTIVITY FIELD SCREENING - WATER

## Last Revision or Review: June 2010

## **Objective and Application**

To provide a qualitative and limited quantitative field screening of water samples to aid in the evaluation of water for the relative presence of ions in the sample.

## Equipment

- Disposable chemical-resitant gloves,
- Sterile container or clean zip top bag,
- Corning Checkmate M90<sup>TM</sup> (or equivalent) equipped with conductivity sensor,
- Corning Checkmate M90<sup>™</sup> (or equivalent) conductivity standard A or B (1413 uS or 12.88 mS), and
- Deionized water.

## Procedures

- Install the conductivity sensor onto the Corning Checkmate M90<sup>TM</sup> meter.
  - 1. Locate the pins of the conductivity sensor and push firmly into the meter.
  - 2. Remove the sensor wetting cap and slide the vent sleeve to expose the fill hole.
- Calibrate the conductivity meter at least daily in accordance with the manufacturer's specifications. The following procedure will calibrate the Corning Checkmate M90<sup>™</sup> for conductivity measurements.
  - 1. Hold the sensor in free air.
  - 2. Press cal cal 1 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.
  - 3. Place the sensor in the second calibrating medium (conductivity standard A or B).
  - 4. Press cal cal 2 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.
- For each water sample to be screened for conductivity, place a small amount of sample in a sterile container or clean zip top bag. Place the sensor into the sample. Automatic endpoint detection freezes the display when plateau is reached.

#### Documentation

#### **TSOP E.540**

Record the conductivity of the sample on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

## **Attached Supporting Documentation**

Corning Checkmate M90<sup>TM</sup> (or equivalent) Operating Instructions

Corning Checkmate M90<sup>™</sup> (or equivalent) Sensor Information

# E.550 Field Surface Screening – Soil / Photoionization Detector

Last Review or Revision: June 2010

## Objective

- To provide a qualitative and limited quantitative field screening of soil samples to aid in the evaluation of soil for the presence of volatile or semi-volatile organic chemicals.
- The procedure is premised on the physical property of volatile compounds to move from the soil matrix of a freshly obtained soil sample (e.g., split spoon sample, grab sample) to the airborne state as vapor. The measurement of ambient air in close proximity to the surface of the soil will produce an indication of contaminants moving from the soil matrix to air. The relative strength of contaminants in ambient air around the sample will be considered an indication of the relative concentration of chemicals in the soil sample.
- The procedure is semi-quantitative but valid only for the most preliminary qualitative decisionmaking. The procedure is highly susceptible to external air changes in wind velocity, ambient dilution by moving air and ambient influence by contaminants in moving air. The rate and degree of volatilization is susceptible to smearing of the exterior of the sample by the sampler wall in clay soils.
- Measurements cannot be used as the sole indicator of soil contamination or in lieu of prescribed laboratory chemical testing for purposes of regulatory compliance. This procedure is only to be used for sites involving volatile or semi-volatile organic compounds, including tetrachloroethylene (perchloroethylene or PCE). This procedure is not to be used for purposes of health and safety monitoring.

#### Equipment

- Calibration gas from manufacturer
- Photoionization detector equipped with 10.0 eV lamp or greater.
- Forms and indelible ink pen
- Disposable chemical-resistant gloves

#### Procedures

On a daily basis, the unit should be gas calibrated to a manufacturer's gas standard and the results recorded in the field logbook. If the unit does not calibrate, return it to the local Terracon equipment evaluation for evaluation and, if necessary, repair.

Immediately prior to making a field measurement the unit should be operated for approximately 1 minute and any background concentrations noted or zeroed out relative to measurements.

The opened sampler should be placed on disposable plastic material or a surface that can be cleaned between tests to avoid inadvertent contribution of ionizable vapor from previous tests.

Using a clean stainless steel knife or other cutting tool, slice through the sample to expose fresh soil material not smeared by the sampler or excavating equipment. Immediately begin the test procedure as follows.

- Place the tip of the PID probe between ½ and 1 inches from the surface of the exposed soil.
- Maintaining the proper and constant distance of the probe tip from the sample move the probe very slowly over the surface of sample material, giving good coverage to all portions of the sample.
- Total test time should be in excess of 1 minute for volatile chemicals of concern and in excess of 3 minutes for semi-volatile chemicals of concern.
- Record the highest reading obtained as parts per million (ppm) calibration gas equivalents (i.e., TEI580 calibrated to isobutylene would be expressed as ppmi). Record unusual fluctuations and any noticeable physical correlation to the sample (i.e., "highest readings at ½-1 feet below ground surface over a sand seam in the split spoon sample").

## Sample Disposal

Soil samples should be returned to the site and included in auger soil or excavation soil for proper disposal.

#### Documentation

Record the highest reading in calibration gas equivalents on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded; date, job number, project name, sampling location, sample interval (if appropriate) sample identification, samplers name, and general observations.

## Attached Supporting Documents

• Thermo Environmental Instruments, Inc. OVM/Datalogger Model 580B Operating Manual

# E.552 Field Headspace Screening – Soil / Photoionization Detector

## LAST REVIEW OR REVISION: June 2010

## OBJECTIVE

- To provide a qualitative and limited quantitative field screening of soil samples to aid in the evaluation of soil for the presence of volatile or semi-volatile organic chemicals.
- The procedure is premised on the physical property of volatile compounds to move from the soil matrix to the airborne state as vapor. The amount of airborne material as vapor will be a relative concentration between samples if the volume of sample, volume of air, temperature and period of testing remain reasonably constant. The measurement is semi-quantitative for qualitative decision-making.
- Measurements cannot be used as the sole indicator of soil contamination or in lieu of prescribed laboratory chemical testing for purposes of regulatory compliance. This procedure is only to be used for sites involving volatile organic compounds, including tetrachloroethylene (perchloroethylene or PCE). This procedure is not to be used for purposes of health and safety monitoring.

#### EQUIPMENT

- Calibration gas from manufacturer
- Photoionization detector equipped with 10.0 eV lamp or greater.
- Test chamber, reusable or disposable, examples:
  - Mason jar with aluminum foil
  - Mason jar equipped with charcoal filter
  - Ziploc® bags or other sealable container of at least 500 cubic centimeters to provide a fixed headspace volume for constancy between tests
  - Rigid, disposable concrete test cylinder mold with plastic cap
- Forms and indelible ink pen
- Disposable chemical-resistant gloves

#### PROCEDURES

On a daily basis, the unit should be gas calibrated to a manufacturer's gas standard and the results recorded in the field logbook. If the unit does not calibrate, return it to the local Terracon equipment evaluation for evaluation and, if necessary, repair.

Immediately prior to making a field measurement the unit should be operated for approximately 1 minute and any background concentrations noted or zeroed out relative to measurements.

Prior to site testing an empty, unused test chamber should be sealed containing nothing but ambient air and allowed to stabilize for 1 minute. Test the chamber headspace to identify background contaminants contributed by the chamber itself. If anything is detected, change to another type of chamber which is inert relative to contributing ionizable materials to the headspace.

- Transfer soil sample representative of the condition to be measured from the sampling device to testing chamber (e.g., mason jar covered by aluminum foil, disposal concrete cylinder mold, Ziploc® bag, or other sealable container).
- Sample material should be representative of the vertical and horizontal cross-section of the sampled interval.
- The volume of soil should remain as constant as is practical for all site tests.
- The soil volume should not exceed 25% of the total volume of the air-filled testing chamber.
- Immediately seal the chamber after transfer and allow the sample to equilibrate for a minimum of 15 minutes at ambient temperatures above 50° F.
- Insert probe into sealed container for reading for 1 minute for volatile compounds of concern and 3 minutes for semi-volatile compounds of concern to account for varied response times.
- Record highest reading obtained as parts per million (ppm) calibration gas equivalents (i.e., TEI580 calibrated to isobutylene would be expressed as ppmi).

## SAMPLE DISPOSAL

Soil samples should be returned to the site and included in auger soil or excavation soil for proper disposal.

## DOCUMENTATION

Record the highest reading in calibration gas equivalents on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded; date, job number, project name, sampling location, sample interval (if appropriate) sample identification, samplers name, and general observations.

## OTHER SUPPORTING DOCUMENTS

• Thermo Environmental Instruments, Inc. OVM/Datalogger Model 580B Operating Manual

# E.554 Field Screening – Air / Photoionization Detector

## LAST REVIEW OR REVISION: June 2010

## **OBJECTIVE AND APPLICATION**

- To provide a qualitative and limited quantitative field screening of ambient air to aid in the evaluation of air quality relative to monitoring health and safety of workers involved in environmental field activities involving known or potential volatile or semi-volatile organic chemicals of concern.
- The procedure is premised on the physical property of volatile compounds to move to the airborne state as vapor. The amount of airborne material as vapor will be a relative concentration dependent on air currents and dilution. The measurement is semi-quantitative for qualitative decision-making.
- The intent is to monitor airborne concentrations of chemicals of concern in the breathing zone of workers. Unless otherwise specified by the health and safety plan, the breathing zone will be considered the height above ground surface generally corresponding from knees-to-chin to provide for upright, squatting and bending during field activity by workers.
- This procedure is to be used primarily for purposes of health and safety monitoring. These readings <u>are not</u> a representative screening indication of the levels of chemicals of concern in soils or other media.
- This procedure, though it may be used in combination with, <u>does not</u> replace similar methods and equipment used for confined space entry procedures. Use of this procedure as part of confined space entry testing <u>must</u> be discussed with the Corporate Health and Safety Manager and approved <u>prior</u> to such use.

#### EQUIPMENT

Equipment will only be operated by personnel trained and qualified as competent on use of the machine prior to field use and under the direction of an experienced operator. The following equipment will be used.

- Calibration gas from manufacturer.
- Photoionization detector equipped with 10.0 eV lamp or greater.
- Charger unit and source of 110v power when needed, not in the form of a generator.
- Forms and indelible ink pen
- Disposable chemical-resistant gloves

#### PROCEDURES

On a daily basis, the unit should be gas calibrated to a manufacturer's gas standard and the results recorded in the field logbook. If the unit does not calibrate, return it to the local Terracon equipment evaluation for evaluation and, if necessary, repair.

Immediately prior to making a field measurement the unit should be operated for approximately 1 minute remote from activities engaged in sampling or disturbing contaminants. Any background concentrations should be noted or zeroed out relative to test measurements.

Readings will be taken in the breathing zone. Measurements can be taken continuously unattended (i.e., attached to the drill rig or excavator during equipment operations) or on an attended periodic basis specified by the project manager.

For continuous unattended readings the unit will placed secure from damage or interference from machinery exhausts at an elevation consistent with the breathing zone and in close proximity to workers. The unit will be operated in a mode which will record the maximum reading without having to continuously view the readout. The unit will be operated in alarm mode set at the first response level specified in the project health and safety plan. The maximum will be checked periodically by the operator, but on intervals never exceeding 30 minutes.

For periodic attended readings the unit will be handheld. The operator will hold the unit probe in the breathing zone, moving slowly through the zone in close proximity to workers without interfering with their activity. The reading will be made for no less than 3 minutes to account for response times of ionizable semi-volatile compounds of concern to account for varied response times.

Record readings obtained as parts per million (ppm) calibration gas equivalents (i.e., TEI580 calibrated to isobutylene would be expressed as ppmi).

## DOCUMENTATION

Record readings in calibration gas equivalents on the Acknowledgment Of Instruction Form of the project health and safety plan. Alternatively, they may be recorded on forms or the project field log book provided by the Project Manager. Copies of these records will be attached to and filed with the file copy of the health and safety plan.

Readings will clearly be recorded and labeled as separate from readings made for the purpose of field screening soils for environmental impacts.

## **OTHER SUPPORTING DOCUMENTS**

• Thermo Environmental Instruments, Inc. OVM/Datalogger Model 580B Operating Manual

# E.560 SVOC Field Screening – Soil / Ultraviolet Fluorescence

## LAST REVIEW OR REVISION: June 2010

## **OBJECTIVE AND APPLICATION**

To provide a qualitative field screening of soil samples to aid in the evaluation of soil for the presence of semi-volatile organic compounds.

The procedure is premised on the physical property of long-chain hydrocarbons in petroleum materials to be reactive through fluorescence in response to mild radiation as ultraviolet light. The screening has it's basis in oil field petrology where core samples are bombarded with ultraviolet light to observe for fluorescence caused by the presence of crude oils not readily visible to the human eye.

The procedure is qualitative and sensitive to the ability of the operator to visually discern, describe and record relative degrees and amounts of relative fluorescence.

Measurements should consider the presence of naturally fluorescing minerals and other natural materials found in soil matrices.

Measurements cannot be used as the sole indicator of soil contamination or in lieu of prescribed laboratory chemical testing for purposes of regulatory compliance. This procedure is only to be used for sites involving semi-volatile organic compounds. This procedure <u>is not</u> to be used for purposes of health and safety monitoring.

## EQUIPMENT

Equipment will only be operated by personnel trained and qualified as competent on use of the machine prior to field use and under the direction of an experienced operator.

- Chromato-vue CC-60 Ultraviolet Illumination System (fluorometer) or equivalent equipped with mid- and high-range ultraviolet spectrum lamps.
- Commercial grade trichloroethylene in a low volume, low pressure handheld spray bottle (optional)
- Forms and indelible ink pen
- Disposable chemical-resistant gloves

## PROCEDURES

- Place an aliquot of the soil sample to be screened in the viewing chamber.
- Using the switch on the side of the viewing chamber to change wavelengths, view the sample under ultraviolet light at wavelengths of 254 and 365 nanometers.
- Qualitatively record the relative distribution and intensity of observable fluorescence in the sample viewed under each wavelength. Record the intensity of the fluorescence according to the following scale:
  - FR 0 Not Visible by the observer except as random "pinpoints" of fluorescence resulting minerals in the soil.
  - FR 1 Slight Fluorescence, faint fluorescence distributed visually without distinct pattern of segregation or generally over less than 1% of the exposed sample area viewed.
  - FR 2 Fluorescent, faint to moderate fluorescence distributed visually with distinct pattern of segregation or generally over more than 5% of the exposed sample area viewed.
  - FR 3 Highly Fluorescent, Fluorescent, strong and definitive fluorescence distributed visually with distinct pattern of segregation or generally over more than 10% of the exposed sample area viewed.
- If directed by the project manager, also record the visually estimated area of the sample surface which fluoresces relative to the total sample exposed as a percentage.
- If specified by the Project Manager, moisten the sample with a mist of trichloroethylene solvent and view a second time, again recording the relative distribution and intensity of fluorescence.

## SAMPLE DISPOSAL

Soil samples should be returned to the site and included in auger soil or excavation soil for proper disposal. If the trichloroethylene option is used, samples may require disposal as waste. Consult with your project manager before using the trichloroethylene option.

## DOCUMENTATION

#### TSOP E.560

Record the intensity and fluorescence readings on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

#### **OTHER SUPPORTING DOCUMENTS**

• Chromato-vue CC-60 Ultraviolet Illumination System, Operations Manual, UVP, Inc.

#### STANDARD OPERATING PROCEDURE

## E.570 TEMPERATURE FIELD SCREENING - WATER

#### Last Revision or Review: June 2010

#### **Objective and Application**

To provide a qualitative and limited quantitative field screening of water samples to aid in the evaluation of water temperature.

#### Equipment

- Disposable chemical-resistant gloves,
- Thermometer,
- Sterile container or zip top bag, and
- Deionized water.

#### Procedures

The sample containment area of the temperature meter or the measuring end of the thermometer should be cleaned with deionized water before each use.

As the temperature of the water sample will begin to normalize to ambient temperature as soon as the sample is removed from its point of origin (monitoring well, stream, etc.), temperature should be the first field measurement obtained. For each water sample to be tested, place a minimum of 100 milliliters (mL) of sample in the sterile container or zip top bag. If less than 100 mL is used, the temperature of the water sample may not stabilize prior to reaching ambient temperatures. Insert the measuring end of thermometer into the water sample. The thermometer should not rest against the sides of the sterile container while taking the measurement. Allow the thermometer readings to stabilize, and record the final temperature.

#### Documentation

Record the temperature of the sample on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

#### Other Supporting Documentation

**ASTM E1** Standard Specification for ASTM Thermometers.

#### STANDARD OPERATING PROCEDURE

## E.580 TURBIDITY FIELD SCREENING - WATER

#### Last Revision or Review: June 2010

#### **Objective and Application**

To provide a qualitative and limited quantitative field screening of water samples to aid in the evaluation of water for turbidity, or the presence of visible suspended solids.

#### Equipment

- Disposable chmical-resistant gloves.
- LaMotte Turbidimeter Model 2008 or equivalent.
- Deionized water.

#### Procedures

Before testing water samples, calibrate the turbidity meter according to the manufacturer's recommendation. Calibration should occur as often as specified by the Project Manager, but at a minimum should be conducted at the manufacturers specified intervals, and at a minimum on a daily basis. The specialized sample containers supplied with the meter should be cleaned with deionized water prior to each use.

For each water sample to be tested, place a small amount of sample in the sample container supplied with the turbidity meter. Agitate the sample if needed to suspend any settled solids. Allow the turbidity readings to stabilize. Warning – do not allow the sample to remain still long enough for suspended solids to begin settling out of suspension; this could result in the meter underestimating the turbidity of the sample. Record the turbidity measured by the meter.

#### Documentation

Record the turbidity of the sample on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

#### **Attached Supporting Information**

LaMotte Turbidimeter Model 2008 Operating Manual

Understanding Turbidity Measurement, HACH Technical Center for Applied Analytical Chemistry

#### **Other Supporting Information**

ASTM D1889-00 Standard Test Method for Turbidity of Water

## E.590

# AIRBORNE LEAD AND PARTICULATE MATTER MONITORING SAMPLE COLLECTION

#### Last Revision or Review: June 2010

#### **Objective and Application**

The following procedure is for collection of samples for measurement of total airborne particulate matter and airborne lead particulate. The methods will provide representative samples for laboratory analysis using NIOSH Method 7300.

#### Equipment

The following equipment and supplies will be needed for this testing:

- Air monitoring cassettes containing mixed cellulose ester filters with a 0.8 µm pore size pre-weighed and numbered by the laboratory.
- Rotometer or other flowmeter accurate to the 0.1 L/min. Flowmeters used for calibration of air sample pumps will be calibrated against a primary standard on a quarterly basis.
- Air monitoring pump (battery operated or ac powered depending on application) capable of provided a 1 to 4 L/min flow rate through the sample filter.
- Vinyl tubing to connect pump to cassette.
- Cassette stand or other means to fix cassette in place as appropriate to testing location.
- Chain of Custody.

#### Procedures

Select a location for sample that is subject to full ambient air flow but not subject to vibration that could dislodge particulates from the surface of the filter. A location protected from precipitation should be selected, if available. Excessive moisture will interfere with sample analysis and could effect the flow rate.

Label cassette and record laboratory identification number on chain of custody. Assemble sampling pump, tubing and stand in sample collection location. The base of the cassette should be a press fit into the interior of the tubing. The joint between the tubing and cassette may be reinforced with tape if site conditions make this necessary. Cassette must be tilted downward at a 45° angle from horizontal. Cassette and pump should be placed so that the sample does not have to be moved after sample calibration.

A short section of vinyl tubing should be used to connect the flowmeter to the inlet port of the cassette. An adapter fitting may be needed to connect the tubing to the cassette. If a rotometer

#### **TSOP E.590**

is used for flow calibration, the instrument must be held upright for accurate readings. Turn on the pump and measure the flow rate. Using the flow control on the pump, adjust the flow to the desired reading between 1.0 and 4.0 L/min. Record the sampling start time and beginning flow rate.

After completion of the sample collection, inspect the pump and cassette for any problems that may affect the sample validity (wet filter, stand blown over, etc.) and record any problems noted. Measure the flow rate using the procedure described above. The average of the two flow rates will be used by the laboratory to calculate the sample volume. Turn off the pump and cap the inlet and outlet ports on the filter cassette. Record the sampling completion time and ending flow rate.

#### Documentation

In addition to chain of custody documentation, the following must be provided to the laboratory performing sample analysis:

- Average flow rate (average of beginning and ending readings)
- Sample duration
- Filter number
- Requested analytical method

The following additional information must be collected for Terracon's project files:

- Sample date
- Sample times
- Sample location
- Activities performed by during the sampling
- Wind orientation (upwind, downwind, etc.)
- Weather conditions

#### Attached Supporting Information

- Industrial Hygiene Technical Manual Chapter II: Standard Methods for Sampling Air Contaminants
- NIOSH Method 7300 Elements by ICP

# E.600 H<sub>2</sub>S FIELD SCREENING – FIELD DETECTOR

#### Last Revision or Review: June 2010

#### Objective

To provide a qualitative and limited quantitative field screening of soil samples to aid in the evaluation of soil for the presence of hydrogen sulfide vapors.

#### Equipment

- Monitoring equipment
- Hydrogen sulfide field detector.
- Monitoring vessel
  - Mason jar with aluminum foil Mason jar equipped with charcoal filter
  - zip top bags
- Forms and indelible ink pen
- Disposable chemical-resistant gloves

#### Procedures

- a) Backhoe
  - Evaluate soil in bucket of backhoe as it is removed from excavation
  - Remove surficial soil and expose new soil surface may want to create a depression using your gloved hand.
  - Insert probe into depression or within 1/2 1 inch of surface.
  - Allow probe to stabilize if possible may want to record range due to large fluctuations.
  - If soil is cohesive (silt & clay), break apart and expose the probe to fresh surface.
- b) Split Barrel Sampler/CHSS or other field extractable samplers
  - Insert or wave probe over exposed soil collected using split barrel sampler immediately upon recovery.
  - If soil is cohesive, break apart and expose the probe to fresh surface.
  - Check observable sand seams and wherever there is a change in strata.
- c) Specific (pseudo quantitative)
  - Transfer soil sample from sampling device to testing chamber (mason jar, aluminum foil covered, zip top bag).
  - Insert probe into ziptop bag or mason jar, through aluminum foil, or attach to modified mason jar for reading
  - Record highest reading obtained.

### Sample Disposal

Soil samples should be returned to the site and included in auger soil or excavation soil for proper disposal.

#### Documentation

Record the highest reading obtained on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

#### STANDARD OPERATING PROCEDURE

## E.605 METHANE MONITORING – FIELD DETECTOR

Last Review or Revision: June 2010

#### Objective

To provide a qualitative and limited quantitative field screening of soil samples to aid in the evaluation of soil for the presence of methane.

#### Equipment

- Monitoring equipment
- Methane gas field detector.
- Monitoring vessel
  - Mason jar with aluminum foil
  - Mason jar equipped with charcoal filter

Ziptop bags

- Forms and indelible ink pen
- Disposable chemical-resistant gloves

#### Procedures

- a) Backhoe
  - Evaluate soil in bucket of backhoe as it is removed from excavation
  - Remove surficial soil and expose new soil surface may want to create a depression using your gloved hand.
  - Insert probe into depression or within 1/2 1 inch of surface.
  - Allow probe to stabilize if possible may want to record range due to large fluctuations.
  - If soil is cohesive (silt & clay), break apart and expose the probe to fresh surface.
- b) Split Barrel Sampler/CHSS or other field extractable samplers
  - Insert or wave probe over exposed soil collected using split barrel sampler immediately upon recovery.

- If soil is cohesive, break apart and expose the probe to fresh surface.
- Check observable sand seams and wherever there is a change in strata.
- c) Specific (pseudo quantitative)
  - Transfer soil sample from sampling device to testing chamber (mason jar, aluminum foil covered, ziptop bag).
  - Insert probe into ziptop bag or mason jar, through aluminum foil, or attach to modified mason jar for reading
  - Record highest reading obtained.

#### Sample Disposal

Soil samples should be returned to the site and included in auger soil or excavation soil for proper disposal.

#### Documentation

Record the highest reading obtained on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

#### STANDARD OPERATING PROCEDURE

# E.610 RADIOACTIVITY – FIELD DETECTOR

#### Last Revision or Review: June 2010

#### **Objective & Application:** To provide a quantitative field screening for low-level radiation.

Use of the radioactivity field detector will be required where the potential exists for low-level radiation to be present at a project site. If radioactive waste may be present at the project site, the Project Manager, in conjunction with the Terracon Corporate Health and Safety Manager, will develop site-specific procedures. Terracon personnel conducting field testing for radioactive materials require <u>special radiological safety and nuclear gauge training</u>. Once a site has been "cleared" for the potential of radiation hazards, non-trained personnel will be allowed on-site. The unit will only be calibrated and used by Terracon technicians certified with training in use of nuclear testing equipment, both gauge operation/calibration and radiological safety. Training will be to requirements of 49CFR172, Subpart H.

#### Equipment:

- Victoreen Model 190 Survey Meter or equivalent
- Marking tape or stakes
- Measuring wheel or tape

#### **Procedures:**

Grid the area to be screened into quadrants. Use the measuring device to locate the grid nodes, and mark the area using tape or stakes.

Collect radiation readings at several monitoring points within each quadrant.

Collect radiation readings from the ground surface to a height of approximately one (1) meter above the ground. For comparison, background radiation readings should also be obtained in a similar manner from at least three (3) monitoring locations near the project site.

#### **Documentation:**

Record pertinent data in the field log book or on the supplied forms. Pertinent data will vary based on the project; however, the following data must always be recorded; time, date, job number, project name, sampling location, samplers name, and general observations.

# E.620 POLYCHLORINATED BIPHENYL FIELD SCREENING – CLOR-N-OIL FIELD DETECTOR

Last Review or Revision: June 2010

#### Objective

To provide a qualitative and limited quantitative field screening of electrical transformer oils to aid in the evaluation of the oil for the presence of polychlorinated biphenyls (PCBs).

#### Equipment

- Clor-N-Oil PCB Screening Kit or equivalent
- Disposable chemical-resistant gloves

#### Procedures

- Remove contents of Clor-N-Oil PCB Screening kit. Check contents to ensure that all items are present and intact. Place the two (2) plastic tubes into the holder at the front of the box.
- Unscrew the black dispenser cap from Tube #1. Using the plastic pipette, transfer exactly 10 ml (up to the black line) of transformer oil to be tested into the tube. Replace the black dispenser cap securely.
- Break the bottom (colorless) ampule in the tube by compressing the sides of the tub. Mix thoroughly by shaking the tube vigorously for 10 seconds. (Make sure that the colorless ampule is broken first, the gray one second). Allow the reaction to proceed for an additional 50 seconds (total of one minute), while shaking intermittently several times.
- Remove the caps from both tubes and pour the clear buffer solution from Tube #2 (white cap) into Tube #1. Replace the black cap tightly on Tub #1 and shake vigorously for 10 seconds. Vent the tube by partially unscrewing the dispenser cap. Close securely and shake well for an additional 10 seconds. Vent again, tighten cap, and stand tube upside down on its cap. The oil mixture should no longer appear gray. Allow the phases to separate for a full two minutes. If the oil layer is below the buffer layer, discontinue the test at this point as the oil is primarily pure PCB (Askarel). If the oil layer is above the water layer, continue the test.
- Place the plastic filtration funnel into Tube #2. Position Tube #1 over funnel and open nozzle on dispenser cap. Dispense 5 mls of the clear solution through the filter into Tube #2 (up to the 5 ml line) by squeezing the sides of Tube #1. Do not allow any of the oil to pass through the filter. Close the nozzle on the dispenser cap on Tube #1 and remove the filter funnel from

Tube #2. Replace the cap on tube #2 Break the bottom (colorless) ampule and shake for 10 seconds. Break the top (colored) ampule and shake for 10 seconds.

 Observe the resultant color immediately and compare to the color chart below for chlorine determination. If the solution appears purple, the oil sample contains less than 20 ppm PCB. If it appears yellow or colorless, it MAY contain more than 20 ppm PBC and should be tested further by a PCB specific method. Disregard any color that may develop in a thin layer of oil that might form on tip of the solution.

#### Sample Disposal

 Open the "Disposal Ampule" container and drop the ampule into Tube #2. Replace the cap on the test tube. Crush the ampule by squeezing the sides of the tube. Shake for five (5) seconds. This reagent immobilizes the mercury so that the kit passes the EPA's TCLP test. If the test indicates that the oil does not contain PCBs, the kit may be disposed as regular waste. If the test indicates that the oil contains PCBs, check with the Project Manager for proper disposal in accordance with local regulations.

#### Attached Supporting Documents

• Instructions for Clor-N-Oil 20 PCB Screening Kit, Dexsil Corporation.

#### Documentation

Record the results of the colormetric test on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, test results, and general observations.

# E.630 X-RAY FLUORESCENCE (XRF) FIELD MEASUREMENT FOR AIRBORNE DUST

#### Last Revision or Review: June 2010

**Objective & Application:** To provide a quantitative field screening of airborne dust for the presence of inorganic metals and other relevant compounds within the capabilities of the commercial unit known as Spectrace9000. XRF technology has been evaluated by the USEPA, which has developed an operating procedure for their Emergency Response section. That guidance was used in developing this TSOP.

The unit is suitable for field analysis in the hands of trained technical staff. Terracon use of the unit requires <u>special radiological safety and nuclear gauge training</u>. The unit will only be calibrated and used by Terracon technicians certified with training in use of nuclear testing equipment, both gauge operation/calibration and radiological safety. Training will be to requirements of 49CFR172, Subpart H.

#### Equipment:

- Spectrace 9000 Portable XRF Analyzer or equivalent
- Thin filter media (for sample collection/viewing/testing)
- Stable platform and reliable 110v power source for temporary laboratory setting
- Spare battery pack for mobile field use

#### **Procedures:**

- Connect the battery. To connect the battery, set the electronics unit on its face and use the flat blade screwdriver provided to loosen the two ¼-turn fasteners on the back. Remove the battery pack. Inside, find the cord with the red cap (it covers a three-pronged plug). Remove the cap and plug it into the battery pack. Put the pack back into the unit-it can only fit one way. Tighten the fasteners.
- Carefully plug the right angle end of the probe cable into the electronics unit and the other end into the probe handle.
- Press the ON key. In two or three seconds, the display announces the version number of the software. Set the correct date and time according to the manufacturer's instructions.

#### **Field Lab Screening**

It is often preferable to establish a field laboratory during environmental explorations. This allows the unit to be used to screen multiple samples as part of other sampling activities. The platform should be stable, clean and protected from weather. Mobile vehicles and field vans are acceptable. A source of 110v power is preferred <u>– DO NOT use the unit with a field generator</u>, power fluctuations can severely damage or destroy the electronics of the test unit.

- Prior to collecting dust samples onto the sterile filter media, screen each clean filter with the Spectrace9000 or equivalent prior to sample collection. This will establish a baseline metals concentration for each filter prior to sampling.
- Air samples obtained in the field will be collected by impacting airborne dust onto sterile filter media per instructions from the Project Manager. Place the filter media containing the sample onto the probe for analysis.
- Download the test results to a laptop computer as directed by the manufacturer in the operating manual.

#### Documentation:

Record pertinent data in the field log book or on the supplied forms. Pertinent data will vary based on the project; however, the following data must always be recorded; time, date, job number, project name, sampling location, samplers name, and general observations.

#### **Attached Supporting Documents:**

Spectrace 9000 Portable XRF Analyzer Operations Manual, provided by manufacturer.

SPECTRACE 9000 Field Portable X-Ray Fluorescence Operating Procedures, EPA Emergency Response Team (EPA/ERT) SOP#1713, 1995.

# E.634 X-RAY FLUORESCENCE (XRF) FIELD MEASUREMENT FOR LEAD PAINT

#### Last Revision or Review: June 2010

**Objective & Application:** To provide a quantitative field screening of painted surfaces to aid in the evaluation of the paint for the presence of lead and other relevant compounds within the capabilities of the commercial unit known as Spectrace9000 or equivalent. XRF technology has been evaluated by the USEPA, which has developed an operating procedure for their Emergency Response section. That guidance was used in developing this TSOP.

The unit is suitable for field analysis in the hands of trained technical staff. Terracon use of the unit requires <u>special radiological safety and nuclear gauge training</u>. The unit will only be calibrated and used by Terracon technicians certified with training in use of nuclear testing equipment, both gauge operation/calibration and radiological safety. Training will be to requirements of 49CFR172, Subpart H.

#### Equipment:

- Spectrace 9000 Portable XRF Analyzer or equivalent
- Stable platform and reliable 110v power source for temporary laboratory setting
- Spare battery pack for mobile field use
- Tamper/leveler for use in preparing soil/fill for in-situ measurements
- Ziploc® disposable plastic bags for storing ex-situ samples in temporary laboratory setting

#### **Procedures:**

- Connect the battery. To connect the battery, set the electronics unit on its face and use the flat blade screwdriver provided to loosen the two ¼-turn fasteners on the back. Remove the battery pack. Inside, find the cord with the red cap (it covers a three-pronged plug). Remove the cap and plug it into the battery pack. Put the pack back into the unit-it can only fit one way. Tighten the fasteners.
- Carefully plug the right angle end of the probe cable into the electronics unit and the other end into the probe handle.
- Press the ON key. In two or three seconds, the display announces the version number of the software. Set the correct date and time according to the manufacturer's instructions.
- Ideally, the sample should be flat, larger than one inch in diameter, and should be placed in contact with the face of the probe. Most times, it is easier to take the analyzer to the sample (in-situ) than it is to bring the sample to the analyzer (ex-situ).

#### **TSOP E.634**

• To measure the lead concentrations in the area to be sampled, place the probe end over the sample area, keeping the sample window in contact with the sample media. Push the CONT or probe button to take a measurement. Depending on the instrument settings, the test will run for thirty to two hundred (30-200) seconds.

#### **Documentation:**

Record pertinent data in the field log book or on the supplied forms. Pertinent data will vary based on the project; however, the following data must always be recorded; time, date, job number, project name, sampling location, samplers name, and general observations.

#### Other Supporting Documents (See E.630 for reproduction):

Spectrace 9000 Portable XRF Analyzer Operations Manual, provided by manufacturer.

SPECTRACE 9000 Field Portable X-Ray Fluorescence Operating Procedures, EPA Emergency Response Team (EPA/ERT) SOP#1713, 1995.

# E.638 X-RAY FLUORESCENCE (XRF) FIELD MEASUREMENT FOR SOILS/FILLS

#### Last Revision or Review: June 2010

**Objective & Application:** To provide a quantitative field screening of soil samples to aid in the evaluation of soils/fills for the presence of inorganic metals and other relevant compounds within the capabilities of the commercial unit known as Spectrace9000 or equivalent. The unit can be used to test in-situ surface materials or to test representative surface or subsurface samples in a temporary laboratory setting. XRF technology has been evaluated by the USEPA, which has developed an operating procedure for their Emergency Response section. That guidance was used in developing this TSOP.

The unit is suitable for field analysis in the hands of trained technical staff. Terracon use of the unit requires <u>special radiological safety and nuclear gauge training</u>. The unit will only be calibrated and used by Terracon technicians certified with training in use of nuclear testing equipment, both gauge operation/calibration and radiological safety. Training will be to requirements of 49CFR172, Subpart H.

#### Equipment:

- Spectrace 9000 Portable XRF Analyzer or equivalent
- Sample cups (for lab viewing/testing)
- Mylar film (for lab viewing/testing)
- Stable platform and reliable 110v power source for temporary laboratory setting
- Spare battery pack for mobile field use
- Tamper/leveler for use in preparing soil/fill for in-situ measurements
- Ziploc® disposable plastic bags for storing ex-situ samples in temporary laboratory setting
- Disposable chemical-resistant gloves

#### **Procedures:**

- Connect the battery. To connect the battery, set the electronics unit on its face and use the flat blade screwdriver provided to loosen the two ¼-turn fasteners on the back. Remove the battery pack. Inside, find the cord with the red cap (it covers a three-pronged plug). Remove the cap and plug it into the battery pack. Put the pack back into the unit-it can only fit one way. Tighten the fasteners.
- Carefully plug the right angle end of the probe cable into the electronics unit and the other end into the probe handle.

#### **TSOP E.638**

• Press the ON key. In two or three seconds, the display announces the version number of the software. Set the correct date and time according to the manufacturer's instructions.

#### Field Screening (In-Situ)

- For screening in-situ samples in the field, the sample cups and mylar film are not required. Ideally, the sample should be flat, larger than one inch in diameter, and should be placed in contact with the face of the probe. Most times, it is easier to take the analyzer to the sample (in-situ) than it is to bring the sample to the analyzer (ex-situ).
- Best results are obtained on reasonably dry, flat, compacted surfaces of fine-grained soils. Whenever possible, flatten and compact the area to be measured an appropriate decontaminated tool. In-situ density does not overly affect the reading, it is permissible to tamp the area flat with a leveling tool.
- Good results can be obtained at moisture contents up to about 25%. Coarse-grained soil conditions may not permit a truly representative sample and may adversely affect the analytical results. Such samples should be prepared before analysis (ex-situ).
- To measure the metals concentrations in the area to be sampled, place the probe end over the sample area, keeping the sample window in contact with the sample media. Push the CONT or probe button to take a measurement. Depending on the soil matrix and the measurements being taken, the test will run for thirty to two hundred (30-200) seconds.

#### Field Lab Screening (Ex-Situ)

It is often preferable to establish a field laboratory during environmental explorations. This allows the unit to be used to screen multiple samples as part of other drilling and sampling activities. The platform should be stable, clean and protected from weather. Mobile vehicles and field vans are acceptable. A source of 110v power is preferred <u>– DO NOT use the unit with a field generator</u>, power fluctuations can severely damage or destroy the electronics of the test unit.

- Samples obtained in the field will require transfer to the sample cups to prior to laboratory measurement with the unit. To load a sample cup, fill the cup nearly full with the sample. If the sample is powdered, tap the cup a little to settle the contents and, if necessary, add more sample until the cup is at least <sup>3</sup>/<sub>4</sub> full. Tear off a square piece of the mylar film. Lay it over the open cup approximately centered. Examine the sample cup ring. One inside edge is rather sharp and the other inside edge is smooth and rounded. Place the ring over the cup with the rounded edge down. Press the ring down slowly and evenly until it is flush with the end of the cup. The ring will slide down and snap into position leaving the film taut and wrinkle-free. If wrinkles do occur, they can be removed by pulling on the excess film or starting over with a new piece of film.
- Turn the filled and sealed sample cup upside down and tap the cup on the bench to thoroughly settle and compact the contents.

- The sample is now ready to be placed film side down on the probe for analysis. The 30mm adapter ring should be placed in the large hole n the shield cup base. This adapter ring locates the sample cup reproducibly over the probe aperture.
- To measure the metals concentrations in the area to be sampled, place the probe end over the sample area, keeping the sample window in contact with the sample media. Push the CONT or probe button to take a measurement. Depending on the soil matrix and the measurements being taken, the test will run for thirty to two hundred (30-200) seconds.
- Download the test results to a laptop computer as directed by the manufacturer in the operating manual.

#### **Documentation:**

Record pertinent data in the field log book or on the supplied forms. Pertinent data will vary based on the project; however, the following data must always be recorded; time, date, job number, project name, sampling location, samplers name, and general observations.

#### Other Supporting Documents (See E.630 for reproduction):

Spectrace 9000 Portable XRF Analyzer Operations Manual, provided by manufacturer.

SPECTRACE 9000 Field Portable X-Ray Fluorescence Operating Procedures, EPA Emergency Response Team (EPA/ERT) SOP#1713, 1995.

#### STANDARD OPERATING PROCEDURE

## E.700 WELL CONSTRUCTION - TEMPORARY

#### Last Review or Revision: June 2010

#### Objective

To provide standard procedures for the design and installation of temporary monitoring wells that will result in reliable construction and provide for the collection of representative groundwater samples.

#### Background

The standard practice for design and installation of monitoring wells is contained in ASTM D 5092 -90 "Design and Installation of Groundwater Monitoring Wells in Aquifers." Terracon procedures incorporate the provisions of ASTM D 5092 that describe the specific procedures to be used in installing monitoring wells in unconsolidated formations for subsurface characterization of sites. The ASTM standard should be referred to when installation of monitoring wells is required outside of the temporary monitoring wells described here.

#### Equipment

The augers required for 2-inch diameter monitoring wells are 4-1/4-inch inside diameter hollow stem augers. For 4-inch wells the 6-1/4-inch inside diameter hollow stem augers are recommended. Flight augers are not recommended for installation of monitoring wells unless aquifer conditions are well known and will allow installation of screen and riser pipe without formation cave-in.

#### **Monitoring Well Material**

- a) Riser Pipe
  - The riser pipe used is threaded, flush jointed Tri-Loc (or equivalent) Schedule 40 PVC pipe. This riser pipe has been found to provide a reliable and durable well.
- b) Screen
  - Slot openings generally used for monitoring wells are 0.010 and 0.020 inch. The slot opening commonly used is 0.010 which will be effective with the use of gravel pack #20-40. If a larger slot opening is desired (.020), a gravel pack of #10-20 is recommended.

#### c) Gravel Pack

The standard gravel pack used is graded as #20-40 which is suited for use with a slot size of .010. Gravel pack should extend approximately two feet above the slotted screen. Gravel pack grading should be specified by the Project Manager or the Drilling Coordinator of the NE Division.

d) Bentonite

Bentonite pellets (1/4-inch size) are used to provide a relatively impermeable layer above the screen and gravel pack. One 5-gallon bucket of bentonite pellets should provide a two foot layer in the annulus of a boring created with 4 1/4-inch inside diameter augers and containing 2-inch monitoring well pipe.

e) Surface Security Materials

The well is secured with an expansion cap utilizing a keyed alike lock. A commercial flush mount protector or above-grade metal protector is used based on surface use conditions or client requirements.

#### Procedures

a) Background

Personnel involved in drilling operations must be familiar with the drilling procedures in ASTM D 5092. The Project Manager will specify the sampling interval, decontamination requirements, screen slot size, gravel pack size, and completion and survey requirements.

b) Mobilization

The project manager will prepare the mobilization sheets for the required drilling operation. The mobilization sheets should summarize types and quantities of well materials, drilling supplies, appropriate equipment, boring depths and boring locations. The enclosed tables should be referred to for calculating the volumes of gravel pack, bentonite, and grout required.

c) Field Operations

The field crews will review the mobilization sheets for adequacy. Unless otherwise specified the field crew will determine the specific use of equipment such as grout trailer, ready mix etc. The field crews will complete the monitoring wells as specified and complete forms for describing boring logs and well details. Any deviation from standard procedures or unusual observations should be noted by the field crews and reported to the project manager.

d) Well Security

The well is secured in accordance with the project manager's instructions and TSOPs E.900, E.905, E.910, or E.920. The proper well security materials to be used in completing the top of a monitoring well should be specified by the project manager and based on the expected ground surface use, regulatory requirements, and client desires. Care should be taken in installation of monitoring wells that will be subjected to vehicle traffic.

e) Well Documentation.

The well will be plugged and abandoned in accordance with TSOP E.1700.

e) Plugging and Abandonment.

The attached field boring log and monitoring well completion sheets are to be completed for each monitoring well.

#### **Attached Supporting Documents**

- Volume of Soil Boring, Annulus around 2" and 4" Casings, and Grout Mixtures
- **Terracon Form 130** Log of Boring No., Monitoring Well Details, and Boring Elevations.
- ASTM D 5092-90 Design and Installation of Groundwater Monitoring Wells
- Brainard Kilman Field Facts

CUBIC FEET PER LINEAR FOOT

### VOLUME OF SOIL BORING

#### GALLONS PER LINEAR FOOT

002.0	DIAME	TFR O	F SOII	BORIN	G	
				INSIDE I		ER OF
				HOLLOV		
				3.25	4.25	6.25
LENGTH						
(ft.)	2	4	6	7.25	8.25	10.25
1	0.028	0.088	0.201	0.29	0.37	0.57
2	0.06	0.18	0.4	0.6	0.7	1.1
3	0.08	0.26	0.6	0.9	1.1	1.7
4	0.11	0.35	0.8	1.2	1.5	2.3
5	0.14	0.44	1.0	1.5	1.9	2.9
6	0.17	0.53	1.2	1.7	2.2	3.4
7	0.20	0.62	1.4	2.0	2.6	4.0
8	0.22	0.70	1.6	2.3	3.0	4.6
9	0.25	0.79	1.8	2.6	3.3	5.1
10	0.28	0.88	2.0	2.9	3.7	5.7
11	0.31	0.97	2.2	3.2	4.1	6.3
12	0.34	1.06	2.4	3.5	4.4	6.8
13	0.36	1.14	2.6	3.8	4.8	7.4
14	0.39	1.23	2.8	4.1	5.2	8.0
15	0.42	1.32	3.0	4.4	5.6	8.6
16	0.45	1.41	3.2	4.6	5.9	9.1
17	0.48	1.50	3.4	4.9	6.3	9.7
18	0.50	1.58	3.6	5.2	6.7	10.3
19	0.53	1.67	3.8	5.5	7.0	10.8
20	0.56	1.76	4.0	5.8	7.4	11.4
22	0.62	1.94	4.4	6.4	8.1	12.5
24	0.67	2.11	4.8	7.0	8.9	13.7
25	0.70	2.20	5.0	7.3	9.3	14.3
26	0.73	2.29	5.2	7.5	9.6	14.8
28	0.78		5.6	8.1	10.4	16.0
30	0.84	2.64	6.0	8.7	11.1	17.1
32	0.90	2.82	6.4	9.3	11.8	18.2
34	0.95	2.99	6.8	9.9	12.6	19.4
35	0.98	3.08	7.0	10.2	13.0	20.0
36	1.01	3.17	7.2	10.4	13.3	20.5
38	1.06	3.34	7.6	11.0	14.1	21.7
40	1.12	3.52	8.0	11.6	14.8	22.8
45	1.3	4.0	9.0	13.1	16.7	25.7
50 55	1.4 1.5	4.4 4.8	10.1 11.1	14.5 16.0	18.5 20.4	28.5 31.4
			12.1	17.4		34.2
60 65	1.7 1 8	5.3		17.4	22.2 24.1	
65 70	1.8 2.0	5.7 6.2	13.1 14.1	20.3	24.1 25.9	37.1 39.9
70	2.0	6.6	14.1	20.3 21.8	25.9 27.8	39.9 42.8
80	2.1	0.0 7.0	16.1	21.0	27.0 29.6	42.0 45.6
85	2.2	7.5	17.1	23.2	31.5	48.5
90	2.4 2.5	7.9	17.1	24.7	33.3	40.5 51.3
90 95	2.5	8.4	19.1	27.6	35.2	54.2
100	2.7	8.8	20.1	27.0	37.0	54.2 57.0
125	3.5	11.0	25.1	36.3	46.3	71.3
150	4.2	13.2	30.2	43.5	55.5	85.5
175	4.9	15.4	35.2	50.8	64.8	99.8
200	5.6	17.6	40.2	58.0	74.0	114.0
200	0.0		.0.2	00.0	. 1.0	

	DIANE	IERU	F SUIL		DIAMETE	
					V STEM A	
				3.25	4.25	6.25
LENGTH	ĺ	1	1	5.25	4.25	0.25
(ft.)	2	4	6	7.25	8.25	10.25
1	0.17	0.66	1.5	2.14	2.78	4.29
2	0.17	1.3	3.0	4.3	5.6	4.25
3	0.5	2.0	4.5	6.4	8.3	12.9
4	0.7	2.6	6.0	8.6	11.1	17.2
5	0.9	3.3	7.5	10.7	13.9	21.5
6	1.0	4.0	9.0	12.8	16.7	25.7
7	1.2	4.6	10.5	15.0	19.5	30.0
8	1.4	5.3	12.0	17.1	22.2	34.3
9	1.5	5.9	13.5	19.3	25.0	38.6
10	1.7	6.6	15.0	21.4	27.8	42.9
11	1.9	7.3	16.5	23.5	30.6	47.2
12	2.0	7.9	18.0	25.7	33.4	51.5
13	2.2	8.6	19.5	27.8	36.1	55.8
14	2.4	9.2	21.0	30.0	38.9	60.1
15	2.6	9.9	22.5	32.1	41.7	64.4
16	2.7	10.6	24.0	34.2	44.5	68.6
17	2.9	11.2	25.5	36.4	47.3	72.9
18 19	3.1 3.2	11.9 12.5	27.0	38.5	50.0	77.2 81.5
20	3.2 3.4	12.5	28.5 30.0	40.7 42.8	52.8 55.6	85.8
20	3.7	14.5	33.0	47.1	61.2	94.4
24	4.1	15.8	36.0	51.4	66.7	103.0
25	4.3	16.5	37.5	53.5	69.5	107.3
26	4.4	17.2	39.0	55.6	72.3	111.5
28	4.8	18.5	42.0	59.9	77.8	120.1
30	5.1	19.8	45.0	64.2	83.4	128.7
32	5.4	21.1	48.0	68.5	89.0	137.3
34	5.8	22.4	51.0	72.8	94.5	145.9
35	6.0	23.1	52.5	74.9	97.3	150.2
36	6.1	23.8	54.0	77.0	100.1	154.4
38	6.5	25.1	57.0	81.3	105.6	163.0
40	6.8	26.4	60.0	85.6	111.2	171.6
45	7.7	29.7	67.5	96.3	125.1	193.1
50	8.5	33.0	75.0	107.0	139.0	214.5
55	9.4	36.3	82.5	117.7	152.9	236.0
60 65	10.2 11.1	39.6	90.0 97.5	128.4 139.1	166.8 180.7	257.4
65 70	11.1	42.9 46.2	97.5 105.0	139.1	194.6	278.9
70	12.8	40.2 49.5	105.0	149.0	208.5	300.3 321.8
80	13.6	49.5 52.8	12.5	171.2	200.5	343.2
85	14.5	56.1	127.5	181.9	236.3	364.7
90	15.3	59.4	135.0	192.6	250.2	386.1
95	16.2	62.7	142.5	203.3	264.1	407.6
100	17.0	66.0	150.0	214.0	278.0	429.0
125	21.3	82.5	187.5	267.5	347.5	536.3
150	25.5	99.0	225.0	321.0	417.0	643.5
175	29.8	115.5	262.5	374.5	486.5	750.8
200	34.0	132.0	300.0	428.0	556.0	858.0

				NEAR F								EAR F		
	Ľ	DIAMET	ER OF	SOIL B	ORINO	3			[	DIAMET	ER OF	SOIL E	BORING	3
			1	INSIDE	DIAMET	ER OF						INSIDE	E DIAMET	ER OF
				HOLLOV	N STEM	AUGER						HOLLO	W STEM	AUGER
				3.25	4.25	6.25						3.25	4.25	6.25
LENGTH								LENGTH						0.20
(ft.)	2	4	6	7.25	8.25	10.25		(ft.)	2	4	6	7.25	8.25	10.25
1	0	0.065	0.178	0.26	0.34	0.54		1	0	0.491	1.33	1.91	2.55	4.06
2	0	0.003	0.170	0.20	0.34	1.1		2	0	1.0	2.7	3.8	5.1	8.1
23	0	0.13	0.4	0.5	1.0	1.1		23	0	1.5	4.0	5.7	7.7	12.2
4		0.20			1.0	2.2		4		2.0	5.3		10.2	16.2
4 5	0 0	0.20	0.7 0.9	1.0 1.3	1.4	2.2		4 5	0 0	2.0	5.3 6.7	7.6 9.6	12.8	20.3
6	0	0.33	1.1	1.6	2.0	3.2		6	0	2.5		11.5	15.3	20.3
7						3.2 3.8		7			8.0 9.3		17.9	24.4
8	0	0.46 0.52	1.2	1.8 2.1	2.4 2.7	3.0 4.3			0	3.4 3.9	9.3 10.6	13.4 15.3	20.4	20.4 32.5
8 9	0	0.52	1.4 1.6	2.1	2.7 3.1	4.3 4.9		8 9	0 0	5.9 4.4	12.0	17.2	20.4	36.5
9 10	0 0	0.59	1.0	2.5	3.1	4.9 5.4		9 10	0	4.4	13.3	17.2	25.0	40.6
10	0				3.4	5.9		10	0		14.6	21.0	28.1	40.0
11		0.72 0.78	2.0 2.1	2.9 3.1	3.7 4.1			11		5.4 5.9	14.6 16.0	21.0	30.6	44.7
12	0 0	0.78	2.1	3.1 3.4	4.1 4.4	6.5 7.0		12	0 0	5.9 6.4	16.0	22.9	30.6	48.7 52.8
					4.4 4.8					6.9		24.0	35.2	52.8
14 15	0 0	0.91 0.98	2.5 2.7	3.6 3.9	4.0 5.1	7.6 8.1		14 15	0 0	0.9 7.4	18.6 20.0	28.7	38.3	60.9
15	0	1.04	2.7		5.4			16	0		20.0			65.0
10		1.04	2.0 3.0	4.2	5.4 5.8	8.6 9.2		10		7.9 8.3	21.3	30.6 32.5	40.8 43.4	69.0
17	0	1.17	3.0	4.4 4.7	5.0 6.1	9.2 9.7		17	0 0	0.3 8.8	22.0	34.4	45.9	73.1
10	0	1.17	3.2 3.4	4.7	6.5	9.7 10.3		10	0	0.0 9.3	25.9 25.3	36.3	45.9	77.1
20	0 0	1.24	3.4 3.6	4.9 5.2	6.8	10.3		20	0	9.3 9.8	26.6	38.2	40.5 51.0	81.2
20		1.43	3.9	5.2	7.5	11.9		20	0					89.3
22	0	1.43	3.9 4.3	5.7 6.2	7.5 8.2	13.0		22 24		10.8 11.8	29.3 31.9	42.0 45.8	56.1 61.2	09.3 97.4
24 25	0	1.63	4.5 4.5	6.5	o.∠ 8.5	13.0		24 25	0 0	12.3	33.3	45.8	63.8	101.5
25	0 0	1.69	4.5	6.8	8.8	14.0		25	0	12.3	34.6	47.8	66.3	101.5
20	0	1.82	4.0 5.0	7.3	9.5	15.1		20	0	13.7	37.2	53.5	71.4	113.7
30	0	1.95	5.3	7.8	10.2	16.2		30	0	14.7	39.9	57.3	76.5	121.8
30	0	2.08	5.3 5.7	7.0 8.3	10.2	17.3		30	0	14.7	42.6	61.1	81.6	121.0
34	0	2.00	6.1	8.8	11.6	18.4		32 34	0	16.7	42.0	64.9	86.7	138.0
35	0	2.21	6.2	9.1	11.9	18.9		34	0	17.2	46.6	66.9	89.3	142.1
36	0	2.20	6.4	9.4	12.2	19.4		36	0	17.2	47.9	68.8	91.8	146.2
38	0	2.34	6.8	9.4	12.2	20.5		38	0	18.7	50.5	72.6	96.9	154.3
40	0	2.47	0.0 7.1	9.9 10.4	12.9	20.5		38 40	0	19.6	53.2	72.0	102.0	162.4
40	0	2.00	8.0	11.7	15.3	24.3		40 45	0	22.1	59.9	86.0	114.8	182.7
40 50	0	3.3	8.9	13.0	17.0	27.0		40 50	0	24.6	66.5	95.5	127.5	203.0
55	0	3.6	9.8	14.3	18.7	29.7		55	0	27.0	73.2		140.3	203.0
60	0	3.9	10.7	15.6	20.4	32.4		60	0	29.5	79.8		153.0	243.6
65	0	4.2	11.6	16.9	20.4	35.1		65	0	31.9	86.5		165.8	
70	0	4.6	12.5	18.2	23.8	37.8		70	0	34.4	93.1			
70	0	4.9	13.4	19.5	25.5	40.5		70	0	36.8	99.8		191.3	
80	0	5.2	14.2	20.8	27.2	43.2		80	0		106.4			324.8
85	0	5.5	15.1	20.0	28.9	45.9		85	0	41.7	113.1		216.8	345.1
90	0	5.9	16.0	23.4	30.6	48.6		90	0	44.2				365.4
95	0	6.2	16.9	24.7	32.3	51.3		95	0		126.4		242.3	385.7
100	0	6.5	17.8	26.0	34.0	54.0		100	0	49.1	133.0		255.0	406.0
125	0	8.1	22.3	32.5	42.5	67.5		125	0			238.8		507.5
150	0	9.8	26.7	39.0	51.0	81.0		150	0	73.7		286.5	382.5	609.0
175	0	11.4	31.2	45.5	59.5	94.5		175	0			334.3		710.5
200	Ő	13.0	35.6	52.0	68.0			200	Ő			382.0		
200	0	10.0	00.0	52.0	00.0	100.0	Ι.	200	0	00.2	200.0	002.0	0.0.0	0.2.0

#### VOLUME OF ANNULUS AROUND 2" CASING CUBIC FEET PER LINEAR FOOT GALLONS PER LINEAR FOOT

5

	CUBIC	FEET F	PER LI			ANNUL	.03	AROUND			PER LIN	IEAR F	оот	
				SOIL E		G						F SOIL I		3
				INSIDE	DIAMET	ER OF						INSIDE	DIAMET	ER OF
				HOLLOV	V STEM	AUGER						HOLLO	W STEM	AUGER
				3.25	4.25	6.25						3.25	4.25	6.25
LENGTH								LENGTH						
(ft.)	2	4	6	7.25	8.25	10.25		(ft.)	2	4	6	7.25	8.25	10.25
1	0	0	0.11	0.2	0.26	0.46		1	0	0	0.84	1.48	1.95	3.46
2	0	0	0.2	0.4	0.5	0.9		2	0	0	1.7	3.0	3.9	6.9
3	0	0	0.3	0.6	0.8	1.4		3	0	0	2.5	4.4	5.9	10.4
4	0	0	0.4	0.8	1.0	1.8		4	0	0	3.4	5.9	7.8	13.8
5	0	0	0.6	1.0	1.3	2.3		5	0	0	4.2	7.4	9.8	17.3
6	0	0	0.7	1.2	1.6	2.8		6	0	0	5.0	8.9	11.7	20.8
7	0	0	0.8	1.4	1.8	3.2		7	0	0	5.9	10.4	13.7	24.2
8	0	0	0.9	1.6	2.1	3.7		8	0	0	6.7	11.8	15.6	27.7
9	0	0	1.0	1.8	2.3	4.1		9	0	0	7.6	13.3	17.6	31.1
10	0	0	1.1	2.0	2.6	4.6		10	0	0	8.4	14.8	19.5	34.6
11	0	0	1.2	2.2	2.9	5.1		11	0	0	9.2	16.3	21.5	38.1
12	0	0	1.3	2.4	3.1	5.5		12	0	0	10.1	17.8	23.4	41.5
13	0	0	1.4	2.6	3.4	6.0		13	0	0	10.9	19.2	25.4	45.0
14	0	0	1.5	2.8	3.6	6.4		14	0	0	11.8	20.7	27.3	48.4
15	0	0	1.7	3.0	3.9	6.9		15	0	0	12.6	22.2	29.3	51.9
16	0	0	1.8	3.2	4.2	7.4		16	0	0	13.4	23.7	31.2	55.4
17	0	0	1.9	3.4	4.4	7.8		17	0	0	14.3	25.2	33.2	58.8
18	0	0	2.0	3.6	4.7	8.3		18	0	0	15.1	26.6	35.1	62.3
19 20	0 0	0 0	2.1 2.2	3.8 4.0	4.9 5.2	8.7 9.2		19 20	0 0	0 0	16.0 16.8	28.1 29.6	37.1 39.0	65.7 69.2
20	0	0	2.2	4.0	5.2	10.1		20	0	0	18.5	32.6	42.9	76.1
22	0	0	2.4 2.6	4.4 4.8	6.2	11.0		22	0	0	20.2	35.5	42.9	83.0
25	0	0	2.8	5.0	6.5	11.5		25	0	0	21.0	37.0	48.8	86.5
26	0	0	2.9	5.2	6.8	12.0		26	0	0	21.8	38.5	50.7	90.0
28	Ő	Ő	3.1	5.6	7.3	12.9		28	Õ	Õ	23.5	41.4	54.6	96.9
30	0	0	3.3	6.0	7.8	13.8		30	0	0	25.2	44.4	58.5	103.8
32	0	0	3.5	6.4	8.3	14.7		32	0	0	26.9	47.4	62.4	110.7
34	0	0	3.7	6.8	8.8	15.6		34	0	0	28.6	50.3	66.3	117.6
35	0	0	3.9	7.0	9.1	16.1		35	0	0	29.4	51.8	68.3	121.1
36	0	0	4.0	7.2	9.4	16.6		36	0	0	30.2	53.3	70.2	124.6
38	0	0	4.2	7.6	9.9	17.5		38	0	0	31.9	56.2	74.1	131.5
40	0	0	4.4	8.0	10.4	18.4		40	0	0	33.6	59.2	78.0	138.4
45	0	0	5.0	9.0	11.7	20.7		45	0	0	37.8	66.6	87.8	155.7
50	0	0	5.5	10.0	13.0	23.0		50	0	0	42.0	74.0	97.5	173.0
55		0	6.1	11.0	14.3	25.3		55	0	0	46.2	81.4	107.3	190.3
60		0	6.6	12.0	15.6	27.6		60 65	0	0	50.4	88.8	117.0	207.6
65	0	0	7.2	13.0	16.9	29.9		65 70	0	0	54.6	96.2	126.8	224.9
70	0	0	7.7	14.0	18.2	32.2		70 75	0	0	58.8 63.0	103.6	136.5	242.2
75 80	0 0	0 0	8.3 8.8	15.0 16.0	19.5 20.8	34.5 36.8		75 80	0 0	0 0	63.0 67.2	111.0 118.4	146.3 156.0	259.5 276.8
85	0	0	0.0 9.4	17.0	20.8	39.1		85	0	0	71.4	125.8	165.8	270.8
90	0	0	9.4 9.9	17.0	22.1	39.1 41.4		80 90	0	0	71.4	125.0	175.5	311.4
95	0	0	10.5	19.0	24.7	43.7		90 95	0	0	79.8	140.6	185.3	328.7
100		0	11.0	20.0	26.0	46.0		100	0	0	84.0	148.0	195.0	346.0
125	0	0	13.8	25.0	32.5	57.5		125	0	0	105.0	185.0	243.8	432.5
150	0	0	16.5	30.0	39.0	69.0		150	0	0	126.0	222.0	292.5	519.0
175	Ő	Ő	19.3	35.0	45.5	80.5		175	Ő	Õ	147.0	259.0	341.3	605.5
200		0	22.0	40.0	52.0	92.0		200	0	0	168.0	296.0	390.0	692.0

#### VOLUME OF ANNULUS AROUND 4" CASING INEAR FOOT GALLONS PER LINEAR FOOT

6

#### **GROUT MIXTURES** PORTLAND CEMENT GROUT

#### **BENTONITE GROUT**

CUBIC	CEMENT	BENTONITE	WATER	CUBIC	BENTONITE	WATER
FEET	94 lb. sack	lbs.	gallons	FEET	50 lb sack	gallons
1	0.6339	3.17	5.07	1	0.444	6.22
1.577	1.0	5.0	8.0	2	0.9	12.4
2	1.27	6.3	10.1	2.25	1	14
3	1.90	9.5	15.2	3	1.3	18.7
4	2.54	12.7	20.3	4	1.8	24.9
5	3.17	15.9	25.4	5	2.2	31.1
6	3.80	19.0	30.4	6	2.7	37.3
7	4.44	22.2	35.5	7	3.1	43.5
8	5.07	25.4	40.6	8	3.6	49.8
9	5.71	28.5	45.6	9	4.0	56.0
10	6.34	31.7	50.7	10	4.4	62.2
11	6.97	34.9	55.8	11	4.9	68.4
12	7.61	38.0	60.8	12	5.3	74.6
13	8.24	41.2	65.9	13	5.8	80.9
14	8.87	44.4	71.0	14	6.2	87.1
15	9.51	47.6	76.1	15	6.7	93.3
16	10.14	50.7	81.1	16	7.1	99.5
17	10.78	53.9	86.2	17	7.5	105.7
18	11.41	57.1	91.3	18	8.0	112.0
19	12.04	60.2	96.3	19	8.4	118.2
20	12.68	63.4	101.4	20	8.9	124.4
22	13.95	69.7	111.5	22	9.8	136.8
24	15.21	76.1	121.7	24	10.7	149.3
25	15.85	79.3	126.8	25	11.1	155.5
26	16.48	82.4	131.8	26	11.5	161.7
28	17.75	88.8	142.0	28	12.4	174.2
30	19.02	95.1	152.1	30	13.3	186.6
32	20.28	101.4	162.2	32	14.2	199.0
34	21.55	107.8	172.4	34	15.1	211.5
35	22.19	111.0	177.5	35	15.5	217.7
36	22.82	114.1	182.5	36	16.0	223.9
38	24.09	120.5	192.7	38	16.9	236.4
40	25.36	126.8	202.8	40	17.8	248.8
45	28.5	142.7	228.2	45	20.0	279.9
50	31.7	158.5	253.5	50	22.2	311.0
55	34.9	174.4	278.9	55	24.4	342.1
60	38.0	190.2	304.2	60	26.6	373.2
65	41.2	206.1	329.6	65	28.9	404.3
70	44.4	221.9	354.9	70	31.1	435.4
75	47.5	237.8	380.3	75	33.3	466.5
80	50.7	253.6	405.6	80	35.5	497.6
85	53.9	269.5	431.0	85	37.7	528.7
90	57.1	285.3	456.3	90	40.0	559.8
95	60.2	301.2	481.7	95	42.2	590.9
100	63.4	317.0	507.0	100	44.4	622.0
125	79.2	396.3	633.8	125	55.5	777.5
150	95.1	475.5	760.5	150	66.6	933.0
175	110.9	554.8	887.3	175	77.7	1088.5
200	126.8	634.0	1014.0	200	88.8	1244.0

## E.800 WELL CONSTRUCTION – PERMANENT

#### Last Revision or Review: June 2010

#### Objective

To provide standard procedures for the design and installation of routine monitoring wells that will result in reliable construction and provide for the collection of representative groundwater samples.

#### Background

The standard practice for design and installation of monitoring wells is contained in ASTM D 5092 -90 "Design and Installation of Groundwater Monitoring Wells in Aquifers." Terracon procedures incorporate the provisions of ASTM D 5092 which describe the specific procedures to be used in installing monitoring wells in unconsolidated formations. The ASTM standard should be referred to when installation of monitoring wells is required outside of the routine monitoring wells described here.

#### Equipment

The augers required for 2-inch diameter monitoring wells are 4-1/4-inch inside diameter hollow stem augers. For 4-inch wells the 6-1/4-inch inside diameter hollow stem augers are recommended. Flight augers are not recommended for installation of monitoring wells unless aquifer conditions are well known and will allow installation of screen and riser pipe without formation cave-in.

#### **Monitoring Well Material**

a) Riser Pipe

The riser pipe used is threaded, flush jointed Tri-Loc (or equivalent) Schedule 40 PVC pipe. This riser pipe has been found to provide a reliable and durable well.

b) Screen

Slot openings generally used for monitoring wells are 0.010 and 0.020 inch. The slot opening commonly used is 0.010 which will be effective with the use of gravel pack #20-40. If a larger slot opening is desired (.020), a gravel pack of #10-20 is recommended.

c) Gravel Pack

The standard gravel pack used is graded as #20-40 which is suited for use with a slot size of 0.010. Gravel pack should extend approximately two feet above the slotted screen. Gravel pack grading should be specified by the Project Manager or the Drilling Coordinator of the NE Division.

**TSOP E.800** 

#### Terracon

d) Bentonite

Bentonite pellets (1/4-inch size) are used to provide a relatively impermeable layer above the screen and gravel pack. One 5-gallon bucket of bentonite pellets should provide a two foot layer in the annulus of a boring created with 4 and 1/4-inch inside diameter augers and containing 2-inch monitoring well pipe.

e) Surface Security Materials

The well is secured in accordance with the project manager's instructions and TSOPs E.900, E.905, E.910, or E.920.

#### Procedures

a) Background

Personnel involved in drilling operations must be familiar with the drilling procedures in ASTM D 5092. The Project Manager will specify the sampling interval, decontamination requirements, screen slot size, gravel pack size, and completion and survey requirements.

b) Mobilization

The project manager will prepare the mobilization sheets for the required drilling operation. The mobilization sheets should summarize types and quantities of well materials, drilling supplies, appropriate equipment, boring depths and boring locations. The enclosed tables should be referred to for calculating the volumes of gravel pack, bentonite, and grout required.

c) Field Operations

The field crews will review the mobilization sheets for adequacy. Unless otherwise specified the field crew will determine the specific use of equipment such as grout trailer, ready mix etc. The field crews will complete the monitoring wells as specified and complete forms for describing boring logs and well details. Any deviation from standard procedures or unusual observations should be noted by the field crews and reported to the project manager.

d) Well Security

The well is secured in accordance with the project manager's instructions and TSOPs E.900, E.905, E.910, or E.920. The proper well security materials to be used in completing the top of a monitoring well should be specified by the project manager and based on the expected ground surface use, regulatory requirements, and client desires. Care should be taken in installation of monitoring wells that will be subjected to vehicle traffic.

e) Well Documentation.

The attached field boring log and monitoring well completion sheets are to be completed for each monitoring well.

### **Attached Supporting Documents**

- Volume of Soil Boring, Annulus around 2" and 4" Casings, and Grout Mixtures
- **Terracon Form 130** Log of Boring No., Monitoring Well Details, and Boring Elevations.
- **ASTM D 5092-90** Design and Installation of Groundwater Monitoring Wells
- Brainard Kilman Field Facts

#### CUBIC FEET PER LINEAR FOOT

#### VOLUME OF SOIL BORING GALLONS PER LINEAR FOOT

CUBIC			IEAR F			
	DIAME	TER O	F SOIL			
				INSIDE I		
				HOLLOV	V STEM	AUGER
				3.25	4.25	6.25
LENGTH						
(ft.)	2	4	6	7.25	8.25	10.25
1	0.028	0.088	0.201	0.29	0.37	0.57
2	0.06	0.18	0.4	0.6	0.7	1.1
3	0.08	0.26	0.6	0.9	1.1	1.7
4	0.11	0.35	0.8	1.2	1.5	2.3
5	0.14	0.44	1.0	1.5	1.9	2.9
6	0.17	0.53	1.2	1.7	2.2	3.4
7	0.20	0.62	1.4	2.0	2.6	4.0
8	0.22	0.70	1.6	2.3	3.0	4.6
9	0.25	0.79	1.8	2.6	3.3	5.1
10	0.28	0.88	2.0	2.9	3.7	5.7
11	0.31	0.97	2.2	3.2	4.1	6.3
12	0.34	1.06	2.4	3.5	4.4	6.8
13	0.36	1.14	2.6	3.8	4.8	7.4
14	0.39	1.23	2.8	4.1	5.2	8.0
15	0.42	1.32	3.0	4.4	5.6	8.6
16	0.45	1.41	3.2	4.6	5.9	9.1
17	0.48	1.50	3.4	4.9	6.3	9.7
18	0.50	1.58	3.6	5.2	6.7	10.3
19	0.53	1.67	3.8	5.5	7.0	10.8
20	0.56	1.76	4.0	5.8	7.4	11.4
22	0.62	1.94	4.4	6.4	8.1	12.5
24	0.67	2.11	4.8	7.0	8.9	13.7
25	0.70	2.20	5.0	7.3	9.3	14.3
26	0.73	2.29	5.2	7.5	9.6	14.8
28	0.78	2.46	5.6	8.1	10.4	16.0
30	0.84	2.64	6.0	8.7	11.1	17.1
32	0.90	2.82	6.4	9.3	11.8	18.2
34	0.95	2.99	6.8	9.9	12.6	19.4
35	0.98	3.08	7.0	10.2	13.0	20.0
36	1.01	3.17	7.2	10.4	13.3	20.5
38	1.06	3.34	7.6	11.0	14.1	21.7
40	1.12	3.52	8.0	11.6	14.8	22.8
45	1.3	4.0	9.0	13.1	16.7	25.7
50	1.4	4.4	10.1	14.5	18.5	28.5
55	1.5	4.8	11.1	16.0	20.4	31.4
60	1.7	5.3	12.1	17.4	22.2	34.2
65	1.8	5.7	13.1	18.9	24.1	37.1
70	2.0	6.2	14.1	20.3	25.9	39.9
75	2.1	6.6	15.1	21.8	27.8	42.8
80	2.2	7.0	16.1	23.2	29.6	45.6
85	2.4	7.5	17.1	24.7	31.5	48.5
90	2.5	7.9	18.1	26.1	33.3	51.3
95	2.7	8.4	19.1	27.6	35.2	54.2
100	2.8	8.8	20.1	29.0	37.0	57.0
125	3.5	11.0	25.1	36.3	46.3	71.3
150	4.2	13.2	30.2	43.5	55.5	85.5
175	4.9	15.4	35.2	50.8	64.8	99.8
200	5.6	17.6	40.2	58.0	74.0	114.0

GALLO						
	DIAME	TER O	F SOIL			
					DIAMETE	
				HOLLOV	V STEM A	AUGER
				3.25	4.25	6.25
LENGTH						
(ft.)	2	4	6	7.25	8.25	10.25
1	0.17		1.5	2.14	2.78	4.29
		0.66				
2	0.3	1.3	3.0	4.3	5.6	8.6
3	0.5	2.0	4.5	6.4	8.3	12.9
4	0.7	2.6	6.0	8.6	11.1	17.2
5	0.9	3.3	7.5	10.7	13.9	21.5
6	1.0	4.0	9.0	12.8	16.7	25.7
7	1.2	4.6	10.5	15.0	19.5	30.0
8	1.4	5.3	12.0	17.1	22.2	34.3
9	1.5	5.9	13.5	19.3	25.0	38.6
10	1.7	6.6	15.0	21.4	27.8	42.9
11	1.9	7.3	16.5	23.5	30.6	47.2
12	2.0	7.9	18.0	25.7	33.4	51.5
13	2.2	8.6	19.5	27.8	36.1	55.8
14	2.2	9.2	21.0	30.0	38.9	60.1
14	2.4	9.9	21.0	32.1	41.7	64.4
16 17	2.7	10.6	24.0	34.2	44.5	68.6
	2.9	11.2	25.5	36.4	47.3	72.9
18	3.1	11.9	27.0	38.5	50.0	77.2
19	3.2	12.5	28.5	40.7	52.8	81.5
20	3.4	13.2	30.0	42.8	55.6	85.8
22	3.7	14.5	33.0	47.1	61.2	94.4
24	4.1	15.8	36.0	51.4	66.7	103.0
25	4.3	16.5	37.5	53.5	69.5	107.3
26	4.4	17.2	39.0	55.6	72.3	111.5
28	4.8	18.5	42.0	59.9	77.8	120.1
30	5.1	19.8	45.0	64.2	83.4	128.7
32	5.4	21.1	48.0	68.5	89.0	137.3
34	5.8	22.4	51.0	72.8	94.5	145.9
35	6.0	23.1	52.5	74.9	97.3	150.2
36	6.1	23.8	54.0	77.0	100.1	154.4
38	6.5	25.1	57.0	81.3	105.6	163.0
40	6.8	26.4	60.0	85.6	111.2	171.6
45	7.7	29.7	67.5	96.3	125.1	193.1
40 50	8.5	33.0	75.0	107.0	139.0	214.5
55	9.4	36.3	82.5	117.7	152.9	236.0
	10.2	39.6		128.4	166.8	257.4
60 65			90.0 07.5	120.4		
65 70	11.1	42.9	97.5		180.7	278.9
70	11.9	46.2	105.0	149.8	194.6	300.3
75	12.8	49.5	112.5	160.5	208.5	321.8
80	13.6	52.8	120.0	171.2	222.4	343.2
85	14.5	56.1	127.5	181.9	236.3	364.7
90	15.3	59.4	135.0	192.6	250.2	386.1
95	16.2	62.7	142.5	203.3	264.1	407.6
100	17.0	66.0	150.0	214.0	278.0	429.0
125	21.3	82.5	187.5	267.5	347.5	536.3
150	25.5	99.0	225.0	321.0	417.0	643.5
175	29.8	115.5	262.5	374.5	486.5	750.8
200	34.0	132.0	300.0	428.0	556.0	858.0
200	0 110		000.0	0.0	220.0	000.0

VOLUME OF ANNULUS AROUND 2" CASING CUBIC FEET PER LINEAR FOOT CUBIC FEET PER LINEAR FOOT CUBIC FEET OF COMPARING

	<u> </u>	DIAMET	ER OF	SOIL E		G
					DIAME	
				HOLLO\	N STEM	AUGER
				3.25	4.25	6.25
LENGTH						
(ft.)	2	4	6	7.25	8.25	10.25
1	0	0.065	0.178	0.26	0.34	0.54
2	0	0.13	0.4	0.5	0.7	1.1
3	0	0.20	0.5	0.8	1.0	1.6
4	0	0.26	0.7	1.0	1.4	2.2
5	0	0.33	0.9	1.3	1.7	2.7
6	0	0.39	1.1	1.6	2.0	3.2
7	0	0.46	1.2	1.8	2.4	3.8
8 9	0	0.52	1.4	2.1	2.7	4.3
9 10	0 0	0.59 0.65	1.6 1.8	2.3 2.6	3.1 3.4	4.9 5.4
10	0	0.03	2.0	2.0	3.4	5.9
12	0	0.72	2.0	3.1	4.1	6.5
13	0	0.85	2.3	3.4	4.4	7.0
14	Ő	0.91	2.5	3.6	4.8	7.6
15	Ő	0.98	2.7	3.9	5.1	8.1
16	0	1.04	2.8	4.2	5.4	8.6
17	0	1.11	3.0	4.4	5.8	9.2
18	0	1.17	3.2	4.7	6.1	9.7
19	0	1.24	3.4	4.9	6.5	10.3
20	0	1.30	3.6	5.2	6.8	10.8
22	0	1.43	3.9	5.7	7.5	11.9
24	0	1.56	4.3	6.2	8.2	13.0
25	0	1.63	4.5	6.5	8.5	13.5
26	0	1.69	4.6	6.8	8.8	14.0
28	0	1.82	5.0	7.3	9.5	15.1
30 32	0 0	1.95 2.08	5.3 5.7	7.8 8.3	10.2 10.9	16.2 17.3
32	0	2.00	6.1	0.3 8.8	11.6	17.3
35	0	2.21	6.2	9.1	11.9	18.9
36	0	2.34	6.4	9.4	12.2	19.4
38	0	2.47	6.8	9.9	12.9	20.5
40	Ő	2.60	7.1	10.4	13.6	21.6
45	0	2.9	8.0	11.7	15.3	24.3
50	0	3.3	8.9	13.0	17.0	27.0
55	0	3.6	9.8	14.3	18.7	29.7
60	0	3.9	10.7	15.6	20.4	32.4
65	0	4.2	11.6	16.9	22.1	35.1
70	0	4.6	12.5	18.2	23.8	37.8
75	0	4.9	13.4	19.5	25.5	40.5
80	0	5.2	14.2	20.8	27.2	43.2
85	0	5.5	15.1	22.1	28.9	45.9
90 95	0 0	5.9 6.2	16.0 16.9	23.4 24.7	30.6 32.3	48.6 51.3
100	0	6.2 6.5	17.8	24.7	32.3 34.0	51.3 54.0
125	0	8.1	22.3	32.5	42.5	67.5
120	0	9.8	26.7	39.0	51.0	81.0
175	0	11.4	31.2	45.5	59.5	94.5
200	0	13.0	35.6	52.0	68.0	108.0
	<u> </u>	. 0.0	00.0	02.0	00.0	

GALLONS PER LINEAR FOOT DIAMETER OF SOIL BORING										
	[	DIAMET	ER OF							
					DIAMET					
				HOLLO	W STEM					
				3.25	4.25	6.25				
LENGTH										
(ft.)	2	4	6	7.25	8.25	10.25				
1	0	0.491	1.33	1.91	2.55	4.06				
2	0	1.0	2.7	3.8	5.1	8.1				
3	0	1.5	4.0	5.7	7.7	12.2				
4	0	2.0	5.3	7.6	10.2	16.2				
5	0	2.5	6.7	9.6	12.8	20.3				
6	0	2.9	8.0	11.5	15.3	24.4				
7	0	3.4	9.3	13.4	17.9	28.4				
8	0	3.9	10.6	15.3	20.4	32.5				
9	0	4.4	12.0	17.2	23.0	36.5				
10	0	4.9	13.3	19.1	25.5	40.6				
11	0	5.4	14.6	21.0	28.1	44.7				
12	0	5.9	16.0	22.9	30.6	48.7				
13	0	6.4	17.3	24.8	33.2	52.8				
14 15	0 0	6.9 7.4	18.6 20.0	26.7 28.7	35.7 38.3	56.8				
16	0		20.0	30.6	40.8	60.9 65.0				
10	0	7.9 8.3	21.3	32.5	40.8	69.0				
18	0	8.8	23.9	34.4	45.9	73.1				
10	0	9.3	25.3	36.3	48.5	77.1				
20	0	9.8	26.6	38.2	51.0	81.2				
22	0	10.8	29.3	42.0	56.1	89.3				
24	Ő	11.8	31.9	45.8	61.2	97.4				
25	0	12.3	33.3	47.8	63.8	101.5				
26	0	12.8	34.6	49.7	66.3	105.6				
28	0	13.7	37.2	53.5	71.4	113.7				
30	0	14.7	39.9	57.3	76.5	121.8				
32	0	15.7	42.6	61.1	81.6	129.9				
34	0	16.7	45.2	64.9	86.7	138.0				
35	0	17.2	46.6	66.9	89.3	142.1				
36	0	17.7	47.9	68.8	91.8	146.2				
38	0	18.7	50.5	72.6	96.9	154.3				
40	0	19.6	53.2	76.4	102.0	162.4				
45	0	22.1	59.9	86.0	114.8	182.7				
50	0	24.6	66.5	95.5	127.5	203.0				
55	0	27.0	73.2	105.1	140.3	223.3				
60 65	0 0	29.5 31.9	79.8 86.5	114.6 124.2	153.0 165.8	243.6 263.9				
70	0	31.9 34.4	93.1	124.2	178.5	263.9 284.2				
70	0	34.4 36.8	93.1 99.8	133.7	176.5	204.2 304.5				
80	0	39.3	106.4	152.8	204.0	324.8				
85	0	41.7	113.1	162.4	216.8	345.1				
90	0	44.2	119.7	171.9	229.5	365.4				
95	0	46.6	126.4	181.5	242.3	385.7				
100	Ő	49.1	133.0	191.0	255.0	406.0				
125	Ő	61.4	166.3	238.8	318.8	507.5				
150	0	73.7	199.5	286.5	382.5	609.0				
175	0	85.9	232.8	334.3	446.3	710.5				
200	0	98.2	266.0	382.0	510.0	812.0				

VOLUME OF ANNULUS AROUND 4" CASING CUBIC FEET PER LINEAR FOOT \_\_\_\_\_ GALLONS PER LINEAR FOOT

(	CUBIC					
	D	IAMET	ER OF	SOIL E		
				-	DIAMET	
					V STEM	
LENGTH		i	1	3.25	4.25	6.25
(ft.)	2	4	6	7.25	8.25	10.25
(1)			0.11		0.25	10.25 0.46
2	0 0	0 0	0.11	0.2 0.4	0.20	0.40
3	0	0	0.3	0.6	0.8	1.4
4	Ő	Ő	0.4	0.8	1.0	1.8
5	0	0	0.6	1.0	1.3	2.3
6	0	0	0.7	1.2	1.6	2.8
7	0	0	0.8	1.4	1.8	3.2
8	0	0	0.9	1.6	2.1	3.7
9	0	0	1.0	1.8	2.3	4.1
10	0	0	1.1	2.0	2.6	4.6
11	0	0	1.2	2.2	2.9	5.1
12 13	0 0	0 0	1.3 1.4	2.4 2.6	3.1 3.4	5.5 6.0
13	0	0	1.4	2.0	3.4	6.4
15	0	0	1.7	3.0	3.9	6.9
16	0	0	1.8	3.2	4.2	7.4
17	0	0	1.9	3.4	4.4	7.8
18	0	0	2.0	3.6	4.7	8.3
19	0	0	2.1	3.8	4.9	8.7
20	0	0	2.2	4.0	5.2	9.2
22	0	0	2.4	4.4	5.7	10.1
24 25	0	0	2.6 2.8	4.8 5.0	6.2	11.0
25 26	0 0	0 0	2.0 2.9	5.0	6.5 6.8	11.5 12.0
28	0	0	3.1	5.6	7.3	12.0
30	0	0	3.3	6.0	7.8	13.8
32	0	0	3.5	6.4	8.3	14.7
34	0	0	3.7	6.8	8.8	15.6
35	0	0	3.9	7.0	9.1	16.1
36	0	0	4.0	7.2	9.4	16.6
38	0	0	4.2	7.6	9.9	17.5
40	0	0	4.4	8.0	10.4	18.4
45 50	0 0	0 0	5.0 5.5	9.0 10.0	11.7 13.0	20.7 23.0
55	0	0	6.1	11.0	14.3	25.3
60	0	0	6.6	12.0	15.6	27.6
65	0	0	7.2	13.0	16.9	29.9
70	0	Ő	7.7	14.0	18.2	32.2
75	0	0	8.3	15.0	19.5	34.5
80	0	0	8.8	16.0	20.8	36.8
85	0	0	9.4	17.0	22.1	39.1
90	0	0	9.9	18.0	23.4	41.4
95 100	0	0	10.5	19.0	24.7	43.7
100 125	0 0	0 0	11.0 13.8	20.0 25.0	26.0 32.5	46.0 57.5
123	0	0	16.5	30.0	39.0	69.0
175	0	0	19.3	35.0	45.5	80.5
200	0	0	22.0	40.0	52.0	92.0
	J	5				

·			PER LIN			
	Ľ	DIAME	TER O	SOIL I		
					DIAMET	
					W STEM	AUGER
				3.25	4.25	6.25
LENGTH						
(ft.)	2	4	6	7.25	8.25	10.25
1	0	0	0.84	1.48	1.95	3.46
2	0	0	1.7	3.0	3.9	6.9
3	0	0	2.5	4.4	5.9	10.4
4	0	0	3.4	5.9	7.8	13.8
5	0	0	4.2	7.4	9.8	17.3
6	0	0	5.0	8.9	11.7	20.8
7	0	0	5.9	10.4	13.7	24.2
8	0	0	6.7	11.8	15.6	27.7
9	0	0	7.6	13.3	17.6	31.1
10	0	0	8.4	14.8	19.5	34.6
11 12	0 0	0 0	9.2 10.1	16.3 17.8	21.5 23.4	38.1 41.5
12	0	0	10.1	17.0	25.4 25.4	41.5
14	0	0	11.8	20.7	27.3	48.4
15	0	0	12.6	22.2	29.3	51.9
16	0	0	13.4	23.7	31.2	55.4
17	0	Ő	14.3	25.2	33.2	58.8
18	0	0	15.1	26.6	35.1	62.3
19	0	0	16.0	28.1	37.1	65.7
20	0	0	16.8	29.6	39.0	69.2
22	0	0	18.5	32.6	42.9	76.1
24	0	0	20.2	35.5	46.8	83.0
25	0	0	21.0	37.0	48.8	86.5
26	0	0	21.8	38.5	50.7	90.0
28	0	0	23.5	41.4	54.6	96.9
30	0	0	25.2	44.4	58.5	103.8
32	0	0	26.9	47.4	62.4	110.7
34	0	0	28.6	50.3	66.3	117.6
35	0 0	0	29.4	51.8	68.3	121.1
36 38		0	30.2 31.9	53.3	70.2 74.1	124.6 131.5
40	0 0	0	33.6	56.2 59.2	74.1	131.5
40	0	0	37.8	66.6	87.8	155.7
50	0	0	42.0	74.0	97.5	173.0
55	0	0	46.2	81.4	107.3	190.3
60	0	0	50.4	88.8	117.0	207.6
65	Ő	Ő	54.6	96.2	126.8	224.9
70	0	0	58.8	103.6	136.5	242.2
75	0	0	63.0	111.0	146.3	259.5
80	0	0	67.2	118.4	156.0	276.8
85	0	0	71.4	125.8	165.8	294.1
90	0	0	75.6	133.2	175.5	311.4
95	0	0	79.8	140.6	185.3	328.7
100	0	0	84.0	148.0	195.0	346.0
125	0	0	105.0	185.0	243.8	432.5
150	0	0	126.0	222.0	292.5	519.0
175	0	0	147.0	259.0	341.3	605.5
200	0	0	168.0	296.0	390.0	692.0

#### **GROUT MIXTURES**

#### **BENTONITE GROUT**

			-	_
PORTL	AND	CEMENT	GROUT	

CUBIC FEET	CEMENT 94 lb. sack	BENTONITE lbs.	WATER gallons	CUBIC FEET	BENTONITE 50 lb sack	WATER gallons
1	0.6339	3.17	5.07	1	0.444	6.22
1.577	1.0	5.0	8.0	2	0.9	12.4
2	1.27	6.3	10.1	2.25	1	14
3	1.90	9.5	15.2	3	1.3	18.7
4	2.54	12.7	20.3	4	1.8	24.9
5	3.17	15.9	25.4	5	2.2	31.1
6	3.80	19.0	30.4	6	2.7	37.3
7	4.44	22.2	35.5	7	3.1	43.5
8	5.07	25.4	40.6	8	3.6	49.8
9	5.71	28.5	45.6	9	4.0	56.0
10	6.34	31.7	50.7	10	4.4	62.2
11	6.97	34.9	55.8	11	4.9	68.4
12	7.61	38.0	60.8	12	5.3	74.6
13	8.24	41.2	65.9	13	5.8	80.9
14	8.87	44.4	71.0	14	6.2	87.1
15	9.51	47.6	76.1	15	6.7	93.3
16	10.14	50.7	81.1	16	7.1	99.5
17	10.78	53.9	86.2	17	7.5	105.7
18	11.41	57.1	91.3	18	8.0	112.0
19	12.04	60.2	96.3	19	8.4	118.2
20	12.68	63.4	101.4	20	8.9	124.4
22	13.95	69.7	111.5	22	9.8	136.8
24	15.21	76.1	121.7	24	10.7	149.3
25	15.85	79.3	126.8	25	11.1	155.5
26	16.48	82.4	131.8	26	11.5	161.7
28	17.75	88.8	142.0	28	12.4	174.2
30	19.02	95.1	152.1	30	13.3	186.6
32	20.28	101.4	162.2	32	14.2	199.0
34	21.55	107.8	172.4	34	15.1	211.5
35	22.19	111.0	177.5	35	15.5	217.7
36	22.82	114.1	182.5	36	16.0	223.9
38	24.09	120.5	192.7	38	16.9	236.4
40	25.36	126.8	202.8	40	17.8	248.8
45	28.5	142.7	228.2	45	20.0	279.9
50 55	31.7 34.9	158.5	253.5	50 55	22.2	311.0
		174.4	278.9	60	24.4	342.1 373.2
60 65	38.0 41.2	190.2 206.1	304.2 329.6	60 65	26.6 28.9	373.2 404.3
70	41.2	200.1	354.9	03 70	31.1	404.3
70	44.4	237.8	380.3	70	33.3	466.5
80	50.7	253.6	405.6	80	35.5	400.5
85	53.9	269.5	431.0	85	37.7	528.7
90	57.1	285.3	451.0	85 90	40.0	528.7
95	60.2	301.2	481.7	90 95	42.2	590.9
100	63.4	317.0	507.0	100	44.4	622.0
125	79.2	396.3	633.8	125	55.5	777.5
150	95.1	475.5	760.5	150	66.6	933.0
175	110.9	554.8	887.3	175	77.7	1088.5
200	126.8	634.0	1014.0	200	88.8	1244.0



# E.850 WELL CONSTRUCTION –DIRECT PUSH (PREPACKED SCREEN)

Last Revision or Review: June 2010

#### Objective

To provide standard procedures for the design and installation of routine monitoring wells that will result in reliable construction and provide for the collection of representative groundwater samples.

#### Background

- The industry practice for design and direct push installation of monitoring wells is contained in ASTM D 6725-01 "Standard Practice for Direct Push Installation of Prepacked Screen Monitoring Wells in Unconsolidated Aquifers". Terracon procedures incorporate the provisions of ASTM D 6725 which describe the specific procedures to be used in installing monitoring wells in unconsolidated formations using direct push technology.
- Terracon adopts the attached "Geoprobe® 0.5-IN. x 1.4-IN. OD and ).75-IN. x 1.4-IN. OD Prepacked Screen Monitoring Wells Standard Operating Procedure", Technical Bulletin No. 962000, September 1996, Revised June 2002 (GSOP) and used with permission by Internet download from <a href="http://www.geoprobe.com/literature/pdfdownload.htm">http://www.geoprobe.com/literature/pdfdownload.htm</a>.

#### Equipment

The equipment shall be as generally described in Terracon Standard Operating Procedure E400 and as specified in GSOP Section 3 Required Equipment or industry equivalent.

#### Monitoring Well Material

Materials shall be as specified in Section 3 Required Equipment or industry equivalent.

#### Procedures

a) Background

Personnel involved in operations must be familiar with the procedures in ASTM D 6725. The Project Manager will specify the sampling interval, decontamination requirements, screen slot size, and completion and survey requirements.

b) Mobilization

The project manager will prepare the mobilization sheets for the required operation. The mobilization sheets should summarize types and quantities of materials, supplies, appropriate equipment, depths and locations.

c) Field Operations

The field crews will review the mobilization sheets for adequacy. The field crews will complete the monitoring wells as specified and complete forms for describing soil logs and well details. Any deviation from standard procedures or unusual observations should be noted by the field crews and reported to the project manager.

Construction of the prepacked screen monitoring well will be as specified in GSOP *Section 4: Well Installation.* 

d) Well Security

The well is secured in accordance with the project manager's instructions and TSOPs E.900, E.905, E.910, E.920 as modified by GSOP Section 4.5 Surface *Cover / Well Protection.* The proper well security materials to be used in completing the top of a monitoring well should be specified by the project manager and based on the expected ground surface use, regulatory requirements, and client desires. Care should be taken in installation of monitoring wells that will be subjected to vehicle traffic.

e) Well Documentation.

The standard field boring log and monitoring well completion sheets are to be completed for each monitoring well.

#### Attached Supporting Documents

- Terracon Form 130 Log of Boring No., Monitoring Well Details, and Boring Elevations.
- Geoprobe® 0.5-IN. x 1.4-IN. OD and ).75-IN. x 1.4-IN. OD Prepacked Screen Monitoring Wells Standard Operating Procedure, Technical Bulletin No. 962000, September 1996, Revised June 2002, Kejr, Inc.
- **ASTM D 6725-01** Standard Practice for Direct Push Installation of Prepacked Screen Monitoring Wells in Unconsolidated Aquifers.

#### STANDARD OPERATING PROCEDURE

## E.900 WELL SECURITY – TYPE A (Simple Cap)

LAST REVIEW OR REVISION: June 2010

- **OBJECTIVE:** To provide standard procedures for capping a monitoring well in the field in conditions where;
- The potential threat of persons deliberately contaminating groundwater used for sampling and testing through the surface well casing is non-existent
- The well is within a secured area or enclosure otherwise providing security
- The probability of inadvertent structural damage to the well casing is not likely
- The procedure does not provide an indicator if the well has been entered by other than authorized project personnel, thereby not allowing the sampler to halt sampling or flag samples as suspect.
- This procedure is low cost, low maintenance and provides no wellhead protection for sample integrity except against fugitive airborne dust. It provides the minimum level of protection for exposed well casing, inadvertent or deliberate, surface traffic.
- Special Note: This procedure may used at client request for other higher threat conditions providing the client understands and accepts the risk associated with sabotaged wells.

#### EQUIPMENT

- Copy of completed field well construction log, field or report version.
  - Terracon Form No. 130 MONITORING WELL DETAILS, see figure enclosed.
  - Terracon Final Report Log from project report.
- Cap compatible with well casing material
  - PVC slip-type end cap of proper inside diameter to match well casing. For wells greater than 3-inches in diameter the flange of the slip cap should be slotted with a hacksaw free of oils prior to installation to prevent seizing of the cap to well casing in temperature changes.
  - PVC or metal threaded caps manufactured to match the well casing thread and diameter.



- Hand-operated compression pipe cutter appropriate to material and without interference to the integrity of the sampling (requires no solvents or oils).
- Wellhead marking materials ONLY FOR EXTERNAL APPLICATION AWAY FROM WELLHEAD CASING OPENING

• Compatible markers or adhesive labels to permanently mark casing without solvent/residue interference with potential chemicals of analysis.

### PROCEDURES

Inspect the casing opening for adhered soils, sand or other materials which might interfere with the slip cap or threaded plug. Clear and clean appropriate with project cleaning or decontamination procedures. The top of casing should be visually true-and-round, visually free of chips, cracks or other physical damage which could interfere with the expanding gasket of the locking cap. The casing should be flat and level across the opening, within approximately  $\pm$ 10-degrees of perpendicular relative to the casing side wall.

If the top of casing is damaged, use the pipe cutter to square and repair the casing opening. Record the change in length to 0.1-inch relative to the original casing rise recorded on Form 130 or Report Log Form and report to the Terracon Project Manager.

Hand place the slip cap over the well casing, do NOT drive in place mechanically. For threaded plugs hand tighten the cap only, USE NO TOOLS.

Physically mark the casing with well identification. Record and return documentation to the Project Manager and file.

### DOCUMENTATION

Record any adjustments or repairs on completed Form 130 (Back) *Monitoring Well Details* and return to Project Manager.

### OTHER SUPPORTING DOCUMENTS

TSOP E.700	Well Construction – Temporary	August 2000
TSOP E.800	Well Construction – Permanent	November 2001

#### FORM 130 - BACK

MONITORING WELL DETAILS	E	BORING ELEVATIONS				
<ol> <li>Fill in applicable dimensions and identify backfill materials</li> <li>Sketch screen installation (if more than one section used)</li> </ol>	Benchmar	k				
	Elevation		Assumed		🗋 Given	
	Boring/ Turning Point	Backsight	Height Instrument	Foresight	Elevation	
HETTYPE         HETTYPE         Top Cap         2.5' Riser         5' Riser         5' Riser         10' Riser         2.5' Screen         5' Screen         5' Screen         10' Screen         Bottom Point         Sand         Bentonite         Grout         Concrete         Protective Casing         Bottom Plates						
Protective steel cover?  Yes (Lock Type) Type (Key number No	)					
Screen slot size or fabric type						
Well Installation Accepted by Driller						

# E.905 WELL SECURITY – TYPE B (Locking Expansion Cap)

### LAST REVIEW OR REVISION: June 2010

- **OBJECTIVE:** To provide standard procedures for physically securing monitoring wells in the field where the potential threat of persons deliberately contaminating groundwater used for sampling and testing through the surface well casing is low and the probability of inadvertent structural damage to the well casing is not likely.
- The procedure provides an indicator if the well has been entered by other than authorized project personnel, allowing the sampler to halt sampling or flag samples as suspect.
- This procedure is low cost, low maintenance and provides the lowest amount of wellhead protection for sample integrity. It provides the minimum level of protection for exposed well casing, inadvertent or deliberate, by surface traffic.
- Special Note: This procedure may used at client request for other higher threat conditions providing the client understands and accepts the risk associated with sabotaged wells.

### EQUIPMENT

- Copy of completed field well construction log, field or report version.
  - Terracon Form No. 130 MONITORING WELL DETAILS, see figure enclosed.
  - Terracon Final Report Log from project report.
- Lockable caps
  - With inert or chemically compatible expanding gasket of material not affected as to loss of expansion or damage down to temperatures of –10 degrees Fahrenheit.
  - Cherne Industries, Inc. Gripper® or Morrison-Dubuque #678XA, or equivalents approved by Terracon Equipment/Supplies Manager.
- Keyed-alike padlocks, hardened shank not required.



- Hand-operated compression pipe cutter appropriate to material and without interference to the integrity of the sampling (requires no solvents or oils).
- Wellhead marking materials ONLY FOR EXTERNAL APPLICATION AWAY FROM WELLHEAD CASING OPENING
  - Compatible markers or adhesive labels to permanently mark cap without solvent/residue interference with potential chemicals of analysis.
  - 2-inch diameter brass tag-and-ring to physically attach to padlock or cap
  - Steel number punch set for brass tags, if applicable.
  - Hammer for punch set, if applicable.
  - Small metal block or hand anvil for punch, set.

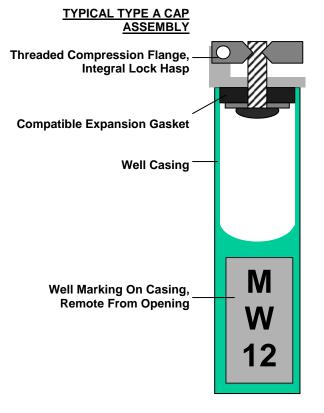
• Pliers or small box end wrench

### PROCEDURES

Inspect the casing opening for adhered soils, sand or other materials which might interfere with the expanding seal. Clear and clean appropriate with project cleaning or decontamination procedures. The top of casing should be visually true-and-round, visually free of chips, cracks or other physical damage which could interfere with the expanding gasket of the locking cap. The casing should be flat and level across the opening, within approximately +10degrees of perpendicular relative to the casing side wall.

If the top of casing is damaged, use the pipe cutter to square and repair the casing opening. Record the change in length to 0.1-inch relative to the original casing rise recorded on Form 130 or Report Log Form and report to the Terracon Project Manager.

Loosen the compression nut/flange on the lockable expansion cap, but do not remove the compression nut/flange. Insert the cap into the well opening. Holding the cap in place with one hand against the casing, with the other hand tighten the compression nut/flange. Tighten to maximum hand pressure.



Using a hand tool, rotate the compression nut/flange less than one revolution to align it with the padlock hasp of the cap. DO NOT over tighten. Excessive compression can split or crack PVC well casing and can damage or break the expansion cap inside steel well casing.

Insert the brass well tag onto the padlock shank, if applicable. Insert the keyed-alike padlock into the expansion cap.

Physically mark or tag the key with the project number, and the well identification if more than one type of keyed-lock is used for the project. Return the key(s) with documentation and return to the Project Manager for future access.

### DOCUMENTATION

### TSOP E.905

#### Terracon

Record any adjustments or repairs on completed Form 130 (Back) Monitoring Well Details and

FORM 130 - BACK

MONITORING WELL DETAILS	E	BORING	ELEV	ATION	S
<ol> <li>Fill in applicable dimensions and identify backfill materials</li> <li>Sketch screen installation (if more than one section used)</li> </ol>	Benchmar	k			
	Elevation			Assumed	Giver
	Boring/ Turning Point	Backsight	Height Instrument	Foresight	Elevation
Hotl Type         Hotl Type					
Protective steel cover?  Yes (Lock Type) Type (Key number	)				
□ No					
Screen slot size or fabric type Driller Driller					

return with key to Project Manager.

### OTHER SUPPORTING DOCUMENTS

TSOP E.700	Well Construction – Temporary	August 2000
<b>TSOP E.800</b>	Well Construction – Permanent	November 2001

## E.910 WELL SECURITY – TYPE C (Protective Casing)

### LAST REVIEW OR REVISION: June 2010

- **OBJECTIVE:** To provide standard procedures for physically securing monitoring wells in the field where the potential threat of persons deliberately contaminating groundwater used for sampling and testing through the surface well casing is moderate to high or the probability of inadvertent structural damage to the well casing is likely.
- The procedure provides an indicator if the well has been entered by other than authorized project personnel intent on unauthorized access to the well, allowing the sampler to halt sampling or flag samples as suspect.
- This procedure is of moderate cost, requires a moderate technical efficiency on the part of the field person and provides a high degree of wellhead protection for sample integrity. It provides a high level of protection against damage, inadvertent or deliberate, for exposed well casing from surface traffic.
- This procedure should be conducted at the time of original well construction, but no later than 48 hours afterward. Until final security construction can be completed, the wellhead should be temporarily secured using *TSOPs E.900 Well Security- Type A* (*Simple Cap*) or *E.905 Well Security Type B* (Locking Expansion Cap).

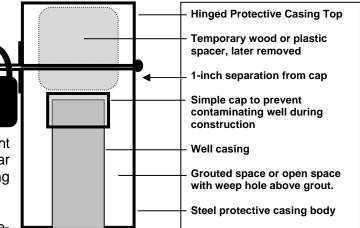
### EQUIPMENT

- Copy of field well construction log.
  - Terracon Form No. 130 MONITORING WELL DETAILS, see figure enclosed.
- Drilling or excavating equipment capable of soil removal from around the well casing to provide sufficient dimensions for construction, if the protective casing is not installed in the original boring annulus at the time of drilling and well construction.
- Steel protective casing of commercial manufacture matched by Terracon Project Manager to diameter of monitoring well. The protective casing will extend over and enclose the well casing. The protective casing will be of heavy gauge metal, if painted or coated against corrosion, the coating will be commercially applied and cured well before field mobilization – DO NOT re-paint protective casings in the field to prevent potential chemical contamination of the well.
- A 0.125-inch diameter weep hole will be drilled into the protective casing at a distance of at least 24-inches below the top edge of the opened protective casing. The weep hole allows water accumulation from sampling or intruding rain to escape the interior of the protective casing. <u>Accumulating water confined within the protective casing can crush the interior well casing when it freezes.</u> Alternatively, the weep hole can be drilled after construction for best position using a portable battery-powered drill.
- Well casing caps or plugs, expansion or threaded.

- With inert or chemically compatible expanding gasket of material not affected as to loss of expansion or damage down to temperatures of -10 degrees Fahrenheit.
- Simple slip caps as used in TSOP E.900
- Keyed-alike padlocks, hardened >0.25-inch diameter hasp required.
- Hand-operated compression pipe cutter appropriate to material and without interference to the integrity of the sampling (requires no solvents or oils).
- Wellhead marking materials ONLY FOR EXTERNAL APPLICATION AWAY FROM WELLHEAD CASING OPENING
  - Compatible markers or adhesive labels to permanently mark cap without solvent/residue interference with potential chemicals of analysis.
  - 2-inch diameter brass tag-and-ring to physically attach to padlock or cap
  - Steel number punch set for brass tags, if applicable.
  - Hammer for punch set, if applicable.
  - Small metal block or hand anvil for punch, set.
  - Stencils and spray latex paint for marking closed casing, no petroleum-based paint.
- Soft wire and wire cutters
- Pliers or small box end wrench
- Battery-powered drill and 1/8<sup>th</sup> (0.125) –inch steel drill bit, if constructing weep holes in the field.
- Casing spacer(s) of at least one-half the interior diameter of the protective steel casing, flat on top and bottom and constructed of plastic or wood. The spacer will rest temporarily on top of the sealed well casing inside protective casing's hinged top when closed. The spacer should position the top of the well casing 2-inches, <u>+0</u>.5 inches, below the top edge of the opened steel protective casing. This spacer is later removed after the cement grout has set and holds the protective casing in place.

### PROCEDURES

Visually inspect the top well casing after well construction and placement of the bentonite seal in the boring annulus. Inspect the casing opening for adhered soils, sand or other materials which might interfere with the expanding seal. Clear and clean appropriate with project cleaning or decontamination procedures.



The top of casing should be visually true-

and-round, visually free of chips, cracks or other physical

damage which could interfere with the expanding gasket of the locking cap. The casing should be

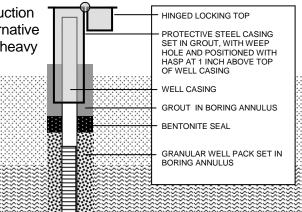
### TSOP E.910

#### Terracon

flat and level across the opening, within approximately  $\pm 10$ -degrees of perpendicular relative to the casing side wall. If the top of casing is damaged, use the pipe cutter to square and repair the casing opening. Record the change in length to 0.1-inch relative to the original casing riser length recorded on Form 130.

Temporarily seal the well casing during well construction using a simple cap as in TSOP E.905. An alternative method is to cover the top of the casing with heavy gauge (4-mil or greater) clean plastic bags held in place by rubber bands. Avoid use of tape to prevent residual adhesives in the immediate vicinity of the well opening.

Have at hand the spacer and pre-cut piece of soft steel wire approximately 4-inches in length. Within 10 minutes of placement of the cement grout to surface, lower the steel protective casing over the well casing. Take



care to visually place the well casing at the center of the protective casing. While one person holds the protective casing centered, the second person should place the spacer on top of the well casing, carefully close the hinged cover and tightly wire the padlock flanges together. Wire the padlock flanges such that the padlock hasp can still passthrough the hole. DO NOT rely on the padlock to hold the hinged cover and protective casing in position while the grout hardens.

Mark, label or stencil the well identification on the exterior of the protective casing. Insert the brass well tag onto the padlock shank, if applicable. Place the padlock hasp through the padlock flanges and secure.

Physically mark or tag the key with the project number, and the well identification if more than one type of keyed-lock is used for the project. Return the key(s) with documentation and return to the Project Manager for future access.

The wire and spacer may be removed after the grout has cured (hardened) for at least 8 hours.

<u>SPECIAL NOTE:</u> If project specifications require the interior space of the protective casing to be filled with grout, do so after the initial construction. Thereafter DO NOT introduce neat cement grout (pure Portland cement and water), the heat of hydration and expansion of the grout confined within the steel casing will crush PVC well casings. Use grout consisting of an approximate 1:3 dry volume mixture of bentonite powder and Portland cement, respectively. Add water to a pourable slurry. With the simple well cap still in place, introduce slurry by funnel into the open space around the well casing. Take care not to spill grout on the well cap or cover/hinge assembly. Bring the grout volume to a height of 3-inches,  $\pm 0.5$ -inch, below the bottom of the cap to allow for some slight expansion of the grout during hydration. DO NOT inadvertently grout the cap in place.

### DOCUMENTATION

FORM	130	- BACK

MONITORING WELL DETAILS	BORING ELEVATIONS
<ol> <li>Fill in applicable dimensions and identify backfill materials</li> <li>Sketch screen installation (if more than one section used)</li> </ol>	Benchmark
	Elevation Assumed Give
	Boring/ Turning Height Point Backsight Instrument Foresight Elevation
Hetl Type       Top Cap         Yop Cap       2.5' Riser         5' Riser       5' Riser         10' Riser       2.5' Screen         5' Screen       5' Screen         5' Screen       5' Screen         Bottom Point       Sand         Bentonite       Grout         Concrete       Protective Casing         Bottom Plates	
Protective steel cover?  Yes (Lock Type)	
Type (Key number	
Screen slot size or fabric type	
Well Installation Accepted by Driller	

Record dimensions, or other adjustments or repairs on completed Form 130 (Back) *Monitoring Well Details* and return with key to Project Manager.

### OTHER SUPPORTING DOCUMENTS

TSOP E.900	Well Security – Type A (Simple Cap)	
TSOP E.800	Well Construction – Permanent	

### E.920 WELL SECURITY – TYPE D (Flushmount)

### LAST REVIEW OR REVISION: June 2010

- **OBJECTIVE and INTENT:** To provide standard procedures for physically securing monitoring wells in the field where the potential threat of persons deliberately contaminating groundwater used for sampling and testing through the surface well casing is moderate to high and the probability of inadvertent structural damage by traffic is very high or client operations require no interference with traffic for safety issues.
- The procedure provides an indicator if the well has been entered by other than authorized project personnel intent on unauthorized access to the well, allowing the sampler to halt sampling or flag samples as suspect.
- This procedure is of moderate to high cost, requires a moderate to high technical efficiency on the part of field staff and provides a high degree of wellhead protection for sample integrity. It provides a high level of protection against mechanical damage, inadvertent or deliberate, from surface traffic. Flushmount construction requires field staff to exercise greater care in construction and to be more precise in positioning the top of well casing relative to final elevation. The ability to shorten "too long" or trim damaged well casing is greatly restricted inside the flushmount covers. Flushmount well covers have a higher degree of post-construction maintenance to prevent possible contamination of the groundwater system being evaluated. <u>Flush mount wells should NOT be the first choice of protection and should only be used where necessity or the client specifications demand, see Special Notes below.</u>
- This procedure should be conducted at the time of original well construction, but no later than 48 hours afterward.
- <u>Special Technical Note 1:</u> Although designed with water-resistant gaskets, flushmount well covers are historically subject to surface water intrusion; from rain, flood, spillage during groundwater purging/sampling or floor washings. Seals deteriorate and cast-metal lids can crack under temperature fluctuations. Water collecting inside the cover basin, possibly contaminated by contact with surface, can then enter a vented well casing and contaminate the groundwater intended for sampling. Accumulated water can readily freeze if the well is constructed outside of heated structures. Confined within the cover, expanding ice can crush/crack the well casing and allow water into the well when it thaws. While the well casing may often resist freeze-thaw damage to some extent, the accumulated water frozen over the well casing will prevent access to the well without extraordinary measures. It is imperative that the seals be maintained and replaced regularly and that the covers be tightened uniformly when sealing.
- <u>Special Safety Note 2:</u> As a precaution against seal failure, the well casing cannot be vented and must also be permanently secured using *TSOP E.905 Well Security Type B (Locking Expansion Cap)*. However, different from E.905, the tightening of the compression nut/flange should be hand tight plus <u>two</u> revolutions to align the hasp. While this provides a watertight seal for the well casing inside the flushmount cover, the unvented column of air

trapped inside the well casing has no where to escape as groundwater levels fluctuate up and down in the well casing. Pressure can build up and care should be taken in removing the expansion caps for sampling. In addition, in some hydrologic conditions the trapped air column, if not dissipated through un-submerged well screen, can artificially depress water levels measured within the well casing. <u>Water level measurements may NOT be</u> representative of stabilized groundwater levels in the subsurface.

### EQUIPMENT

- Copy of field well construction log.
  - Terracon Form No. 130 MONITORING WELL DETAILS, see figure enclosed.
- Drilling or excavating equipment capable of soil removal from around the well casing to provide sufficient dimensions for construction, if the flushmount cover is not installed in the original boring annulus at the time of drilling and well construction.
- Steel flushmount vault and cover of commercial manufacture matched by Terracon Project Manager to diameter of monitoring well. The flushmount vault and cover will extend over and enclose the well casing. The flushmount vault and cover will be of heavy gauge metal, if painted or coated against corrosion, the coating will be commercially applied and cured well before field mobilization – DO NOT re-paint flushmount vault and covers in the field to prevent potential chemical contamination of the well.
- Well casing expansion locking plugs with inert or chemically compatible expanding gasket of material not affected as to loss of expansion or damage down to temperatures of -10 degrees Fahrenheit.
- Keyed-alike padlocks, hardened hasp required.
- Hand-operated compression pipe cutter appropriate to material and without interference to the integrity of the sampling (requires no solvents or oils).
- Wellhead marking materials ONLY FOR EXTERNAL APPLICATION AWAY FROM WELLHEAD CASING OPENING
  - Compatible markers or adhesive labels to permanently mark flushmount cover without solvent/residue interference with potential chemicals of analysis.
  - 2-inch diameter brass tag-and-ring to physically attach to padlock or cap
  - Steel number punch set for brass tags, if applicable.
  - Hammer for punch set, if applicable.
  - Small metal block or hand anvil for punch, set.
- Special socket tool by manufacturer matched to flushmount cover security bolts.
- Piece of 0.5- to 0.75-inch thick plywood or lumber to use as spacer/gauge for well cap clearance.

### PROCEDURES

Visually inspect the top well casing after well construction and placement of the bentonite seal in the boring annulus. Inspect the casing opening for adhered soils, sand or other materials which might interfere with the expanding seal. Clear and clean appropriate with project cleaning or decontamination procedures.

The top of casing should be visually true-and-round, visually free of chips, cracks or other physical damage which could interfere with the expanding gasket of the locking cap. The casing should be flat and level across the opening, within approximately  $\pm 10$ -degrees of perpendicular relative to the casing side wall. The top of casing should be at final design elevation, considering these three essential items in exercising professional judgment in the field;

- With the flushmount vault and cover bolted in place placed over the well, the locking expansion plug <u>and padlock</u> should have no less than 0.5-inch clearance from the bottom of the vault cover. <u>Vault designs differ!</u> Do not assume the same amount of clearance for all vault/cover assemblies (e.g., One manufacturer incorporates ribbed strengthening members cast in the underside of the flushmount cover that can increase maximum cross-sectional thickness for clearance by ~1-inch).
- 2. Trimming the well casing height should always be done <u>prior</u> to grouting in the vault/cover assembly. The top of cover is not truly flushmount, it should rise approximately 0.125-inches above surface to prevent small amounts of rain or standing water on the cover as a low place and yet clear snowplows, sweepers, etc.
- 3. Do not set the top of casing too low to allow for clearance, it should be set as high as possible inside the vault. This protects against minor water accumulation and freezing inside the vault (i.e., minor infiltration through cracked cover or deteriorated seal between sampling events) which might occur, yet maximizes the potential for keeping the lock and expansion cap from being covered by water/ice.

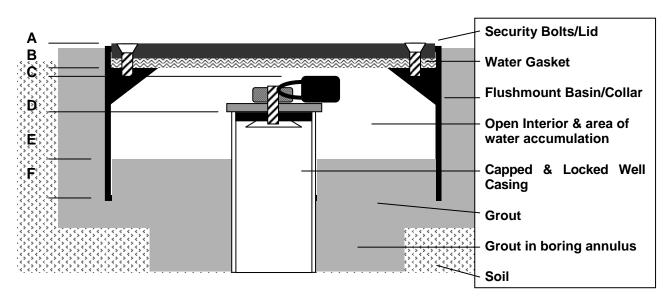
If the top of casing is damaged, use the pipe cutter to square and repair the casing opening. Record the change in length to 0.1-inch relative to the original casing riser length recorded on Form 130. Seal the well casing at all times during well construction using the expansion locking cap with padlock in place as in TSOP E.905.

Have at hand a piece of 0.5- to 0.75-inch thick plywood to use as a temporary spacer/gauge for clearance. For example, referencing figure below;

A - Ground surface	0.0 inches elevation
B - Flushmount cover thickness	-0.5 inches
C - Clearance to cover from cap/lock (spacer)	-0.5 inches
D - Thickness of cap/lock above top of casing	-1.0 inches
Top of well casing from surface	-2.0 inches

Place the final cement grout to an elevation of surface minus the length of the vault assembly collar less ~2 inches. The intent is to bring the grout up into and around the base/collar of the flushmount basin/assembly approximately 2 inches to seal the assembly, yet not interfere with the locking expansion cap and still provide the maximum interior volume for small amounts of unplanned water infiltration. For example, referencing figure below;

A - Ground surface	0.0 inches elevation
Length of collar/basin	-8.5 inches
F - Distance grout rise inside the basin/collar	+2.0 inches
Top of grout from surface	-6.5 inches from surface



With the spacer held in place inside the cover, set the closed (lid bolted in place) flushmount cover over the well and into the grout no later than 10 minutes after initial grout placement. Although the well casing should already be centered in the middle of the boring and area removed by hand tools or coring (in the case of slabs), take care to visually place the well casing at the center of the flushmount vault and cover. Allow the grout to undergo a firm set, typically 15 to 30 minutes for cement mixtures.

Grout the exterior of the basin, free of water and debris to surface with a stiff, but flowable neat cement grout. Standard air-entrained mixtures or commercial concrete may be used against freeze-thaw if approved by a Terracon geotechnical engineer. Slope grout upward to edges of vault cover for final finishing.

Mark, label or stencil the well identification on the exterior of the flushmount vault and cover, and/or insert the brass well tag onto the padlock shank. Place the padlock hasp through the padlock flanges and secure.

Physically mark or tag the key with the project number, and the well identification if more than one type of keyed-lock is used for the project. Return the key(s) with documentation and return to the Project Manager for future access.

FORM 130 - BACK

MONITORING WELL DETAILS		orante	A has been been W.	ATION	5
. Fill in applicable dimensions and identify backfill materials 2. Sketch screen installation (if more than one section used)	Benchmar	<			
	Elevation	Elevation		Assumed	
	Boring/ Turning Point	Backsight	Height Instrument	Foresight	Elevation
Hetl Type         Hetl Type					
Protective steel cover?  Yes (Lock Type) Type (Key number No					
Screen slot size or fabric type					

Take care not to spill grout on the well cap or cover/hinge assembly. Bring the grout volume to a height of 3-inches,  $\pm$ -inch, below the bottom of the cap to allow for some slight expansion of the grout during hydration. DO NOT inadvertently grout the cap in place.

### DOCUMENTATION

Record dimensions, or other adjustments or repairs on completed Form 130 (Back) *Monitoring Well Details* and return with key to Project Manager.

### **OTHER SUPPORTING DOCUMENTS**

TSOP E.900	Well Security – Type B (Locking Expansion Cap)	
TSOP E.800	Well Construction – Permanent	

## E.1300 WELL DEVELOPMENT – VOLUMETRIC

### Last Review or Revision: June 2010

### Objective

- The objective of monitoring well development is to remove adulterated water in the well, sand pack, and surrounding saturated formations that formed during drilling and well installation. Development should be considered the final step in completing a groundwater monitoring well.
- Initially, well development should purge any drilling fluids or other foreign materials from the monitoring well. Development should be performed as soon as practical after the well installation, but no sooner than 48 hours after the grouting is complete.

### Equipment

- Development should be accomplished with the use of a pump, bottom filling bailer, or nitrogen lift device. Measuring equipment should be consistent with that specified by the project Manager or Equipment Manager at mobilization and consistent with TSOP E.1820.
- Protective safety equipment will as specified in the program of assessment and as directed by the Terracon Site Health & Safety Plan consistent with materials and concentrations specified therein.

### Procedure

- Determine the geometry of the monitoring well (i.e., measure static water level, depth of well, determine volume of fluid lost while drilling.) See attached form.
- Determine the volume of one "standing volume" of water in the well, defined as the volume of standing water within the well casing plus the volume within the gravel pack (assuming 30% porosity).
- For monitoring wells where the boring was made without the use of drilling fluids, three (3x) times the standing water volume in the well should be removed.
- For those wells where the boring was advanced using drilling fluids, remove five (5x) times the standing volume plus three (3x) times the volume of water lost during drilling.
- If low permeability materials prevent the well from yielding adequate volumes of water, alternative development procedures will be implemented at the direction of the Project Manager (i.e. removal of fluids to dryness, allow well to recharge and then recheck quality of fluids).

### **TSOP E.1300**

- Complete the monitoring well development form if supplied by the Project Manager or record well development details in the field log book. Pertinent data will vary based on the parameters and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.
- Store or discharge the development water consistent with direction of the Project Manager and as allowed by law. Reference E.2220.

### Attached Supporting Documentation

- Form 130 Log of Boring No., Monitoring Well Details, and Boring Elevations
- Terracon Volumetric Chart for Well Development
- Brainard Kilman Field Facts

### Other References

- a) ASTM D4448 Standard Guide For Sampling Groundwater Monitoring Wells
- b) TSOP E.1900 Groundwater Sampling Bailer
- c) TSOP E.2000 Low Flow Pumping

### E.1400 WELL DEVELOPMENT – PARAMETRIC

Last Review or Revision: June 2010

### 1. OBJECTIVE

- The objective of monitoring well development is to remove adulterated water in the well, sand pack, and surrounding saturated formations which formed during drilling and well installation. Development should be considered the final step in completing a groundwater monitoring well.
- Initially, well development should purge any drilling fluids or other foreign materials from the monitoring well. Development should be performed as soon as practical after the well installation, but no sooner than 48 hours after the grouting is complete.
- Parametric stabilization is necessary when the groundwater may not be "clear" due to macroscopic particulate movement in the soil/fill matrix. This is not uncommon in old industrial fills containing flyash or carbon black. Parametric attempts to identify that the condition of physical parameters in formation water entering the well is reasonably comparable to water in the adjacent formation.
- 1. EQUIPMENT
- Development should be accomplished with the use of a pump, bottom filling bailer, or nitrogen lift device. Measuring equipment should be consistent with that specified by the project Manager or Equipment Manager at mobilization and consistent with TSOP E.1820.
- Protective safety equipment will be utilized as specified in the program of assessment and as directed by the Terracon Site Health & Safety Plan consistent with materials and concentrations specified therein.
- 2. PROCEDURE
- Determine the geometry of the monitoring well (i.e., measure static water level, depth of well, determine volume of fluid lost while drilling.) See attached form.
- Determine the volume of one "standing volume" of water in the well, defined as the volume of standing water within the well casing plus the volume within the gravel pack (assuming 30% porosity).

- Sample and record a baseline series of measurements for water in the well casing for;
  - Temperature; degrees Fahrenheit (F)
  - pH; standard units

**TSOP E.1400** 

- Specific Conductance; micromhos (μmhos)
- Turbidity; nephelometric turbidity units (NTUs)
- Remove three (3) additional standing volumes from the well, recording a measurement series at the end of each volume.
- If each of the parameters have demonstrated repeatability for three (3) consecutive readings the well will be considered developed comparable to formation water conditions;
  - Temperature; + 1 degree F
  - pH; + 0.3 standard units
  - Specific Conductance; <u>+</u> 20 μmhos
  - Turbidity; <u>+</u> 10 NTUs
- If parameters for each have not stabilized, continue to remove and measure non-stabilized parameters each additional standing volume, up to a total of ten (≤10x) the initial standing well volume. At this point the well will be considered reasonably comparable in condition to conditions of the surrounding formation water.
- For those wells where the boring was advanced using drilling fluids, remove the volume of water lost during drilling plus the one standing volume prior to taking the baseline measurement series.
- If low permeability materials prevent the well from yielding adequate volumes of water, alternative development procedures using TSOP E.2000 will be implemented at the request of the Project Manager.
- Complete the monitoring well development form.
- Store or discharge the development waters consistent with direction of the project Manager and as allowed by law. Reference E.2220.
- 4. ATTACHED SUPPORTING DOCUMENTATION
- Brainard Kilman Field Facts
- 5. OTHER REFERENCES
  - a) ASTM D4448 Standard Guide For Sampling Groundwater Monitoring Wells
  - b) TSOP E.1900 Groundwater Sampling Bailer

#### Terracon

- C)
- d)
- TSOP E.2000 Low Flow Pumping TSOP E.530 pH Field Screening Water TSOP E.540 Conductivity Field Screening Water TSOP E.570 Temperature TSOP E.580 Turbidity Field Screening Water e)
- f)
- ġ)

# E.1500 BORING ABANDONMENT – COMMERCIAL SEALANT

### Last Review or Revision: June 2010

### **Objective and Application**

To permanently close soil borings consistent with industry practice and close the soil boring to prevent its serving as a vertical conduit for movement of environmental impacts through soils. Soil borings of 2-inch diameter or greater are considered borings within the definition of this TSOP.

### Equipment

Hand tools appropriate to the job.

Commercially-available sealant materials for well abandonment. Terracon preference for nonslurry fill will be chipped Benseal®, high yield Wyoming bentonite or equivalent.

### **Procedures**

Backfill the soil boring with a mixture of soil cuttings and bentonite or other sealant material as required by state law (i.e. backfill with hydrated bentonite to near surface and cemtn the upper two feet). When specified by the project manager, attempt to place soil cuttings back in the borehole in the order that the soil was removed so that soil is returned to the approximate depth from which it originated.

### **Attached Supporting Documents**

- Volume of Soil Boring, Annulus around 2" and 4" Casings, and Grout Mixtures
- Brainard Kilman Field Facts

### **Other Supporting Documents**

• **ASTM D5299-99** Standard Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities.

### VOLUME OF SOIL BORING

CUBIC	FEET	PER	LINE	AR F	юот	
			_			

GALLONS PER LINEAR FOOT	
	1

	DIAMETER OF SOIL BORING									
	DIAIVIE	IERO	F SUIL	INSIDE DIAMETER OF						
				HOLLOV						
LENGTH		I	1	3.25	4.25	6.25				
						10.05				
(ft.)	2	4	6	7.25	8.25	10.25				
1	0.028	0.088	0.201	0.29	0.37	0.57				
2	0.06	0.18	0.4	0.6	0.7	1.1				
3	0.08	0.26	0.6	0.9	1.1	1.7				
4	0.11	0.35	0.8	1.2	1.5	2.3				
5	0.14	0.44	1.0	1.5	1.9	2.9				
6	0.17	0.53	1.2	1.7	2.2	3.4				
7	0.20	0.62	1.4	2.0	2.6	4.0				
8	0.22	0.70	1.6	2.3	3.0	4.6				
9	0.25	0.79	1.8	2.6	3.3	5.1				
10	0.28	0.88	2.0	2.9	3.7	5.7				
11	0.31	0.97	2.2	3.2	4.1	6.3				
12	0.34	1.06	2.4	3.5	4.4	6.8				
13	0.36	1.14	2.6	3.8	4.8	7.4				
14	0.39	1.23	2.8	4.1	5.2	8.0				
15	0.42	1.32	3.0	4.4	5.6	8.6				
16	0.45	1.41	3.2	4.6	5.9	9.1				
10	0.48	1.50	3.4	4.9	6.3	9.7				
18	0.50	1.58	3.6	5.2	6.7	10.3				
10	0.53	1.67	3.8	5.5	7.0	10.8				
20	0.56	1.76	4.0	5.8	7.4	11.4				
20						12.5				
	0.62	1.94	4.4	6.4	8.1					
24	0.67	2.11	4.8	7.0	8.9	13.7				
25	0.70	2.20	5.0	7.3	9.3	14.3				
26	0.73	2.29	5.2	7.5	9.6	14.8				
28	0.78	2.46	5.6	8.1	10.4	16.0				
30	0.84	2.64	6.0	8.7	11.1	17.1				
32	0.90	2.82	6.4	9.3	11.8	18.2				
34	0.95	2.99	6.8	9.9	12.6	19.4				
35	0.98	3.08	7.0	10.2	13.0	20.0				
36	1.01	3.17	7.2	10.4	13.3	20.5				
38	1.06	3.34	7.6	11.0	14.1	21.7				
40	1.12	3.52	8.0	11.6	14.8	22.8				
45	1.3	4.0	9.0	13.1	16.7	25.7				
50	1.4	4.4	10.1	14.5	18.5	28.5				
55	1.5	4.8	11.1	16.0	20.4	31.4				
60	1.7	5.3	12.1	17.4	22.2	34.2				
65	1.8	5.7	13.1	18.9	24.1	37.1				
70	2.0	6.2	14.1	20.3	25.9	39.9				
75	2.1	6.6	15.1	21.8	27.8	42.8				
80	2.2	7.0	16.1	23.2	29.6	45.6				
85	2.4	7.5	17.1	24.7	31.5	48.5				
90	2.5	7.9	18.1	26.1	33.3	51.3				
95	2.7	8.4	19.1	27.6	35.2	54.2				
100	2.8	8.8	20.1	29.0	37.0	57.0				
125	3.5	11.0	25.1	36.3	46.3	71.3				
150	4.2	13.2	30.2	43.5	55.5	85.5				
175	4.9	15.4	35.2	50.8	64.8	99.8				
200	5.6	17.6	40.2	58.0	74.0	114.0				
200	0.0			00.0						

	DIAMETER OF SOIL BORING										
				INSIDE DIAMETER OF HOLLOW STEM AUGER							
			1	3.25	4.25	6.25					
LENGTH	-										
(ft.)	2	4	6	7.25	8.25	10.25					
1	0.17	0.66	1.5	2.14	2.78	4.29					
2 3	0.3	1.3	3.0	4.3	5.6	8.6					
	0.5	2.0	4.5	6.4	8.3	12.9					
4	0.7	2.6	6.0	8.6	11.1	17.2					
5	0.9	3.3	7.5	10.7	13.9	21.5					
6	1.0	4.0	9.0	12.8	16.7	25.7					
7	1.2	4.6	10.5	15.0	19.5	30.0					
8	1.4	5.3	12.0	17.1	22.2	34.3					
9	1.5	5.9	13.5	19.3	25.0	38.6					
10	1.7	6.6	15.0	21.4	27.8	42.9					
11	1.9	7.3	16.5	23.5	30.6	47.2					
12	2.0	7.9	18.0	25.7	33.4	51.5					
13	2.2	8.6	19.5	27.8	36.1	55.8					
14	2.4	9.2	21.0	30.0	38.9	60.1					
15	2.6	9.9	22.5	32.1	41.7	64.4					
16	2.7	10.6	24.0	34.2	44.5	68.6					
17	2.9	11.2	25.5	36.4	47.3	72.9					
18	3.1	11.9	27.0	38.5	50.0	77.2					
19	3.2	12.5	28.5	40.7	52.8	81.5					
20	3.4	13.2	30.0	42.8	55.6	85.8					
22	3.7	14.5	33.0	47.1	61.2	94.4					
24	4.1	15.8	36.0	51.4	66.7	103.0					
25	4.3	16.5	37.5	53.5	69.5	107.3					
26	4.4	17.2	39.0	55.6	72.3	111.5					
28	4.8	18.5	42.0	59.9	77.8	120.1					
30	5.1	19.8	45.0	64.2	83.4	128.7					
32	5.4	21.1	48.0	68.5	89.0	137.3					
34	5.8	22.4	51.0	72.8	94.5	145.9					
35	6.0	23.1	52.5	74.9	97.3	150.2					
36	6.1	23.8	54.0	77.0	100.1	154.4					
38	6.5	25.1	57.0	81.3	105.6	163.0					
40	6.8	26.4	60.0	85.6	111.2	171.6					
45	7.7	29.7	67.5	96.3	125.1	193.1					
50	8.5	33.0	75.0	107.0	139.0	214.5					
55	9.4	36.3	82.5	117.7	152.9	236.0					
60 65	10.2	39.6	90.0	128.4	166.8	257.4					
65	11.1	42.9	97.5	139.1	180.7	278.9					
70	11.9	46.2	105.0	149.8	194.6	300.3					
75	12.8	49.5	112.5	160.5	208.5	321.8					
80	13.6	52.8	120.0	171.2	222.4	343.2					
85	14.5	56.1	127.5	181.9	236.3	364.7					
90	15.3	59.4	135.0	192.6	250.2	386.1					
95	16.2	62.7	142.5	203.3	264.1	407.6					
100	17.0	66.0	150.0	214.0	278.0	429.0					
125	21.3	82.5	187.5	267.5	347.5	536.3					
150	25.5	99.0	225.0	321.0	417.0	643.5					
175	29.8	115.5	262.5	374.5	486.5	750.8					
200	34.0	132.0	300.0	428.0	556.0	858.0					

	CUBIC	EEET I	۷ PER LII			ANNUL	JS A	ROUND					ООТ	
			ER OF			2			GALLONS PER LINEAR FOOT DIAMETER OF SOIL BORING					
	L		ENOF		DIAMET				L		ENOF			
						AUGER							W STEM	
				3.25	4.25	6.25						3.25	4.25	6.25
LENGTH			1	5.25	7.20	0.25		LENGTH		l	l	0.20	7.20	0.25
(ft.)	2	4	6	7.25	8.25	10.25		(ft.)	2	4	6	7.25	8.25	10.25
(,)	0	0.065	0.178	0.26	0.25	0.54		1	0	0.491	1.33	1.91	2.55	4.06
2	0	0.005	0.178	0.20	0.34	1.1		2	0	1.0	2.7	3.8	2.55	4.00
2	0	0.13	0.4	0.3	1.0	1.6		3	0	1.5	4.0	5.7	7.7	12.2
4	0	0.20	0.7	1.0	1.4	2.2		4	0	2.0	5.3	7.6	10.2	16.2
5	0	0.33	0.9	1.3	1.7	2.7		5	0	2.5	6.7	9.6	12.8	20.3
6	0	0.39	1.1	1.6	2.0	3.2		6	0	2.9	8.0	11.5	15.3	24.4
7	0	0.46	1.2	1.8	2.4	3.8		7	0	3.4	9.3	13.4	17.9	28.4
8	Ő	0.52	1.4	2.1	2.7	4.3		8	0	3.9	10.6	15.3	20.4	32.5
9	Ő	0.59	1.6	2.3	3.1	4.9		9	0 0	4.4	12.0	17.2	23.0	36.5
10	Ő	0.65	1.8	2.6	3.4	5.4		10	0	4.9	13.3	19.1	25.5	40.6
11	0	0.72	2.0	2.9	3.7	5.9		11	0	5.4	14.6	21.0	28.1	44.7
12	Ő	0.78	2.1	3.1	4.1	6.5		12	0	5.9	16.0	22.9	30.6	48.7
13	0	0.85	2.3	3.4	4.4	7.0		13	0	6.4	17.3	24.8	33.2	52.8
14	0	0.91	2.5	3.6	4.8	7.6		14	0	6.9	18.6	26.7	35.7	56.8
15	0	0.98	2.7	3.9	5.1	8.1		15	0	7.4	20.0	28.7	38.3	60.9
16	0	1.04	2.8	4.2	5.4	8.6		16	0	7.9	21.3	30.6	40.8	65.0
17	0	1.11	3.0	4.4	5.8	9.2		17	0	8.3	22.6	32.5	43.4	69.0
18	0	1.17	3.2	4.7	6.1	9.7		18	0	8.8	23.9	34.4	45.9	73.1
19	0	1.24	3.4	4.9	6.5	10.3		19	0	9.3	25.3	36.3	48.5	77.1
20	0	1.30	3.6	5.2	6.8	10.8		20	0	9.8	26.6	38.2	51.0	81.2
22	0	1.43	3.9	5.7	7.5	11.9		22	0	10.8	29.3	42.0	56.1	89.3
24	0	1.56	4.3	6.2	8.2	13.0		24	0	11.8	31.9	45.8	61.2	97.4
25	0	1.63	4.5	6.5	8.5	13.5		25	0	12.3	33.3	47.8	63.8	101.5
26	0	1.69	4.6	6.8	8.8	14.0		26	0	12.8	34.6	49.7	66.3	105.6
28	0	1.82	5.0	7.3	9.5	15.1		28	0	13.7	37.2	53.5	71.4	113.7
30	0	1.95	5.3	7.8	10.2	16.2		30	0	14.7	39.9	57.3	76.5	121.8
32	0	2.08	5.7	8.3	10.9	17.3		32	0	15.7	42.6	61.1	81.6	129.9
34	0	2.21	6.1	8.8	11.6	18.4		34	0	16.7	45.2	64.9	86.7	138.0
35	0	2.28	6.2	9.1	11.9	18.9		35	0	17.2	46.6	66.9	89.3	142.1
36	0	2.34	6.4	9.4	12.2	19.4		36	0	17.7	47.9	68.8	91.8	146.2
38	0	2.47	6.8	9.9	12.9	20.5		38	0	18.7	50.5	72.6	96.9	154.3
40	0	2.60	7.1	10.4	13.6	21.6		40	0	19.6	53.2	76.4	102.0	162.4
45	0	2.9	8.0	11.7	15.3	24.3		45	0	22.1	59.9	86.0	114.8	182.7
50 55	0 0	3.3 3.6	8.9 9.8	13.0 14.3	17.0 18.7	27.0		50 55	0 0	24.6	66.5	95.5 105.1	127.5 140.3	203.0
						29.7			-	27.0	73.2			223.3 243.6
60 65	0	3.9 4.2	10.7	15.6 16.9	20.4 22.1	32.4 35.1		60 65	0	29.5 31.9	79.8 86.5		153.0 165.8	243.6
65 70	0 0	4.2 4.6	11.6 12.5	16.9	22.1	35.1		65 70	0 0	31.9	86.5 93.1	124.2	178.5	263.9
70 75	0	4.6 4.9	12.5	10.2	23.0 25.5	37.8 40.5		70	0	34.4 36.8	93.1 99.8	143.3	176.5	204.2 304.5
80	0	5.2	14.2	20.8	27.2	43.2		80	0	39.3	106.4	152.8	204.0	324.8
85	0	5.5	15.1	20.0	28.9	45.9		85	0	41.7	113.1	162.4	216.8	345.1
90	0	5.9	16.0	23.4	30.6	43.9		90	0	41.7	119.7	171.9	229.5	365.4
90 95	0	6.2	16.9	24.7	32.3	51.3		90 95	0	46.6	126.4	181.5	242.3	385.7
100	0	6.5	17.8	26.0	34.0	54.0		100	0	49.1	133.0	191.0	255.0	406.0
125	Ő	8.1	22.3	32.5	42.5	67.5		125	0	61.4	166.3	238.8	318.8	507.5
150	0	9.8	26.7	39.0	51.0	81.0		150	0	73.7	199.5	286.5	382.5	609.0
175	0	11.4	31.2	45.5	59.5	94.5		175	0	85.9	232.8	334.3	446.3	710.5
200	0	13.0	35.6	52.0	68.0			200	0	98.2	266.0	382.0	510.0	812.0
200	5	.0.0	00.0	52.0	00.0			200	5	00.2		002.0	0.0.0	5.2.0

#### **VOLUME OF ANNULUS AROUND 2" CASING**

	D	IAMET	ER OF		SOIL BORING							
				INSIDE DIAMETER OF								
				HOLLO	V STEM	AUGER						
				3.25	4.25	6.25						
LENGTH												
(ft.)	2	4	6	7.25	8.25	10.25						
1	0	0	0.11	0.2	0.26	0.46						
2	0	0	0.2	0.4	0.5	0.9						
3	0	0	0.3	0.6	0.8	1.4						
4	0	0	0.4	0.8	1.0	1.8						
5	0	0	0.6	1.0	1.3	2.3						
6	0	0	0.0	1.0	1.6	2.8						
7	0	0		1.4	1.8	3.2						
	0		0.8									
8		0	0.9	1.6	2.1	3.7						
9	0	0	1.0	1.8	2.3	4.1						
10	0	0	1.1	2.0	2.6	4.6						
11	0	0	1.2	2.2	2.9	5.1						
12	0	0	1.3	2.4	3.1	5.5						
13	0	0	1.4	2.6	3.4	6.0						
14	0	0	1.5	2.8	3.6	6.4						
15	0	0	1.7	3.0	3.9	6.9						
16	0	0	1.8	3.2	4.2	7.4						
17	0	0	1.9	3.4	4.4	7.8						
18	0	0	2.0	3.6	4.7	8.3						
19	0	0	2.1	3.8	4.9	8.7						
20	0	0	2.2	4.0	5.2	9.2						
22	0	0	2.4	4.4	5.7	10.1						
24	0	0	2.6	4.8	6.2	11.0						
25	0	0	2.8	5.0	6.5	11.5						
26	0	0	2.9	5.2	6.8	12.0						
28	0	0	3.1	5.6	7.3	12.9						
30	0	0	3.3	6.0	7.8	13.8						
32	0	0	3.5	6.4	8.3	14.7						
34	0	0	3.7	6.8	8.8	15.6						
35	0	0	3.9	7.0	9.1	16.1						
36	0	0	4.0	7.2	9.4	16.6						
38	0	0	4.2	7.6	9.9	17.5						
40	0	0	4.4	8.0	10.4	18.4						
45	0	0	5.0	9.0	11.7	20.7						
50	0	0	5.5	10.0	13.0	23.0						
55	0	0	6.1	11.0	14.3	25.3						
60	0	0	6.6	12.0	15.6	27.6						
65	0	0	7.2	13.0	16.9	29.9						
70	0	0	7.7	14.0	18.2	32.2						
75	0	0	8.3	15.0	19.5	34.5						
80	0	0	8.8	16.0	20.8	36.8						
85	0											
		0	9.4	17.0	22.1	39.1						
90 95	0	0	9.9	18.0	23.4	41.4						
95	0	0	10.5	19.0	24.7	43.7						
100	0	0	11.0	20.0	26.0	46.0						
125	0	0	13.8	25.0	32.5	57.5						
150	0	0	16.5	30.0	39.0	69.0						
175	0	0	19.3	35.0	45.5	80.5						
200	0	0	22.0	40.0	52.0	92.0						

### VOLUME OF ANNULUS AROUND 4" CASING CUBIC FEET PER LINEAR FOOT GALLONS PER LINEAR FOOT

DIAMETER OF SOLL BORING           INSIDE DIAMETER OF HOLLOW STEM AUGER           INSIDE DIAMETER OF HOLLOW STEM AUGER           SAS 425 6.25           LENGTH (ft.)         2         4         6           1         0         0         0         10           1         0         0         10         3.25         8.25         10.25           1         0         0         3.25         4.25         10.25           1         0         0         10         10.25           1         0         0         10         10           0         0         10         10         10           0         0         10         10           0         0         10           1         10         10           10 <th colsp<="" th=""><th></th><th colspan="12">GALLONS PER LINEAR FOOT</th></th>	<th></th> <th colspan="12">GALLONS PER LINEAR FOOT</th>		GALLONS PER LINEAR FOOT											
HOLLOW STEM - UGER           LENGTH         I         I         I           (ft.)         2         4         6         7.25         8.25         10.25           1         0         0         0.84         1.48         1.99         3.46           2         0         0         1.77         3.0         3.9         6.9           3         0         0         2.5         4.4         5.9         10.4           4         0         0         3.44         5.9         17.3         13.8           5         0         0         5.9         10.4         13.7         24.2           7         0         0         5.9         10.4         13.7         24.2           6         0         0         5.9         10.4         13.7         24.2           7         0         0         1.48         19.5         34.6           111         0         0         10.9         12.2         34.4         41.5           13         0         0         11.8         20.7         27.3         48.4           15         0         0         12.6         32.5		E	DIAME	TER O										
LENGTH (ft.)         2         4         6         7.25         8.25         10.25           1         0         0         0.84         1.48         1.95         3.46           2         0         0         1.7         3.0         3.9         6.9           3         0         0         2.5         4.4         5.9         10.4           4         0         0         3.4         5.9         7.8         13.8           5         0         0         4.2         7.4         9.8         17.3           6         0         0         5.9         10.4         13.7         24.2           8         0         0         5.9         10.4         13.7         24.2           9         0         0         7.6         13.3         17.6         31.1           10         0         0         9.2         16.3         21.5         38.1           11         0         0         11.8         20.7         7.3         48.4           11         0         0         11.8         20.7         31.4         55.4           13         0         0         15.														
LENGTH (ft.)         2         4         6         7.25         8.25         10.25           1         0         0         0.84         1.48         1.95         3.46           2         0         0         1.7         3.0         3.9         6.9           3         0         0         2.5         4.4         5.9         10.4           4         0         0.34         5.9         7.8         13.8           5         0         0         4.2         7.4         9.8         17.3           6         0         0         5.9         10.4         13.7         24.2           8         0         0         6.7         11.8         15.6         27.7           9         0         0         7.6         13.3         17.6         31.1           10         0         0         8.4         14.8         19.5         34.6           11         0         0         12.6         22.3         35.1           13         0         0         11.8         20.7         31.2         55.4           14         0         0         11.4         23.7 <t< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td></t<>														
(ft.)         2         4         6         7.25         8.25         10.25           1         0         0         1.48         1.95         3.46           2         0         0         1.7         3.0         3.9         6.9           3         0         0         2.5         4.4         5.9         10.4           4         0         0         3.4         5.9         7.8         13.8           5         0         0         5.9         10.4         13.7         24.2           8         0         0         5.9         10.4         13.7         24.2           8         0         0         6.7         11.8         15.6         27.7           9         0         0         7.6         13.3         17.6         31.1           10         0         9.2         16.3         21.5         38.1           12         0         0         10.1         17.8         23.4         41.5           13         0         0         11.8         20.7         27.3         48.4           15         0         0         12.6         23.2         58.8					3.25	4.25	6.25							
1         0         0         0.84         1.48         1.95         3.46           2         0         0         1.7         3.0         3.9         6.9           3         0         0         2.5         4.4         5.9         10.4           4         0         0         3.4         5.9         7.8         13.8           5         0         0         4.2         7.4         9.8         17.3           6         0         5.9         10.4         13.7         24.2           8         0         0         6.7         11.8         15.6         27.7           9         0         0         7.6         13.3         17.6         31.1           10         0         9.2         16.3         21.5         38.1           12         0         0         10.9         19.2         25.4         45.5           13         0         0         12.6         22.2         29.3         51.9           16         0         13.4         23.7         31.2         55.4           17         0         14.3         25.2         33.2         58.8		-		_										
2         0         0         1.7         3.0         3.9         6.9           3         0         0         2.5         4.4         5.9         10.4           4         0         0         3.4         5.9         7.8         13.8           5         0         0         4.2         7.4         9.8         17.3           6         0         0         5.9         10.4         13.7         24.2           8         0         0.6.7         11.8         15.6         27.7           9         0         0         7.6         13.3         17.6         31.1           10         0         9.2         16.3         21.5         38.1           12         0         0         10.1         17.8         23.4         41.5           13         0         0         12.6         22.2         29.3         51.9           14         0         0         13.4         23.7         31.2         55.4           14         0         0         13.4         23.7         31.2         55.4           15         0         0         14.3         25.2         3		2	4											
3002.54.45.910.44003.45.97.813.85004.27.49.817.36005.08.911.720.87005.910.413.724.28006.711.815.627.79007.613.317.631.110008.414.819.534.611009.216.321.538.1120010.117.823.441.5130010.919.225.445.0140013.423.731.255.4150014.325.233.258.8180015.126.635.162.3190016.028.137.165.720018.532.642.976.1240020.235.546.883.0250021.037.048.886.5260021.838.550.790.028022.544.458.5103.8320025.244.458.5103.8320025.274.1131.5340033.659.27														
4         0         0         3.4         5.9         7.8         13.8           5         0         0         4.2         7.4         9.8         17.3           6         0         0         5.9         10.4         13.7         24.2           8         0         0         6.7         11.8         15.6         27.7           9         0         0         7.6         13.3         17.6         31.1           10         0         9.2         16.3         21.5         38.1           11         0         0         10.1         17.8         23.4         41.5           13         0         0         10.9         19.2         25.4         45.0           14         0         0         11.8         20.7         27.3         48.4           15         0         0         12.6         22.2         29.3         51.9           16         0         0         13.4         23.7         31.2         55.4           17         0         0         16.0         28.1         37.1         65.7           20         0         16.0         28.1	2				3.0									
5004.27.49.817.3605.08.911.720.87005.910.413.724.28006.711.815.627.79007.613.317.631.110008.414.819.534.611009.216.321.538.1120010.117.823.441.5130010.919.225.445.0140013.423.731.255.4150012.622.229.351.9160013.423.731.255.4170014.325.233.258.8180015.126.635.162.3190016.829.639.069.2220018.532.642.976.1240020.235.546.883.0250021.838.550.790.0280025.244.458.5103.8320028.650.366.3117.6340028.650.366.3117.7340037.866.687.8155.750029.4<														
6005.08.911.720.87005.910.413.724.28006.711.815.627.79007.613.317.631.11009.216.321.538.1120010.117.823.441.5130010.919.225.445.0140011.820.727.348.4150012.622.229.351.9160013.423.731.255.4170014.325.233.258.8180015.126.635.162.3190016.028.137.165.720018.532.642.976.1240020.235.546.883.0250021.037.048.886.526021.037.048.886.526022.244.458.5103.8320025.244.458.5103.8320025.244.458.5103.8340028.650.366.3117.6350024.974.1131.574.0340033.659.2 </td <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>														
7         0         0         5.9         10.4         13.7         24.2           8         0         0         6.7         11.8         15.6         27.7           9         0         0         7.6         13.3         17.6         31.1           10         0         0         8.4         14.8         19.5         34.6           11         0         0         10.1         17.8         23.4         41.5           13         0         0         10.9         19.2         25.4         45.0           14         0         0         11.8         20.7         27.3         48.4           15         0         0         12.6         22.2         29.3         51.9           16         0         0         13.4         23.7         31.2         55.4           17         0         0         14.3         25.2         33.2         58.8           18         0         0         15.1         26.6         35.1         62.3           19         0         0         17.0         48.8         86.5         26         30.0         69.2           22														
8006.711.815.627.79007.613.317.631.110008.414.819.534.611009.216.321.538.1120010.117.823.441.5130010.919.225.445.0140012.622.229.351.9160013.423.731.255.4170016.126.635.162.3190016.028.137.165.7200016.829.639.069.2220018.532.642.976.1240020.235.546.883.0250021.037.048.886.5260021.838.550.790.0280023.541.454.696.930022.533.370.2124.6350029.451.868.3121.1360031.956.274.1131.5400037.866.687.8155.7500042.074.097.5173.0550046.281.4107.3190.3660 </td <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>														
9         0         0         7.6         13.3         17.6         31.1           10         0         8.4         14.8         19.5         34.6           11         0         0         9.2         16.3         21.5         38.1           12         0         0         10.1         17.8         23.4         41.5           13         0         0         12.6         22.2         29.3         51.9           14         0         0         13.4         23.7         31.2         55.4           17         0         0         14.3         25.2         33.2         58.8           18         0         0         15.1         26.6         35.1         62.3           19         0         0         16.8         29.6         39.0         69.2           22         0         0         18.5         32.6         42.9         76.1           24         0         0         22.2         35.5         46.8         83.0           25         0         0         21.8         38.5         50.7         90.0           28         0         0         25.2 <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>														
10008.414.819.534.611009.216.321.538.1120010.117.823.441.5130010.919.225.445.0140011.820.727.348.4150012.622.229.351.9160013.423.731.255.4170014.325.233.258.8180015.126.635.162.3190016.829.639.069.2220018.532.642.976.1240021.037.048.886.5260021.838.550.790.0280023.541.454.696.9300025.244.458.5103.8320029.451.868.3121.1360033.659.278.0138.4450037.866.687.8155.7500042.074.097.5173.0550058.8103.6136.5242.2750058.8103.6136.5242.2750058.8103.6136.5242.2<														
11009.216.321.538.1120010.117.823.441.5130010.919.225.445.0140011.820.727.348.4150012.622.229.351.9160013.423.731.255.4170014.325.233.258.8180015.126.635.162.3190016.028.137.165.7200018.532.642.976.1240020.235.546.883.0250021.037.048.886.5260021.838.550.790.0280023.541.454.696.9300025.244.458.5103.8320028.650.366.3117.6350029.451.868.3121.1360031.956.274.1131.5400033.659.278.0138.4450037.866.687.8155.7500042.074.097.5173.0550046.281.4107.3190.3<														
120010.117.823.441.5130010.919.225.445.0140011.820.727.348.4150012.622.229.351.9160013.423.731.255.4170014.325.233.258.8180015.126.635.162.3190016.028.137.165.7200018.532.642.976.1240020.235.546.883.0250021.037.048.886.5260021.838.550.790.0280025.244.458.5103.8320026.947.462.4110.7340028.650.366.3117.6350029.451.868.3121.136037.866.687.8155.7500042.074.097.5173.0550054.696.2126.8224.9700058.8103.6136.5242.2750063.0111.0146.3259.5800071.4125.8165.8294.1 <t< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td></t<>														
130010.919.225.445.0140011.820.727.348.4150012.622.229.351.9160013.423.731.255.4170014.325.233.258.8180015.126.635.162.3190016.028.137.165.7200018.532.642.976.1240020.235.546.883.0250021.037.048.886.5260021.838.550.790.0280025.244.458.5103.8320026.947.462.4110.7340028.650.366.3117.6350029.451.868.3121.136031.956.274.1131.5400033.659.278.0138.4450037.866.687.8155.750044.281.4107.3190.360050.488.8117.0207.6650054.696.2126.824.9700058.8103.6136.5242.2750 <td< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td></td<>														
140011.820.727.348.4150012.622.229.351.9160013.423.731.255.4170014.325.233.258.8180015.126.635.162.3190016.028.137.165.7200018.532.642.976.1240020.235.546.883.0250021.037.048.886.5260021.838.550.790.0280025.244.458.5103.8320026.947.462.4110.7340028.650.366.3117.6350029.451.868.3121.136031.956.274.1131.5400033.659.278.0138.4450037.866.687.8155.750044.281.4107.3190.360050.488.8117.0207.6650054.696.2126.824.9700058.8103.6136.5242.2750063.0111.0146.3259.5800														
150012.622.229.351.9160013.423.731.255.4170014.325.233.258.8180015.126.635.162.3190016.028.137.165.7200018.532.642.976.1240020.235.546.883.0250021.037.048.886.5260021.838.550.790.0280025.244.458.5103.8320026.947.462.4110.7340028.650.366.3117.6350029.451.868.3121.136031.956.274.1131.540033.659.278.0138.4450037.866.687.8155.750046.281.4107.3190.360054.696.2126.824.970058.8103.6136.5242.2750063.0111.0146.3259.5800077.6133.2175.5311.4950079.8140.6185.3328.7100084.0 <t< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td></t<>														
160013.423.731.255.4170014.325.233.258.8180015.126.635.162.3190016.028.137.165.7200018.532.642.976.1240020.235.546.883.0250021.037.048.886.5260023.541.454.696.9300025.244.458.5103.8320026.947.462.4110.7340028.650.366.3117.6350029.451.868.3121.136031.956.274.1131.5400033.659.278.0138.4450037.866.687.8155.750044.281.4107.3190.360050.488.8117.0207.6650054.696.2126.824.970058.8103.6136.5242.2750063.0111.0146.3259.5800077.6133.2175.5311.4950079.8140.6185.3328.71000 <td< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td></td<>														
1700 $14.3$ $25.2$ $33.2$ $58.8$ $18$ 00 $15.1$ $26.6$ $35.1$ $62.3$ $19$ 00 $16.0$ $28.1$ $37.1$ $65.7$ $20$ 00 $18.5$ $32.6$ $42.9$ $76.1$ $24$ 00 $20.2$ $35.5$ $46.8$ $83.0$ $25$ 00 $21.0$ $37.0$ $48.8$ $86.5$ $26$ 00 $21.8$ $38.5$ $50.7$ $90.0$ $28$ 00 $23.5$ $41.4$ $54.6$ $96.9$ $30$ 00 $25.2$ $44.4$ $58.5$ $103.8$ $32$ 00 $26.9$ $47.4$ $62.4$ $110.7$ $34$ 00 $28.6$ $50.3$ $66.3$ $117.6$ $35$ 00 $29.4$ $51.8$ $68.3$ $121.1$ $36$ 0 $31.9$ $56.2$ $74.1$ $131.5$ $40$ 00 $33.6$ $59.2$ $78.0$ $138.4$ $45$ 00 $37.8$ $66.6$ $87.8$ $155.7$ $50$ 0 $42.0$ $74.0$ $97.5$ $173.0$ $55$ 00 $58.8$ $103.6$ $136.5$ $242.2$ $75$ 00 $58.8$ $103.6$ $136.5$ $242.2$ $75$ 00 $63.0$ $111.0$ $146.3$ $259.5$ $80$ 00 $77.6$ $133.2$ $175.5$ $311.4$ </td <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>														
180015.126.635.162.3190016.028.137.165.7200018.532.642.976.1240020.235.546.883.0250021.037.048.886.5260023.541.454.696.9300025.244.458.5103.8320026.947.462.4110.7340028.650.366.3117.6350029.451.868.3121.136031.956.274.1131.5400033.659.278.0138.4450037.866.687.8155.750044.281.4107.3190.360050.488.8117.0207.6650054.696.2126.824.970058.8103.6136.5242.2750063.0111.0146.3259.5800077.6133.2175.5311.4950079.8140.6185.3328.7100084.0148.0195.0346.012500125.022.0292.5519.017500														
190016.028.1 $37.1$ $65.7$ 200016.829.6 $39.0$ $69.2$ 220018.5 $32.6$ $42.9$ $76.1$ 240020.2 $35.5$ $46.8$ $83.0$ 250021.0 $37.0$ $48.8$ $86.5$ 260021.8 $38.5$ $50.7$ $90.0$ 280023.5 $41.4$ $54.6$ $96.9$ 300025.2 $44.4$ $58.5$ $103.8$ 320026.9 $47.4$ $62.4$ $110.7$ 340028.6 $50.3$ $66.3$ $117.6$ 350029.4 $51.8$ $68.3$ $121.1$ 3600 $31.9$ $56.2$ $74.1$ $131.5$ 4000 $33.6$ $59.2$ $78.0$ $138.4$ 4500 $37.8$ $66.6$ $87.8$ $155.7$ $50$ 0 $42.0$ $74.0$ $97.5$ $173.0$ $55$ 00 $54.6$ $96.2$ $126.8$ $224.9$ $70$ 0 $58.8$ $103.6$ $136.5$ $242.2$ $75$ 00 $63.0$ $111.0$ $146.3$ $259.5$ $80$ 00 $77.6$ $133.2$ $175.5$ $311.4$ $95$ 00 $79.8$ $140.6$ $185.3$ $328.7$ $100$ 0 $84.0$ $1$				15.1	26.6									
20         0         16.8         29.6         39.0         69.2           22         0         0         18.5         32.6         42.9         76.1           24         0         0         20.2         35.5         46.8         83.0           25         0         0         21.0         37.0         48.8         86.5           26         0         0         23.5         41.4         54.6         96.9           30         0         0         25.2         44.4         58.5         103.8           32         0         0         26.9         47.4         62.4         110.7           34         0         0         28.6         50.3         66.3         117.6           35         0         0         29.4         51.8         68.3         121.1           36         0         31.9         56.2         74.1         131.5           40         0         33.6         59.2         78.0         138.4           45         0         37.8         66.6         87.8         155.7           50         0         46.2         81.4         107.3         190.3<					28.1									
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$														
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$														
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$														
28         0         0         23.5         41.4         54.6         96.9           30         0         0         25.2         44.4         58.5         103.8           32         0         0         26.9         47.4         62.4         110.7           34         0         0         28.6         50.3         66.3         117.6           35         0         0         29.4         51.8         68.3         121.1           36         0         0         30.2         53.3         70.2         124.6           38         0         0         31.9         56.2         74.1         131.5           40         0         0         33.6         59.2         78.0         138.4           45         0         0         37.8         66.6         87.8         155.7           50         0         44.2         81.4         107.3         190.3           60         0         50.4         88.8         117.0         207.6           65         0         0         54.6         96.2         126.8         224.9           70         0         58.8         103.6<														
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	26	0	0	21.8	38.5	50.7	90.0							
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	28	0	0	23.5	41.4	54.6	96.9							
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		0	0		44.4									
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$														
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$														
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$														
40         0         0         33.6         59.2         78.0         138.4           45         0         0         37.8         66.6         87.8         155.7           50         0         0         42.0         74.0         97.5         173.0           55         0         0         46.2         81.4         107.3         190.3           60         0         0         50.4         88.8         117.0         207.6           65         0         0         54.6         96.2         126.8         224.9           70         0         0         58.8         103.6         136.5         242.2           75         0         0         63.0         111.0         146.3         259.5           80         0         0         67.2         118.4         156.0         276.8           85         0         0         71.4         125.8         165.8         294.1           90         0         0         75.6         133.2         175.5         311.4           95         0         0         79.8         140.6         185.3         328.7           100														
45         0         0         37.8         66.6         87.8         155.7           50         0         42.0         74.0         97.5         173.0           55         0         0         46.2         81.4         107.3         190.3           60         0         55.4         88.8         117.0         207.6           65         0         0         54.6         96.2         126.8         224.9           70         0         0         58.8         103.6         136.5         242.2           75         0         0         67.2         118.4         156.0         276.8           80         0         0         71.4         125.8         165.8         294.1           90         0         0         75.6         133.2         175.5         311.4           95         0         0         79.8         140.6         185.3         328.7           100         0         84.0         148.0         195.0         346.0           125         0         105.0         185.0         243.8         432.5           150         0         126.0         222.0         2														
50         0         42.0         74.0         97.5         173.0           55         0         0         46.2         81.4         107.3         190.3           60         0         55.4         88.8         117.0         207.6           65         0         0         54.6         96.2         126.8         224.9           70         0         0         58.8         103.6         136.5         242.2           75         0         0         63.0         111.0         146.3         259.5           80         0         67.2         118.4         156.0         276.8           85         0         0         71.4         125.8         165.8         294.1           90         0         0         75.6         133.2         175.5         311.4           95         0         0         79.8         140.6         185.3         328.7           100         0         0         84.0         148.0         195.0         346.0           125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0 </td <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>														
55         0         0         46.2         81.4         107.3         190.3           60         0         50.4         88.8         117.0         207.6           65         0         0         54.6         96.2         126.8         224.9           70         0         0         58.8         103.6         136.5         242.2           75         0         0         63.0         111.0         146.3         259.5           80         0         0         67.2         118.4         156.0         276.8           85         0         0         71.4         125.8         165.8         294.1           90         0         0         75.6         133.2         175.5         311.4           95         0         0         79.8         140.6         185.3         328.7           100         0         84.0         148.0         195.0         346.0           125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0         222.0         292.5         519.0           175         0         0 </td <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>														
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$														
65         0         0         54.6         96.2         126.8         224.9           70         0         0         58.8         103.6         136.5         242.2           75         0         0         63.0         111.0         146.3         259.5           80         0         0         67.2         118.4         156.0         276.8           85         0         0         75.6         133.2         175.5         311.4           90         0         0         79.8         140.6         185.3         328.7           100         0         0         84.0         148.0         195.0         346.0           125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0         222.0         292.5         519.0           175         0         0         147.0         259.0         341.3         605.5														
70         0         0         58.8         103.6         136.5         242.2           75         0         0         63.0         111.0         146.3         259.5           80         0         0         67.2         118.4         156.0         276.8           85         0         0         75.6         133.2         175.5         311.4           90         0         0         79.8         140.6         185.3         328.7           100         0         0         84.0         148.0         195.0         346.0           125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0         222.0         292.5         519.0           175         0         0         147.0         259.0         341.3         605.5														
75         0         0         63.0         111.0         146.3         259.5           80         0         0         67.2         118.4         156.0         276.8           85         0         0         71.4         125.8         165.8         294.1           90         0         0         75.6         133.2         175.5         311.4           95         0         0         79.8         140.6         185.3         328.7           100         0         0         84.0         148.0         195.0         346.0           125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0         222.0         292.5         519.0           175         0         0         147.0         259.0         341.3         605.5														
80         0         67.2         118.4         156.0         276.8           85         0         0         71.4         125.8         165.8         294.1           90         0         0         75.6         133.2         175.5         311.4           95         0         0         79.8         140.6         185.3         328.7           100         0         0         84.0         148.0         195.0         346.0           125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0         222.0         292.5         519.0           175         0         0         147.0         259.0         341.3         605.5														
85         0         0         71.4         125.8         165.8         294.1           90         0         0         75.6         133.2         175.5         311.4           95         0         0         79.8         140.6         185.3         328.7           100         0         84.0         148.0         195.0         346.0           125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0         222.0         292.5         519.0           175         0         0         147.0         259.0         341.3         605.5														
90         0         75.6         133.2         175.5         311.4           95         0         0         79.8         140.6         185.3         328.7           100         0         0         84.0         148.0         195.0         346.0           125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0         222.0         292.5         519.0           175         0         0         147.0         259.0         341.3         605.5														
95         0         0         79.8         140.6         185.3         328.7           100         0         0         84.0         148.0         195.0         346.0           125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0         222.0         292.5         519.0           175         0         0         147.0         259.0         341.3         605.5														
100         0         84.0         148.0         195.0         346.0           125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0         222.0         292.5         519.0           175         0         0         147.0         259.0         341.3         605.5														
125         0         0         105.0         185.0         243.8         432.5           150         0         0         126.0         222.0         292.5         519.0           175         0         0         147.0         259.0         341.3         605.5														
150         0         0         126.0         222.0         292.5         519.0           175         0         0         147.0         259.0         341.3         605.5														
175 0 0 147.0 259.0 341.3 605.5														
200 0 0 168.0 296.0 390.0 692.0	200	0	0	168.0	296.0	390.0	692.0							

# GROUT MIXTURES PORTLAND CEMENT GROUT

### **BENTONITE GROUT**

CUBIC	CEMENT	BENTONITE	WATER	CUBIC	BENTONITE	WATER
FEET	94 lb. sack	lbs.	gallons	FEET	50 lb sack	gallons
1	0.6339	3.17	5.07	1	0.444	6.22
1.577	1.0	5.0	8.0	2	0.9	12.4
2	1.27	6.3	10.1	2.25	1	14
3	1.90	9.5	15.2	3	1.3	18.7
4	2.54	12.7	20.3	4	1.8	24.9
5	3.17	15.9	25.4	5	2.2	31.1
6	3.80	19.0	30.4	6	2.7	37.3
7	4.44	22.2	35.5	7	3.1	43.5
8	5.07	25.4	40.6	8	3.6	49.8
9	5.71	28.5	45.6	9	4.0	56.0
10	6.34	31.7	50.7	10	4.4	62.2
11	6.97	34.9	55.8	11	4.9	68.4
12	7.61	38.0	60.8	12	5.3	74.6
13	8.24	41.2	65.9	13	5.8	80.9
14	8.87	44.4	71.0	14	6.2	87.1
15	9.51	47.6	76.1	15	6.7	93.3
16	10.14	50.7	81.1	16	7.1	99.5
17	10.78	53.9	86.2	17	7.5	105.7
18	11.41	57.1	91.3	18	8.0	112.0
19	12.04	60.2	96.3	19	8.4	118.2
20	12.68	63.4	101.4	20	8.9	124.4
20	13.95	69.7	111.5	20	9.8	136.8
24	15.21	76.1	121.7	24	10.7	149.3
24	15.85	79.3	126.8	24	11.1	149.5
25	16.48	82.4	131.8	26	11.5	161.7
28	17.75	88.8	142.0	28	12.4	174.2
30	19.02	95.1	142.0	30	13.3	186.6
32	20.28	101.4	162.2	32	14.2	199.0
32	20.28	107.8	172.4	32 34	14.2	211.5
34	22.19	111.0	172.4	34	15.5	211.5
36	22.19	114.1	182.5	36	16.0	217.7
38	24.09	120.5	192.7	38	16.9	236.4
40 45	25.36 28.5	126.8 142.7	202.8 228.2	40 45	17.8	248.8
45 50	31.7	142.7	253.5	45 50	20.0 22.2	279.9
50	34.9			55	22.2	311.0
		174.4	278.9			342.1
60 65	38.0	190.2	304.2	60 65	26.6	373.2
65	41.2	206.1	329.6	65 70	28.9	404.3
70	44.4	221.9	354.9	70 75	31.1	435.4
75	47.5	237.8	380.3	75	33.3	466.5
80	50.7	253.6	405.6	80	35.5	497.6
85	53.9	269.5	431.0	85	37.7	528.7
90	57.1	285.3	456.3	90	40.0	559.8
95	60.2	301.2	481.7	95	42.2	590.9
100	63.4	317.0	507.0	100	44.4	622.0
125	79.2	396.3	633.8	125	55.5	777.5
150	95.1	475.5	760.5	150	66.6	933.0
175	110.9	554.8	887.3	175	77.7	1088.5
200	126.8	634.0	1014.0	200	88.8	1244.0

# E.1600 BORING ABANDONMENT – *TREMIE'* GROUT

### Last Review or Revision: June 2010

### **Objective and Application**

The purpose of this procedure is to describe, in general terms, the principles and methods of securing a borehole from external contaminants after testing is completed.

### Equipment

- Tools for physical measuring procedures and documentation.
- Compatible borehole filling and sealant materials. Terracon preference for non-slurry fill will be chipped Benseal®, high yield Wyoming bentonite or equivalent. Check with Equipment Manager for proper granulation.
- A mechanical pump of sufficient capacity and flow to move grout or bentonite slurry under pressure when required. Consult With the Drill coordinator or Equipment Manager for appropriate sizing.

### Procedures

- The borehole shall be examined to determine if any damaged casing exists that would allow infiltration of contaminants.
- If the casing can not be removed, the borehole shall be redrilled to remove the casing.
- The boreholes shall be grouted to land surface with a side discharge *tremie*' pipe. The grout shall consist of a neat cement of sand, cement and water with a 3 to 5 percent bentonite powder, by weight, additive.
- The grout shall be placed in such a fashion as to prevent any voids or air pockets from forming; side-discharge *tremie*' pipe should be placed in the hole and slowly pulled up as the grout reaches the bottom of the *tremie*' pipe. Shallow holes may be sealed without a *tremie*', by slowing pouring in the grout mixture and "rodding" the mixture to prevent voids or air pockets from forming.
- Borings can be abandoned using the gravity method by filling the borehole from the bottom of the water table with coarse sand (If allowed by law) and from the water table to ground

surface with bentonite pellets. The bentonite pellets shall then be hydrated with lean, potable water.

- Test holes dug with a backhoe to a relatively shallow depth and having a width to depth ratio less than 1:10 may be backfilled with the material that was removed from the test hole and compacted in layers to the approximate density of the undisturbed soil, assuming that the excavated material does not consist of municipal solid waste and is allowed by local or state law. A soil/bentonite mixture or neat cement/bentonite grout may also be used. In areas where an impermeable layer ("cap") has been installed a neat cement/bentonite mixture shall be used to repair the cap.
- Hand augered boreholes shall be filled with dry flaked bentonite from the bottom of the auger hole to land surface and allowed to hydrate.
- A surveyor's monument, noting the borehole location or unique identification number, shall be placed at the land surface to permanently identify the borehole location.
- If the field sampling plan allows the placement of cuttings back in the borehole, then the hole should be filled from the bottom to the top of natural soil with a soil/bentonite mix. Above the natural soil horizon, the hole may be backfilled with cuttings.
- If the samples are obtained with direct push methods and the hole remains open at the completion of sampling, the hole is to be filled with dry flaked bentonite from the bottom of the hole to land surface.

### Attached Supporting Documents

- Volume of Soil Boring, Annulus around 2" and 4" Casings, and Grout Mixtures
- Brainard Kilman Field Facts

### **Other Supporting Documents**

• **ASTM D5299-99** Standard Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities.

## VOLUME OF SOIL BORING

0.05.6				o o =	VOL	UME O
CUBIC			F SOIL		<u> </u>	
	DIANE	TERO	F SUIL	INSIDE I	-	
				HOLLOV		
				3.25	4.25	6.25
LENGTH		l		5.25	4.23	0.25
(ft.)	2	4	6	7.25	8.25	10.25
1	0.028	0.088	0.201	0.29	0.37	0.57
2	0.06	0.18	0.4	0.6	0.7	1.1
3	0.08	0.26	0.6	0.9	1.1	1.7
4	0.11	0.35	0.8	1.2	1.5	2.3
5	0.14	0.44	1.0	1.5	1.9	2.9
6	0.17	0.53	1.2	1.7	2.2	3.4
7	0.20	0.62	1.4	2.0	2.6	4.0
8	0.22	0.70	1.6	2.3	3.0	4.6
9	0.25	0.79	1.8	2.6	3.3	5.1
10	0.28	0.88	2.0	2.9	3.7	5.7
11	0.31	0.97	2.2	3.2 3.5	4.1	6.3
12 13	0.34 0.36	1.06 1.14	2.4 2.6	3.5 3.8	4.4 4.8	6.8 7.4
13	0.30	1.23	2.0	4.1	5.2	8.0
15	0.42	1.32	3.0	4.4	5.6	8.6
16	0.45	1.41	3.2	4.6	5.9	9.1
17	0.48	1.50	3.4	4.9	6.3	9.7
18	0.50	1.58	3.6	5.2	6.7	10.3
19	0.53	1.67	3.8	5.5	7.0	10.8
20	0.56	1.76	4.0	5.8	7.4	11.4
22	0.62	1.94	4.4	6.4	8.1	12.5
24	0.67	2.11	4.8	7.0	8.9	13.7
25	0.70	2.20	5.0	7.3	9.3	14.3
26 28	0.73 0.78	2.29 2.46	5.2 5.6	7.5 8.1	9.6 10.4	14.8 16.0
30	0.84	2.64	6.0	8.7	11.1	17.1
32	0.90	2.82	6.4	9.3	11.8	18.2
34	0.95	2.99	6.8	9.9	12.6	19.4
35	0.98	3.08	7.0	10.2	13.0	20.0
36	1.01	3.17	7.2	10.4	13.3	20.5
38	1.06	3.34	7.6	11.0	14.1	21.7
40	1.12	3.52	8.0	11.6	14.8	22.8
45	1.3	4.0	9.0	13.1	16.7	25.7
50	1.4	4.4	10.1	14.5	18.5	28.5
55	1.5	4.8	11.1	16.0	20.4	31.4
60 65	1.7 1 8	5.3	12.1	17.4 18.0	22.2	34.2 37 1
65 70	1.8 2.0	5.7 6.2	13.1 14.1	18.9 20.3	24.1 25.9	37.1 39.9
75	2.0	6.6	15.1	20.3	27.8	42.8
80	2.2	7.0	16.1	23.2	29.6	45.6
85	2.4	7.5	17.1	24.7	31.5	48.5
90	2.5	7.9	18.1	26.1	33.3	51.3
95	2.7	8.4	19.1	27.6	35.2	54.2
100	2.8	8.8	20.1	29.0	37.0	57.0
125	3.5	11.0	25.1	36.3	46.3	71.3
150	4.2	13.2	30.2	43.5	55.5	85.5
175	4.9	15.4	35.2	50.8	64.8	99.8

UF	SOIL BURING	
	GALLONS PER LINEAR FOOT	

			EAR F				GALLO						
	DIAME	TER O	F SOIL	BORIN				DIAME	TER O	F SOIL	BORIN		
				INSIDE [								DIAMETE	
				HOLLOV								V STEM A	
NOT I			1	3.25	4.25	6.25				1	3.25	4.25	6.25
NGTH							LENGTH						
(ft.)	2	4	6	7.25	8.25	10.25	(ft.)	2	4	6	7.25	8.25	10.25
1	0.028	0.088	0.201	0.29	0.37	0.57	1	0.17	0.66	1.5	2.14	2.78	4.29
2	0.06	0.18	0.4	0.6	0.7	1.1	2	0.3	1.3	3.0	4.3	5.6	8.6
3	0.08	0.26	0.6	0.9	1.1	1.7	3	0.5	2.0	4.5	6.4	8.3	12.9
4	0.11	0.35	0.8	1.2	1.5	2.3	4	0.7	2.6	6.0	8.6	11.1	17.2
5	0.14	0.44	1.0	1.5	1.9	2.9	5 6	0.9	3.3	7.5	10.7	13.9	21.5
6 7	0.17 0.20	0.53 0.62	1.2 1.4	1.7	2.2 2.6	3.4 4.0	6 7	1.0 1.2	4.0 4.6	9.0 10.5	12.8 15.0	16.7 19.5	25.7 30.0
8	0.20	0.62	1.4	2.0 2.3	2.0 3.0	4.0	8	1.4	4.0 5.3	12.0	17.1	22.2	30.0 34.3
9	0.22	0.79	1.8	2.5	3.3	5.1	9	1.4	5.9	13.5	19.3	25.0	38.6
10	0.23	0.88	2.0	2.0	3.7	5.7	10	1.7	6.6	15.0	21.4	27.8	42.9
11	0.31	0.97	2.2	3.2	4.1	6.3	11	1.9	7.3	16.5	23.5	30.6	47.2
12	0.34	1.06	2.4	3.5	4.4	6.8	12	2.0	7.9	18.0	25.7	33.4	51.5
13	0.36	1.14	2.6	3.8	4.8	7.4	13	2.2	8.6	19.5	27.8	36.1	55.8
14	0.39	1.23	2.8	4.1	5.2	8.0	14	2.4	9.2	21.0	30.0	38.9	60.1
15	0.42	1.32	3.0	4.4	5.6	8.6	15	2.6	9.9	22.5	32.1	41.7	64.4
16	0.45	1.41	3.2	4.6	5.9	9.1	16	2.7	10.6	24.0	34.2	44.5	68.6
17	0.48	1.50	3.4	4.9	6.3	9.7	17	2.9	11.2	25.5	36.4	47.3	72.9
18	0.50	1.58	3.6	5.2	6.7	10.3	18	3.1	11.9	27.0	38.5	50.0	77.2
19	0.53	1.67	3.8	5.5	7.0	10.8	19	3.2	12.5	28.5	40.7	52.8	81.5
20	0.56	1.76	4.0	5.8	7.4	11.4	20	3.4	13.2	30.0	42.8	55.6	85.8
22	0.62	1.94	4.4	6.4	8.1	12.5	22	3.7	14.5	33.0	47.1	61.2	94.4
24	0.67	2.11	4.8	7.0	8.9	13.7	24	4.1	15.8	36.0	51.4	66.7	103.0
25	0.70	2.20	5.0	7.3	9.3	14.3	25	4.3	16.5	37.5	53.5	69.5	107.3
26	0.73	2.29	5.2	7.5	9.6	14.8	26	4.4	17.2	39.0	55.6	72.3	111.5
28	0.78	2.46	5.6	8.1	10.4	16.0	28	4.8	18.5	42.0	59.9	77.8	120.1
30	0.84	2.64	6.0	8.7	11.1	17.1	30	5.1	19.8	45.0	64.2	83.4	128.7
32	0.90	2.82	6.4	9.3	11.8	18.2	32	5.4	21.1	48.0	68.5	89.0	137.3
34 35	0.95 0.98	2.99 3.08	6.8 7.0	9.9 10.2	12.6 13.0	19.4 20.0	34 35	5.8	22.4 23.1	51.0 52.5	72.8 74.9	94.5 97.3	145.9 150.2
36	1.01	3.00	7.0	10.2	13.0	20.0	36	6.0 6.1	23.1	52.5 54.0	74.9	97.3	150.2
38	1.06	3.34	7.6	11.0	14.1	20.3	38	6.5	25.1	57.0	81.3	105.6	163.0
40	1.12	3.52	8.0	11.6	14.8	22.8	40	6.8	26.4	60.0	85.6	111.2	171.6
45	1.3	4.0	9.0	13.1	16.7	25.7	45	7.7	29.7	67.5	96.3	125.1	193.1
50	1.4	4.4	10.1	14.5	18.5	28.5	50	8.5	33.0	75.0	107.0	139.0	214.5
55	1.5	4.8	11.1	16.0	20.4	31.4	55	9.4	36.3	82.5	117.7	152.9	236.0
60	1.7	5.3	12.1	17.4	22.2	34.2	60	10.2	39.6	90.0	128.4	166.8	257.4
65	1.8	5.7	13.1	18.9	24.1	37.1	65	11.1	42.9			180.7	278.9
70	2.0	6.2	14.1	20.3	25.9	39.9	70	11.9	46.2	105.0	149.8	194.6	300.3
75	2.1	6.6	15.1	21.8	27.8	42.8	75	12.8	49.5	112.5	160.5	208.5	321.8
80	2.2	7.0	16.1	23.2	29.6	45.6	80	13.6	52.8	120.0	171.2	222.4	343.2
85	2.4	7.5	17.1	24.7	31.5	48.5	85	14.5	56.1	127.5	181.9	236.3	364.7
90	2.5	7.9	18.1	26.1	33.3	51.3	90	15.3	59.4	135.0	192.6	250.2	386.1
95	2.7	8.4	19.1	27.6	35.2	54.2	95	16.2	62.7	142.5	203.3	264.1	407.6
100	2.8	8.8	20.1	29.0	37.0	57.0	100	17.0	66.0	150.0	214.0	278.0	429.0
125	3.5	11.0	25.1	36.3	46.3	71.3	125	21.3	82.5	187.5	267.5	347.5	536.3
150	4.2	13.2	30.2	43.5	55.5	85.5	150	25.5	99.0	225.0	321.0	417.0	643.5
175	4.9	15.4	35.2	50.8	64.8	99.8	175	29.8	115.5	262.5	374.5	486.5	750.8
200	5.6	17.6	40.2	58.0	74.0	114.0	200	34.0	132.0	300.0	428.0	556.0	858.0

(	VOLUME OF ANNULUS AROUND 2" CASING CUBIC FEET PER LINEAR FOOT GALLONS PER LINEAR FOOT													
			ER OF			3	Γ		DIAMETER OF SOIL BORING					
					DIAMET						_		DIAMET	
				HOLLO	N STEM	AUGER						HOLLO	W STEM	AUGER
				3.25	4.25	6.25						3.25	4.25	6.25
LENGTH							I	LENGTH						
(ft.)	2	4	6	7.25	8.25	10.25		(ft.)	2	4	6	7.25	8.25	10.25
1	0	0.065	0.178	0.26	0.34	0.54		1	0	0.491	1.33	1.91	2.55	4.06
2	0	0.13	0.4	0.5	0.7	1.1		2	0	1.0	2.7	3.8	5.1	8.1
3	0	0.20	0.5	0.8	1.0	1.6		3	0	1.5	4.0	5.7	7.7	12.2
4	0	0.26	0.7	1.0	1.4	2.2		4	0	2.0	5.3	7.6	10.2	16.2
5 6	0	0.33	0.9 1.1	1.3 1.6	1.7 2.0	2.7 3.2	_	5 6	0	2.5 2.9	6.7 8.0	9.6 11.5	12.8 15.3	20.3 24.4
7	0	0.39	1.1	1.8	2.0	3.2 3.8		7	0	2.9 3.4	8.0 9.3	13.4	17.9	24.4
8	0	0.40	1.4	2.1	2.4	4.3		8	0	3.9	10.6	15.3	20.4	32.5
9	0	0.59	1.6	2.3	3.1	4.9		9	0	4.4	12.0	17.2	23.0	36.5
10	Ő	0.65	1.8	2.6	3.4	5.4		10	Ő	4.9	13.3	19.1	25.5	40.6
11	0	0.72	2.0	2.9	3.7	5.9	F	11	0	5.4	14.6	21.0	28.1	44.7
12	0	0.78	2.1	3.1	4.1	6.5		12	0	5.9	16.0	22.9	30.6	48.7
13	0	0.85	2.3	3.4	4.4	7.0		13	0	6.4	17.3	24.8	33.2	52.8
14	0	0.91	2.5	3.6	4.8	7.6		14	0	6.9	18.6	26.7	35.7	56.8
15	0	0.98	2.7	3.9	5.1	8.1		15	0	7.4	20.0	28.7	38.3	60.9
16	0	1.04	2.8	4.2	5.4	8.6		16	0	7.9	21.3	30.6	40.8	65.0
17	0	1.11	3.0	4.4	5.8	9.2		17	0	8.3	22.6	32.5	43.4	69.0
18	0	1.17	3.2	4.7	6.1	9.7		18	0	8.8	23.9	34.4	45.9	73.1
19	0	1.24	3.4	4.9	6.5	10.3		19	0	9.3	25.3	36.3	48.5	77.1
20 22	0	1.30	3.6	5.2 5.7	6.8 7.5	10.8	-	20 22	0	9.8	26.6 29.3	38.2	51.0	81.2 89.3
22	0 0	1.43 1.56	3.9 4.3	5.7 6.2	7.5 8.2	11.9 13.0		22 24	0	10.8 11.8	29.3 31.9	42.0 45.8	56.1 61.2	89.3 97.4
24 25	0	1.63	4.5	6.5	8.5	13.5		24 25	0	12.3	33.3	45.8	63.8	101.5
26	0	1.69	4.6	6.8	8.8	14.0		26	0	12.8	34.6	49.7	66.3	105.6
28	Õ	1.82	5.0	7.3	9.5	15.1		28	Ő	13.7	37.2	53.5	71.4	113.7
30	0	1.95	5.3	7.8	10.2	16.2		30	0	14.7	39.9	57.3	76.5	121.8
32	0	2.08	5.7	8.3	10.9	17.3		32	0	15.7	42.6	61.1	81.6	129.9
34	0	2.21	6.1	8.8	11.6	18.4		34	0	16.7	45.2	64.9	86.7	138.0
35	0	2.28	6.2	9.1	11.9	18.9		35	0	17.2	46.6	66.9	89.3	142.1
36	0	2.34	6.4	9.4	12.2	19.4		36	0	17.7	47.9	68.8	91.8	146.2
38	0	2.47	6.8	9.9	12.9	20.5		38	0	18.7	50.5	72.6	96.9	154.3
40	0	2.60	7.1	10.4	13.6	21.6		40	0	19.6	53.2	76.4	102.0	162.4
45	0	2.9	8.0	11.7	15.3	24.3		45	0	22.1	59.9	86.0	114.8	182.7
50 55	0 0	3.3 3.6	8.9 9.8	13.0 14.3	17.0 18.7	27.0 29.7		50 55	0 0	24.6 27.0	66.5 73.2	95.5 105.1	127.5 140.3	203.0 223.3
60	0	3.0	9.0	14.3	20.4	32.4	⊢	60	0	27.0	79.8	114.6	153.0	243.6
65	0	3.9 4.2	11.6	16.9	20.4	32.4 35.1		65	0	29.5 31.9	79.0 86.5	124.2	165.8	263.9
70	0	4.2	12.5	18.2	23.8	37.8		70	0	34.4	93.1	124.2	178.5	203.9
75	0	4.9	13.4	19.5	25.5	40.5		75	0	36.8	99.8	143.3	191.3	304.5
80	Ő	5.2	14.2	20.8	27.2	43.2		80	Ő	39.3	106.4	152.8	204.0	324.8
85	0	5.5	15.1	22.1	28.9	45.9	F	85	0	41.7	113.1	162.4	216.8	345.1
90	0	5.9	16.0	23.4	30.6	48.6		90	0	44.2	119.7	171.9	229.5	365.4
95	0	6.2	16.9	24.7	32.3	51.3		95	0	46.6	126.4	181.5	242.3	385.7
100	0	6.5	17.8	26.0	34.0	54.0		100	0	49.1	133.0	191.0	255.0	406.0
125	0	8.1	22.3	32.5	42.5	67.5	L	125	0	61.4	166.3	238.8	318.8	507.5
150	0	9.8	26.7	39.0	51.0	81.0		150	0	73.7	199.5	286.5	382.5	609.0
175	0	11.4	31.2	45.5	59.5	94.5		175	0	85.9	232.8	334.3	446.3	710.5
200	0	13.0	35.6	52.0	68.0	108.0		200	0	98.2	266.0	382.0	510.0	812.0

### VOLUME OF ANNULUS AROUND 2" CASING

4

	CORIC							GALL	ONSI		NEAR F	001	
	D	IAMET	ER OF	SOIL E				Ľ	DIAME	TER O	F SOIL I		
					DIAMET							DIAMET	
					N STEM	-						W STEM	
			1	3.25	4.25	6.25				1	3.25	4.25	6.25
LENGTH (ft.)	2	4	~	7.05	0.05	10.05	LENGTH (ft.)	2	4	<u> </u>	7.05	0.05	10.05
1	2	4	6 0.11	7.25	8.25 0.26	10.25	1	2	4	6 0.84	7.25 1.48	8.25 1.95	10.25 3.46
2		0	0.11	0.2	0.20	0.40	2	0	0	1.7	3.0	3.9	6.9
3		0	0.2	0.6	0.8	1.4	3	0	0	2.5	4.4	5.9	10.4
4		Ő	0.4	0.8	1.0	1.8	4	0 0	Ő	3.4	5.9	7.8	13.8
5		0	0.6	1.0	1.3	2.3	5	0	0	4.2	7.4	9.8	17.3
6	0	0	0.7	1.2	1.6	2.8	6	0	0	5.0	8.9	11.7	20.8
7		0	0.8	1.4	1.8	3.2	7	0	0	5.9	10.4	13.7	24.2
8		0	0.9	1.6	2.1	3.7	8	0	0	6.7	11.8	15.6	27.7
9		0	1.0	1.8	2.3	4.1	9	0	0	7.6	13.3	17.6	31.1
10		0	1.1	2.0	2.6	4.6	10	0	0	8.4	14.8	19.5	34.6
11		0	1.2 1.3	2.2	2.9 3.1	5.1 5.5	11	0	0	9.2	16.3	21.5	38.1
12 13		0 0	1.3	2.4 2.6	3.1	5.5 6.0	12 13	0 0	0 0	10.1 10.9	17.8 19.2	23.4 25.4	41.5 45.0
14		0	1.5	2.0	3.6	6.4	13	0	0	11.8	20.7	27.3	48.4
15		0	1.7	3.0	3.9	6.9	15	0	0	12.6	22.2	29.3	51.9
16		0	1.8	3.2	4.2	7.4	16	0	0	13.4	23.7	31.2	55.4
17		0	1.9	3.4	4.4	7.8	17	0	0	14.3	25.2	33.2	58.8
18		0	2.0	3.6	4.7	8.3	18	0	0	15.1	26.6	35.1	62.3
19		0	2.1	3.8	4.9	8.7	19	0	0	16.0	28.1	37.1	65.7
20		0	2.2	4.0	5.2	9.2	20	0	0	16.8	29.6	39.0	69.2
22		0	2.4	4.4 4.8	5.7	10.1 11.0	22	0	0	18.5	32.6 35.5	42.9 46.8	76.1
24 25		0 0	2.6 2.8	4.8 5.0	6.2 6.5	11.0	24 25	0 0	0 0	20.2 21.0	35.5 37.0	46.8 48.8	83.0 86.5
26		0	2.0	5.2	6.8	12.0	25	0	0	21.0	38.5	50.7	90.0
28		0	3.1	5.6	7.3	12.9	28	0	0	23.5	41.4	54.6	96.9
30		0	3.3	6.0	7.8	13.8	30	0	0	25.2	44.4	58.5	103.8
32	0	0	3.5	6.4	8.3	14.7	32	0	0	26.9	47.4	62.4	110.7
34		0	3.7	6.8	8.8	15.6	34	0	0	28.6	50.3	66.3	117.6
35		0	3.9	7.0	9.1	16.1	35	0	0	29.4	51.8	68.3	121.1
36		0	4.0	7.2	9.4	16.6	36	0	0	30.2	53.3	70.2	124.6
38		0	4.2	7.6	9.9	17.5	38	0	0	31.9	56.2	74.1	131.5
40 45		0 0	4.4 5.0	8.0 9.0	10.4 11.7	18.4 20.7	40 45	0 0	0 0	33.6 37.8	59.2 66.6	78.0 87.8	138.4 155.7
43 50		0	5.5	10.0	13.0	20.7	43 50	0	0	42.0	74.0	97.5	173.0
55		0	6.1	11.0	14.3	25.3	55	0	0	46.2	81.4	107.3	190.3
60		0	6.6	12.0	15.6		60	0	0	50.4	88.8	117.0	207.6
65		0	7.2	13.0	16.9	29.9	65	0	0	54.6		126.8	224.9
70		0	7.7	14.0	18.2	32.2	70	0	0	58.8	103.6	136.5	242.2
75		0	8.3	15.0	19.5	34.5	75	0	0	63.0	111.0	146.3	259.5
80		0	8.8	16.0	20.8	36.8	80	0	0	67.2	118.4	156.0	276.8
85		0	9.4	17.0	22.1	39.1	85	0	0	71.4	125.8	165.8	294.1
90		0	9.9 10.5	18.0	23.4	41.4	90	0	0	75.6	133.2	175.5	311.4
95 100		0 0	10.5	19.0 20.0	24.7 26.0	43.7 46.0	95 100	0 0	0 0	79.8 84.0	140.6 148.0	185.3 195.0	328.7 346.0
125		0	13.8	20.0	32.5	40.0 57.5	125	0	0	105.0	146.0	243.8	432.5
150		0	16.5	30.0	39.0	69.0	120	0	0	126.0	222.0	292.5	519.0
175		0	19.3	35.0	45.5	80.5	175	0	0	147.0	259.0	341.3	605.5
200		Ő	22.0	40.0	52.0	92.0	200	Ő	Ő	168.0	296.0	390.0	692.0
	•			•		•				•	•	•	•

#### **VOLUME OF ANNULUS AROUND 4" CASING** CUBIC FEET PER LINEAR FOOT **GALLONS PER LINEAR FOOT**

5

### Terracon

# GROUT MIXTURES PORTLAND CEMENT GROUT

### **BENTONITE GROUT**

CUBIC FEET	CEMENT 94 lb. sack	BENTONITE lbs.	WATER gallons	CUBIC FEET	BENTONITE 50 lb sack	WATER gallons
1	0.6339	3.17	5.07	1	0.444	6.22
1.577	1.0	5.0	8.0	2	0.9	12.4
2	1.27	6.3	10.1	2.25	1	14
3	1.90	9.5	15.2	3	1.3	18.7
4	2.54	12.7	20.3	4	1.8	24.9
5	3.17	15.9	25.4	5	2.2	31.1
6	3.80	19.0	30.4	6	2.7	37.3
7	4.44	22.2	35.5	7	3.1	43.5
8	5.07	25.4	40.6	8	3.6	49.8
9	5.71	28.5	45.6	9	4.0	56.0
10	6.34	31.7	50.7	10	4.4	62.2
11	6.97	34.9	55.8	11	4.9	68.4
12	7.61	38.0	60.8	12	5.3	74.6
13	8.24	41.2	65.9	13	5.8	80.9
14	8.87	44.4	71.0	14	6.2	87.1
15	9.51	47.6	76.1	15	6.7	93.3
16	10.14	50.7	81.1	16	7.1	99.5
17	10.78	53.9	86.2	17	7.5	105.7
18	11.41	57.1	91.3	18	8.0	112.0
19	12.04	60.2	96.3	19	8.4	118.2
20	12.68	63.4	101.4	20	8.9	124.4
22	13.95	69.7	111.5	22	9.8	136.8
24	15.21	76.1	121.7	24	10.7	149.3
25	15.85	79.3	126.8	25	11.1	155.5
26	16.48	82.4	131.8	26	11.5	161.7
28	17.75	88.8	142.0	28	12.4	174.2
30	19.02	95.1	152.1	30	13.3	186.6
32	20.28	101.4	162.2	32	14.2	199.0
34	21.55	107.8	172.4	34	15.1	211.5
35	22.19	111.0	177.5	35	15.5	217.7
36	22.82	114.1	182.5	36	16.0	223.9
38	24.09	120.5	192.7	38	16.9	236.4
40	25.36	126.8	202.8	40	17.8	248.8
45	28.5	142.7	228.2	45	20.0	279.9
50	31.7	158.5	253.5	50	22.2	311.0
55	34.9	174.4	278.9	55	24.4	342.1
60	38.0	190.2	304.2	60	26.6	373.2
65	41.2	206.1	329.6	65	28.9	404.3
70	44.4	221.9	354.9	70	31.1	435.4
75	47.5	237.8	380.3	75	33.3	466.5
80	50.7	253.6	405.6	80	35.5	497.6
85	53.9	269.5	431.0	85	37.7	528.7
90	57.1	285.3	456.3	90	40.0	559.8
95	60.2	301.2	481.7	95	42.2	590.9
100	63.4	317.0	507.0	100	44.4	622.0
125	79.2	396.3	633.8	125	55.5	777.5
150	95.1	475.5	760.5	150	66.6	933.0
175	110.9	554.8	887.3	175	77.7	1088.5
200	126.8	634.0	1014.0	200	88.8	1244.0

# E.1700 MONITOR WELL ABANDONMENT

### Last Review or Revision: June 2010

### **Objective and Application**

To permanently close wells drilled as monitoring or recovery wells consistent with lowa codes and close the well structure to prevent its serving as a vertical conduit for movement of environmental impacts through soils. Monitoring wells of 2-inch diameter or greater are considered wells within the definition of this TSOP.

Wellheads and casings, well houses or well pits in themselves are not receptors which are damaged by environmental chemical impact. Water wells, used or unused, are designed to penetrate the groundwater underlying a property. Access to groundwater is the same regardless of use for drinking water, irrigation, non-drinking wash water or other purposes. Often wells no longer in use remain merely because the effort or cost to remove them is considered by the owner to be more than the value.

Relative to ASTM, the physical penetration of soils or fills by a well is considered a potential pathway to carry chemicals to groundwater. This pathway can be inside or outside of the actual well casing. The groundwater, a public natural resource, is considered the end receptor sensitive to environmental impairment.

### Equipment

Terracon drill or other platform capable of conducting the described physical activities. Hand tools appropriate to the job.

Tools for physical measuring procedures and documentation.

Compatible well filling and sealant materials for use with applicable state law. Terracon preference for non-slurry fill will be chipped Benseal®, high yield Wyoming bentonite or equivalent. Check with Equipment Manager for proper granulation and type appropriate to well classification.

A mechanical pump of sufficient capacity and flow to move grout or bentonite slurry under pressure when required. Consult With the Drill coordinator or Equipment Manager for appropriate sizing.

### Training

If the drilling option is required, the drill operator and drill helper will be trained as in TSOP E.300. The Drilling Coordinator will hold an appropriate state driller certification.

Environmental field staff will have;

- Practical working knowledge of hand tools and their applications in the field.
- Enrolled in Terracon medical monitoring program for environmental operations
- Completed 40-Hour OSHA 1910.120 HAZWOPER training, with respiratory protection.
- Current certificate of annual 8-Hour OSHA 1910.120 HAZWOPER Refresher Training

### Procedures

Abandonment of wells are addressed under various requirements as designated by each state. Wells can include, but are not limited to, public and non-public water wells, test wells, observation wells, monitoring wells, agricultural drainage wells, heat pump recirculation wells and private or commercial cooling water wells. Wells not covered by applicable state guidelines in the site is located, but still considered potential pathways by ASTM, are small diameter wells of 2-inch diameter or less, temporary monitoring or observation wells from can be sealed by casing removal and formation collapse and cisterns not accessing groundwater. In general, abandonment of wells follows the methods of "Guidelines for Plugging Abandoned Water Wells", Technical Information Series 15, Geological Survey Bureau, Iowa Department of Natural Resources, 1987.

- Attempt to remove the casing and well screen.
- If the casing can not be removed, the borehole will be overdrilled to remove the casing materials.
- Following removal of the casing, the borehole will be plugged in accordance with E.1500 or E.1600

### Attached Supporting Documentation

- **E.1700.A** *Guidelines for Plugging Abandoned Water Wells*, Technical Information Series 15, Geological Survey Bureau, Iowa Department of Natural Resources, October 1988.
- E.1700.B VOLCLAY GROUT, Internal Terracon Technical Memorandum, August 1986.
- E.1700.C IOWA DEPARTMENT OF NATURAL RESOURCES Abandoned Water Well Plugging Record, Iowa DNR Form 542-1226, Revised February 1990.

### **Other References**

### **TSOP E.1700**

#### Terracon

 Issues & Considerations For Brownfields Restoration, "ON-PROPERTY WELLS AS SENSITIVE RECEPTORS", Terracon, Inc., 1<sup>st</sup> Avenue Revitalization EPA Brownfields Assessment Demonstration Pilot, Coralville, Iowa, #42997048-C, 1999.

# E.1800 PHYSICAL FIELD MEASUREMENTS – SURFACE LAYOUT

### Last Review or Revision: June 2010

### Objective

To provide standard procedures for the gathering of surface layout data to be used in the development of site diagrams.

Layouts obtained using this procedure will be approximate. Distances from available reference features are generally measured using a tape, and angles are estimated. The location of site features will be accurate only to the degree implied by these methods. Typically, Terracon field technicians will strive for a lateral layout precision of  $\pm 5$  feet. If more precise locations are desired, use TSOP E.1808.

### Equipment

- Clipboard,
- Graph Paper,
- Measuring Tape,
- Measuring Wheel,
- Optical Rangefinder (optional), and
- Scale with a straight edge.

### Procedures

Measure distances from available reference features using a tape or measuring wheel and estimate angles.

### **Alternate Methods**

1. Baseline and offsets

Designate a baseline for the X- and Y-dimensions. Measure perpendicular dimensions to site features from the X- and Y-dimension baselines.

2. Triangulation

Locate two (2) stationary points on the site that are significantly separated (e.g., two (2) corners of an on-site building, etc.). Measure distances to site features from each stationary point. Angles need not be measured or estimated if distances from each stationary point are recorded.

3. Distance and direction

### **TSOP E.1800**

### Terracon

Designate a stationary point on the site. Measure distance and angle to site features from the stationary point.

### 4. Rangefinder

An optical rangefinder is an optical device for measuring distances. When looking through the rangefinder at the desired target surface, two images are presented. Using an adjustment knob on the rangefinder, the user can bring the two images together so that they overlap. A scale can then be read which indicates the distance to the target surface. In this manner, distances to relevant features can be estimated without traversing a site. It should be noted that optical rangefinders must be calibrated periodically.

### E.1805 FIELD MEASUREMENT – SURFACE ELEVATIONS

Last Review or Revision: June 2010

### Objective

The purpose of this procedure is to establish proper and accurate methods to be used when surveying elevations on environmental sites.

### Background

On many environmental sites, the horizontal spacing of monitoring wells may be less than 100 feet. The hydraulic gradient between these wells may be less than 0.1 feet, which necessitates an accurate survey of the top of casing elevations to 0.01 feet. Inaccurate elevations in some cases could result in an inaccurate determination of groundwater flow direction.

### Procedures

- a) In performing an elevation survey of a site, the first task is to choose a benchmark (BM). The BM should be of a permanent nature, that will exist over time and will not change in elevation. When using a fire hydrant as a BM, do not use the operating plug or adjustment nut. The elevation on the operating plug can change after the fire hydrant is used. Poured-in-place concrete, sign foundations and hydrants will make a good BM. Attempt to identify a BM with a known elevation to USGS or another datum.
- b) When setting up the level, the instrument person should first ensure that the tripod legs are stabilized and then the instrument can be leveled using the leveling screws. Instruments which are leveled using a bar level need to be turned at right angles and re-leveled until the instrument indicates level in all directions without adjustment. Instruments with a bull's eye level ordinarily need to be leveled once, but the instrument should be turned 360° to ensure that it is accurately leveled.
- c) When using the level, the instrument person should be able to accurately read the rod to 0.01 feet. This entails limiting the distance of the shot so that this accuracy can be maintained. Windy conditions will affect accuracy, especially on shots made where the rod is extended beyond 15 feet in the air. If the instrument person has difficulty reading the rod due to the wind blowing and bending the rod, or because the rod person has difficulty holding the rod in a stable position, the instrument person has the responsibility of resetting to ensure an accurate shot.
- d) Finally, the instrument person should perform a closed loop, utilizing balanced backsight and foresight distances. This requires shooting back to the point of beginning, usually the BM. Upon completion of the loop, the instrument person is responsible for immediately calculating the closure error. On a small site with no turning points, this consists of resetting the instrument, thus creating a turning point and a loop. In these situations, the BM elevation should close with less than 0.01 foot difference. In situations where there are several turning points, the closure error should not be greater than 0.01 foot per 100 linear feet of surveyed distance.

# Attached Supporting Documents Terracon Survey Notes Form

# E.1808 PHYSICAL FIELD MEASUREMENTS – LICENSED SURVEY

Last Revision or Review: June 2010

# Objective

To provide standard procedures for the gathering of surface layout data to be used in the development of site diagrams. This procedure will be used where, because of one or more properties of the site (e.g., large number of points to survey, structural obstructions, etc.) or the need for a certified survey, a licensed surveyor will provide the locations and elevations of site features.

# Equipment

Field equipment will be supplied by the licensed surveyor subcontracted by Terracon. This procedure identifies the procedures for supplying the necessary information to the surveyor for the required measurements. Prior to initiating field services, Terracon and the licensed surveyor will have executed a Terracon Subcontractor Services Agreement.

# Procedures

- Provide the licensed surveyor with a general map of the site and the significant features. Significant features may include, but are not limited to, means of site access, structural obstructions, approximate property boundaries, and the general location of points to be surveyed.
- Negotiate site access for the licensed surveyor with the property owner. Provide the licensed surveyor with a copy of any executed access agreement(s), keys to any gates that may be present, contact information for site representative(s), or any other relevant access information.
- It is important that the licensed surveyor know ahead of time what local or USGS benchmark is to be used for the survey. If a USGS or municipal benchmark is required, the local municipal or county engineering or public roads department should be able to provide a benchmark location and coordinates.
- Provide the licensed surveyor with the required accuracy of lateral and vertical measurements prior to field services. For most Terracon projects, vertical tolerances of ±0.01 feet and lateral tolerances of ±0.5 feet are sufficient.
- Notify all parties involved (e.g., property owners, site managers, etc.) of the date and time that the survey will occur.

# E.1810 FIELD MEASUREMENT – SUBSURFACE SOILS

Last Review or Revision: June 2010

# Objective

To obtain accurate and precise depth measurements from boreholes.

# Equipment

- Monitoring equipment specified by the project manager.
- Decontamination equipment (Alconox detergent, buckets, brushes and water).
- Proper forms as specified by project manager.
- Measuring tape.
- Tool Box.
- Site Map.

# Procedures

- Meet with project manager to discuss project;
- Measure the depth to bottom of borehole by lowering the measuring tape down into the borehole until the tape becomes slack. Raise and lower tape several times to ensure that the bottom of the hole has been accurately defined.
- Observe the measurement on the tape at the reference point to the nearest 0.1 foot.
- Record the borehole depth measurements on the appropriate field form. Write legibly and check the form for accuracy and completeness prior to departing the borehole location.
- If the tape becomes dirty from contacting the side of the borehole, perform proper decontamination procedures before the next measurement.

# E.1820 PHYSICAL FIELD MEASUREMENT - GROUNDWATER

#### Last Review or Revision: June 2010

### 1. OBJECTIVE

To obtain accurate and precise groundwater measurements from monitoring points including monitoring wells, piezometers, observation wells and pumping wells. The groundwater levels may represent static water levels, non-static levels undergoing recovery, or pumping water levels based on site-specific conditions.

### 2. EQUIPMENT

- Monitoring equipment specified by the project manager;
- Decontamination equipment (Alconox detergent, buckets, brushes and water);
- Proper forms as specified by project manager Take copy of most recent data form to use as a field check for representative values. Make sure you have referenced benchmark elevation for each well.
- Measuring devices: electronic water level indicator or interface probe;
- Steel Tape (100' minimum) marked off in units as described above;
- Fuel and water indicating paste;
- Tool Box; and
- Site Map.

# 3. PROCEDURES

- Meet with project manager to discuss project;
- Select the measuring device based on site specific conditions and regulatory requirements. Note: some state regulatory agencies may not allow the use of plumbers chalk or water detection paste. Familiarize yourself with the measurement equipment. Check equipment conditions prior to mobilization, and consider possibility of field break down. Be prepared to use back up equipment.
- Maintain a similar reference point which should be the top of the riser pipe for the monitoring well itself <u>DO NOT USE THE TOP OF THE PROTECTIVE CASING DUE</u> <u>TO POSSIBLE SEASONAL FROST HEAVE OF THE OUTER CASING(S).</u>

- Use the marked point on the riser pipe or the highest point on the riser pipe as the specific reference point unless the riser pipe terminus is level. In this case, use the side of the riser pipe terminus which is closest to the lock clasp on the protective casing.
- Measure the depth to water level by lowering the measuring device down into the well until the water level is encountered. With the electronic devices, an audible or visual signal will be broadcast. This method may be of limited use in steel cased wells.
- Once contact with the groundwater has been established, observe the measurement on the tape at the reference point to the nearest 0.01 foot. Record this measurement to the nearest 0.01 foot.



- If collecting a pumping water level from a well which contains a pump, take care to ensure that the tape/probe is not in contact with the pump intake.
- Collection of water levels from a potable well will require stringent decontamination procedures for organic, inorganic and microbiological concerns. Consult with the project manager for specific procedures.
- Record the water level and well depth measurements on the water level data sheet. Write legibly and check the form for accuracy and completeness prior to departing the well location.
- Perform proper decontamination procedures between each well.
- 4. ATTACHED SUPPORTING REFERENCES

Form 164 10.89 - Groundwater Elevation Data

- 5. OTHER REFERENCES
- Equipment manuals specific to unit prescribed by the Equipment Manager or project Manager.
- "Interface Probe User Guide & Manual", ORS Environmental Equipment, 1991.
- ASTM D4750-87 (Reapproved 1993) "Standard Test method for Determining Subsurface Liquid levels in a Borehole or Monitoring Well (Observation Well)".

# E.1830 PHYSICAL FIELD MEASUREMENT – FREE-PHASE PRODUCT

#### Last Revision or Review: June 2010

# 1. OBJECTIVE

To accurately and precisely determine the thickness of product floating on the groundwater (assuming it is a density of less than 1.0 - light non-aqueous phase liquid - LNAPL) or at the bottom of the well (assuming density of greater than 1.0 - dense non-aqueous phase liquid - DNAPL).

### 2. EQUIPMENT

- Monitoring equipment specified by the project manager.
- Decontamination equipment (scrub buckets with distilled water, Alconox, brushes, clean paper towels and ispopropyl alcohol or other solvent, as required.)
- Proper forms and indelible pen.
- Measuring device;
  - a) Interface Probe.
  - b) Water Level Indicator.
- Fuel indicating paste;
- Toolbox; and
- Site Map.
- 3. PROCEDURE FOR USING INTERFACE PROBE LNAPL
  - Electronic devices such as the interface probe will signal contact with product and groundwater. These instruments should be used based on manufacturer's recommendations.
  - Perform battery test and check decon condition of device prior to leaving the office.
  - Turn on device.
  - Carefully lower the sensor probe out of protective housing and into the well. (Avoid sliding the tape over protective pipe casing edge or monitoring well casing as it may scrape or kink conductor edge measuring tape.)

- As probe encounters product (in excess of 1 mm thick) a tone will sound. Measure fluid depth relative to top of monitoring well casing as discussed in E.1820. Report reading to nearest 0.01 feet. Remove probe and re-check reading three times.
- Lower probe through product layer very slowly and stop when water interface is encountered. Use previous records to prejudge this distance if possible. Water is encountered when a second tone type is generated. Record after 2 readings, recheck water level within 0.01 foot.
- Retrieve tape slowly, carefully cleaning the tape and probe as it is withdrawn from the well. (Never store a contaminated probe or tape in the protective housing or carrying case.)
- 4. PROCEDURE FOR USING INTERFACE PROBE DNAPL
  - \* Electronic devices such as the interface probe will signal contact with product and groundwater. These instruments should be used based on manufacturer's recommendations.
  - \* Perform battery test and check decon condition of device prior to leaving the office.
  - \* Turn on device.
  - \* Carefully lower the sensor probe out of protective housing and into the well. (Avoid sliding the tape over protective pipe casing edge or monitoring well casing as it may scrape or kink conductor edge measuring tape.)
  - \* As probe encounters water, a tone will sound. Measure fluid depth relative to top of monitoring well casing as discussed in E.1820. Report reading to nearest 0.01 feet. Remove probe and re-check reading three times.
  - \* Lower probe through water layer very slowly and stop when water/product interface is encountered. Use previous records to prejudge this distance if possible. Product is encountered when a second tone type is generated. Record after 2 readings, recheck product level within 0.01 foot.
  - \* Retrieve tape slowly, carefully cleaning the tape and probe as it is withdrawn from the well. (Never store a contaminated probe or tape in the protective housing or carrying case.)

# 5. PROCEDURE FOR USING WATER LEVEL INDICATOR

- \* Obtain a water level using the proper measuring device.
- \* Select the measuring device based on site specific conditions and regulatory requirements. NOTE: some state regulatory agencies may not allow the use of water/fuel detection paste.

- \* Maintain a similar reference point which should be the top of the riser pipe for the monitoring well itself. <u>DO NOT USE THE TOP OF THE PROTECTIVE CASING</u>. Use the highest point on the riser pipe as the specific reference point unless the riser pipe terminus is level. In this case, use the side of the riser pipe terminus which is closest to the lock clasp on the protective casing.
- \* Place fuel indicating paste on the water level indicator.
- \* Measure the product thickness by carefully lowering the measuring device until it encounters groundwater based on the previously measured depth. Retrieve the device and measure the height of petroleum product above the water level based on the color change on the paste.
- \* Repeat measurements until three consistent measurements are obtained.
- \* Based on the method employed, it may be necessary to subtract the height of water from the total petroleum wetted height.
- \* Minimize submergence of the indicator below the water level to prevent mixing of petroleum and water.
- \* Record product thickness measurements and visual observations such as color on the water level data sheet. Complete record entry on data sheet. Re-check entries and proceed to next well after properly decontaminating all equipment used down hole.

# ATTACHED SUPPORTING REFERENCES

Form 163 (8/89) - Floating Product and Groundwater Elevation Data

# OTHER REFERENCES

- Equipment manuals specific to unit prescribed by the Equipment Manager or project Manager.
- "Interface Probe User Guide & Manual", ORS Environmental Equipment, 1991.
- ASTM D4750-87 (Reapproved 1993) "Standard Test method for Determining Subsurface Liquid levels in a Borehole or Monitoring Well (Observation Well)".

# E.1840 FIELD MEASUREMENT – HYDRAULIC CONDUCTIVITY TESTING (SLUG)

# Last Review or Revision: June 2010

### Objective

To obtain a representative average horizontal hydraulic conductivity value for the aquifer material around the screened interval.

#### Equipment

Well lock keys.
Data logger and transducer, field printer with power source.
Water level indictor/interface probe.
Time recording device capable of recording in seconds.
Bailer pump or slug device.
Decontamination equipment.
Water supply or collection containers.
Funnel for slug in testing (not recommended).
Solid slug for slug in/slug out testing.

#### Procedures

a) Slug-out test.

- Meet with the Project Manager (PM) to identify which monitoring wells are to be tested, and to identify geology and anticipated conditions.
- After measuring the total depth of the well, place the transducer in the well at least one foot off the well bottom.
- For projects involving more than one well, establish well testing order by moving from least contaminated to most contaminated.
- Select measuring equipment and time interval for test. If aquifer consists of silt, sand, or gravel, the data logger and transducer should be used to record the rapid changes in water levels. A timer and a water level indictor can be used in low permeability aquifers. If a slug test is conducted in an aquifer with a grain size less than the grain size of the gravel pack in the well, the slug test should be at least 45 minutes in duration. This will allow the effects of gravel pack drainage to be minimized and the actual water level change in the aquifer to be measured.
- Measure static water level and total depth of monitoring well to be tested.
- When using the data logger and transducer, start recording elapsed time before removing the volume of water.

- Remove a volume of water from the well using a bailer or pump. Be sure not to lower the water level in the well below the transducer.
- Record water level changes with time as the water level increases toward the static level.
- Initially, water levels should be obtained frequently (minimum every 15 seconds for first five minutes; shorter intervals may be required for more conductive formations).
- Stop recording water level data with time when the water level is within 10% of the static level.
- Perform calculations using methods determined by the PM, if so desired.
- b) Slug-in test
  - Slug-in tests should only be performed in wells where the static water level is above the screened interval or well conditions will not allow a slug-out test to be performed. When a slug-in test has to be performed, enough water should be added to the well so that the changes in water level during the test occur within the riser pipe and not the screened interval.
  - Meet with the PM to identify which monitoring wells are to be tested, identify geology and anticipated conditions.
  - For projects involving more than one well, establish well testing order by moving from least contaminated to most contaminated.
  - Select measuring equipment. If the aquifer consists of silt, sand, or gravel, the data logger and transducer should be used to record the rapid changes in water levels. A timer and water level indicator can be used in low permeability aquifers.
  - Measure static water level and total depth of monitoring well to be tested. After measuring the total depth of the well, place the transducer in the well at least one foot off the bottom.
  - When using the data logger and transducer, start recording elapsed time before adding water.
  - Add water to the well. Be sure to add enough water so that the water level in the well rises above the screened interval.
  - Record water level changes with time as the water level decreases toward the static level.

- Initially, water levels should be obtained frequently (minimum: every 15 seconds for first five minutes; shorter intervals may be required for more permeable formations).
- Stop recording water level data with time when the water level is within 10% of the static level.
- Complete field hydraulic conductivity test form as test progresses.
- Perform calculations using methods determined by the PM, if required.

# ATTACHED SUPPORTING REFERENCES

- In-Situ, Inc., Operator's Manual, Hermit® 1000C
- BRSLUG: A Bouwer-Rice Slug Test Program for Windows, 1995.
- Terracon Field Form: Slug Test Form

# OTHER REFERENCES

**ASTM D4044-96** "Standard Test Method for (Field Procedure) for Instantaneous Change in Head (Slug) Tests for Determining Hydraulic Properties of Aquifiers".

# E.1870 FIELD MEASUREMENT – ELECTROMAGNETIC SURVEY

# Last Revision or Review: June 2010

# 1. OBJECTIVE

To determine the location of metallic subsurface targets using a frequency domain electromagnetic (EM) survey.

# 2. BACKGROUND

Frequency domain EM techniques impart an electromagnetic field below the instrument with a transmitter coil. The magnitude and phase of the induced field is intercepted by a receiver coil and produces an output voltage which is linearly related to subsurface conductivity. The response indicated by the instrument is a bulk measurement of the electrical conductivity of the subsurface materials from the ground surface to the effective depth of investigation for the instrument.

# 3. EQUIPMENT

A Geonics EM-31 or Geonics EM-34 Ground Conductivity Meter will be utilized to perform the EM survey. The EM-31 has an effective depth of exploration of approximately 3 to 6 meters, depending upon the instruments orientation, and provides both the quadraturephase and the inphase components. The EM-34 has an effective exploration depth of approximately 7.5 to 15 meters dependent on coil separation. However, the EM-34 measures only quadrature phase component of the EM field, therefore reducing its effectiveness in identifying buried metallic targets. An electronic digital data recorder, capable of recording both quadrature and inphase components, will be used to record the instrument responses.

# 4. PROCEDURES

a) Preliminary Site Reconnaissance Survey (PSRS)

Prior to initiation of the EM survey, a PSRS will be performed. The purpose of the PSRS will be to evaluate site conditions relative to performance of the EM survey and the results obtained. Site conditions that will be considered include cultural features (i.e. on-site utilities, man-made structures, power lines, and radio transmitters) and the

#### Terracon

geologic setting of the study area (as is ascertainable from a surficial reconnaissance). The results of the PSRS will be used to determine the most effective technique of investigation, instrument selection, grid node spacing, evaluation of data, data processing procedures, and other relevant survey procedures. The results of the PSRS, together with proposed modification (if applicable) to the EM Survey plan, will be presented to the state regulatory agency for review and approval prior to proceeding with the EM survey, if required.

b) Survey Grid

An equal spaced grid will be established on the ground surface in the expected vicinity of the subsurface target. The survey grid will be large enough to ensure target coverage and establish background values, utilized during data evaluation and processing. Each node of the grid will be marked with a surveyors stake, paint spot, or other field suitable marking.

B. Data Collection

Following establishment of the survey grid, station measurements will be made at each of the nodes. Conductivity measurements may be taken in a north-south and east-west instrument orientation when using the EM-31 to improve subsurface resolution. When applying the EM-34, conductivity measurements may be collected at 10 and 20 meter coil separation, resulting in greater depth of investigation. Conductivity readings will be recorded in a field notebook and the electronic data recorder. Variations in conductivity measurements will be utilized in evaluation of potential anomalies. In addition, conductivity fluctuations will be observed when walking between nodes when using the EM-31. In the event a significant fluctuation is observed between nodes, a mid-node reading may be collected.

C. Data Evaluation

Following completion of the EM survey, the conductivity data will be evaluated. The raw data will be downloaded from the digital data recorder to an IBM compatible PC for processing.

To enhance the quality of the data and reduce subjective interpretation of measurements, the raw data will be processed. Processing will entail a statistical and numerical analysis of the data population.

Statistical analysis will consist of determining the average and standard deviation of the data population. Performance of these analyses assumes that background

conductivities and conductivities associated with cultural effects will fall within one standard deviation. Therefore, any data points outside plus or minus one standard deviation of the mean of the data population will be considered anomalous. Based upon these analyses, indicated anomalies may be associated with target signature or significant changes in geologic conditions across the study area.

It is anticipated that the difference in instrument response between the native subsurface materials and the target will result in variation of conductivities. To assist in evaluation of anomalous features, numerical analysis of the data population will be performed. Numerical analysis will consist of a determination of the absolute value of the difference in conductivity from the north-south and east-west orientation. Following determination of the absolute values, statistical analysis as described above will be applied to the data population and the results contoured. After processing, the data will be contoured using a computer contouring program (e.g. SURFER).

Following completion of the data evaluation described above, indicated anomalous features will be evaluated. The anomalies will be evaluated and potential target areas may be identified

# D. QA/QC Procedures

EM units are calibrated by the manufacturer. Calibration is performed over a massive bedrock outcrop of known conductivity which is considered as a geologic "standard". Following calibration, the EM units typically retain their accuracy for an extended period of time.

To ensure accuracy, evaluate instrument drift (if present), and maintain quality control, a site standard will be established in the field. A survey line for the site standard will be established. Each day, prior to start of field activities and following completion of field activities, station measurement will be performed along the site standard. Significant deviations in conductivity measurements between site standard surveys may be used during data evaluation to compensate for instrument drift.

To evaluate repeatability and reliability of generated data measurements, 10% of the grid lines will be resurveyed. Conductivity values obtained along the local standard and along the repeat survey lines will be recorded as described above and reported. In the event that instrument drift is observed, the amount of drift will be evaluated with respect to relative changes in the data set indicated by the statistical analysis described above. In the event that the observed drift exceeds plus or minus one standard deviation of the mean of the data population, the absolute values of the data obtained will be adjusted.

# ATTACHED SUPPORTING REFERENCES

• Operating Manual, Fisher TW-6, Fisher Research Laboratory

# E.1900 GROUNDWATER SAMPLING - BAILER

# Last Review or Revision: June 2010

### Objective

To collect a representative groundwater sample from the sampling point for chemical analysis. This includes the documentation of sampling methods, sampling supplies, and protocol to reduce potential for alteration and or cross-contamination during the sampling event.

#### Equipment

- Monitoring equipment specified by project manager (e.g. pH meter, turbidity meter, conductivity meter, etc).
- Water level indicator;
- Decontamination equipment;
- Proper forms, labels and indelible ink pen;
- Sample containers and packing material, tape, and labels;
- Filtration device and filters (and fixing agents as appropriate);
- Cooler with ice pack and packing media;
- Bucket (calibrated in gallons or liters)
- Disposable bailers
- Keys for locking cap on well;
- Rope steel, nylon, teflon, or polypropylene;
- Deionized (DI) water;
- Knife; and
- Site map.

#### **TSOP E.1900**

### Procedures

a)

- Preparation
  - Meet with Project Manager;
  - Obtain the bottles, forms, and equipment necessary to complete the sampling event;
  - Calibrate all field equipment;
  - Establish sampling well sequence (generally least impacted to most impacted).
- b) Field Activities
  - Water levels collect and record water levels (and well depth, if requested by P.M.)
  - 1) Contamination minimization
    - Use plastic sheet if necessary
    - Use proper bailing techniques (hand over hand) to prevent rope from touching the ground
    - 2) Purging
      - Use a bailer (dedicated).
      - Disposal of water containerize if necessary or collect in a bucket and dispose of on site away from the well to minimize potential vertical migration along the annular well space.
      - May need to vary the purging rate to minimize drawdown and subsequent cascading of water if dealing with very volatile compounds (i.e., vinyl chloride).
- c) Sample Collection
  - 1) Preservation
    - Use containers with proper preservative if necessary.
  - 2) Collection
    - Minimize disturbances which may aerate the sample (i.e., lower bailer slowly into water, pour slowly into sample container, etc.).
    - Pour water from the top of the bailer or insert the bottom emptying device for sample transfer.

- Transfer the groundwater sample directly to the laboratory prepared sample container or the filter cup.
- Samples collected for VOC's should always be collected from a recently filled bailer full of water as soon as it is brought to the surface.
- Collect samples for VOC's by forming a positive meniscus on the sample vial and capping immediately.
- VOC samples must be free of air bubbles (i.e. turn VOA upside down to check for air bubbles).
- Do not over-fill sample containers which contain a preservative.
- Place samples in cooler with ice or blue-ice to maintain a temperature between 2°C and 6°C.
- 3) Data Documentation
  - Record all pertinent sampling information on the sampling container label, sampling information form, chain-of-custody, and shipping form.
  - Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, sampling methodology, parameters to be analyzed, stabilization data, and general observations.
  - Make all entries in the chain-of-custody form prior to leaving the site. Ensure that the chain-of-custody protocol required for the project is maintained.
  - If samples must be shipped, the chain-of-custody form must be enclosed with the samples and the container sealed with Terracon security labels. Obtain a post office receipt, bill of lading or similar document from the shipper to be included as part of the chain-ofcustody documentation. Return one copy of the chain-of-custody documentation to the project manager.

# **Attached Supporting Documents**

• **ASTM D4448-85a** Standard Guide for Sampling Monitoring Wells.

# E.2000 GROUNDWATER SAMPLING – LOW FLOW GROUNDWATER SAMPLING

# Last Revision or Review: June 2010

# Objective

To collect a representative groundwater sample from the sampling point for chemical analysis. This procedure should be used when attempting to minimize stress to the aquifer due to monitoring well sampling. This procedure includes the documentation of sampling methods, sampling supplies and protocol to reduce potential for alteration and or cross-contamination during the sampling event.

# Equipment

- Groundwater Elevation Data form;
- laboratory Chain-of-Custody form;
- laboratory sample labels;
- field logbook;
- indelible ink pen;
- pH, temperature, and specific conductance meter;
- turbidity meter;
- sample containers and packing material;
- cooler with ice pack and packing media;
- well purging equipment disposable bailers and string;
- sampling device low-flow peristaltic pump;
- mobile, secure shed to protect equipment during sampling (insulated during colder weather);
- keys for locking cap on well and secure shed;
- deionized (DI) water; and
- site map.

# Procedures

Preparation - Meet with Project Manager;

- obtain the sample containers, forms, and equipment necessary to complete the sampling event;
- calibrate all field equipment i.e., temperature, pH, specific conductance meter and turbidity meter;

#### **TSOP E.2000**

#### Terracon

- document equipment calibration in field logbook; and
- establish sampling sequence.

# **Monitoring Well Purging**

- document all field activities in field logbook and field forms
- water levels collect and record water levels
- purge monitoring wells
  - using bailers, remove three (3) well casing volumes of water from each well (or until well goes dry)
  - collect purge water in a bucket and dispose of on site away from the well to minimize potential vertical migration along the annular well space
- return to site after stabilization period of two (2) days
- contamination minimization
  - use plastic sheet as an apron to isolate the wellhead by splitting sufficiently to pass the well protector and slide over wellhead onto ground.

# Sampling

- use a low-flow sampling pump
- determine proper depth of purging device, approximately half the distance of the water column or approximate middle of the well screen
- attach new section of pump tubing to pump
- lower pump suction tubing slowly (to minimize disturbance) into well to midpoint of sampling zone
- start pump at its lowest setting and slowly increase speed until discharge occurs.
- check water level and adjust pump speed to maintain drawdown at less than four (4) inches and pump no faster than 0.1 L/min
- stabilization parameters
  - record turbidity, temperature, pH, and specific conductance at regular intervals
- transfer the groundwater sample directly to the laboratory prepared sample containers
  - fill sample containers by allowing pump discharge to flow gently down the side of the container with minimal disturbance
  - do not over-fill sample containers which contain a preservative
  - place samples in cooler with ice

# **Data Documentation**

- record all pertinent sampling information on the sampling container label, sampling information form, chain-of-custody, and shipping form.
- pertinent data will vary based on the parameter and the form; however, the following data must be recorded time, date, job number, project name, sampling location, samplers name,

sampling methodology, parameters to be analyzed, stabilization data, and general observations.

• make appropriate entries in the chain-of-custody form at time of sample collection - ensure that the chain-of-custody protocol required for the project is maintained

# ATTACHED SUPPORTING REFERENCES

Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells, EPA Region 1, July 30, 1996, Revision 2.

# E.2100 SOIL VAPOR SAMPLING

# Last Revision or Review: June 2010

### Objective

The objective of soil vapor sampling is to provide an estimate of the concentration of vapors that may be released by subsurface soils into adjacent structures (basements, etc.). This procedure is intended to provide general guidelines. State-specific guidelines or requirements supercede the information presented in this TSOP.

#### Procedures

Soil gas must be sampled at the location of maximum soil concentrations, and at a depth above the water table expected to exhibit the highest gas reading based on field screening and analytical results. In order to verify the soil gas measurement is representative of the maximum expected gas level, two gas samples must be taken at least two weeks apart, with one of the samples taken during a seasonal period of lowest groundwater elevation and, if applicable, below the frost line. The following exploratory methods may be used to obtain soil vapor samples:

**Option 1:** A hollow, small-diameter (minimum 0.5-inch outside diameter), threaded steel casing fitted with a loose-fitting end plug is driven to the appropriate sampling depth. The casing is retracted a minimum of 12 inches to expose the soils in the sidewalls. The end plug should fit such that it remains in place at the bottom of the hole when the casing is retracted. The top of the casing is capped. The borehole is sealed around the annulus between the casing and borehole sidewall to prevent cross-contamination or dilution with the ambient air. Allow the soil air to stabilize for at least one hour prior to sampling. When direct-push technologies are used as a means of obtaining soil vapor samples, analysis using portable equipment is not acceptable. Samples must be collected using specialized sampler tubes and sent to a laboratory for analysis.

**Option 2:** A small-diameter (suggested 3-inch) hand auger boring is extended to appropriate sampling depth. A hollow, 1-inch diameter, threaded PVC casing perforated in the lower 12 inches is placed in the borehole. Sand backfill is placed to a depth not to exceed 18 inches above the bottom of the boring, covering the perforated section of the casing. The remainder of the borehole must be filled with hydrated bentonite to seal around the casing. The top of the casing is capped. Allow the soil air to stabilize for at least one hour prior to sampling.

Soil gas samples must be collected in Tedlar bags or Summa canisters and analyzed using NIOSH Method 1501, TO-14, TO-15, or other approved method. Soil gas is collected by means

### **TSOP E.2100**

#### Terracon

of adsorption onto solid activated carbon media. Glass tube samplers that comply with NIOSH Method 1501 and piston-type vacuum samplers are available commercially. The vacuum sampler used must be capable of drawing two hundred milliliters (200 ml) of casing air through the carbon media by either single or incremental operation. The pump must be factory calibrated according to manufacturer's specifications, and fitted with an indicator which visibly shows when the sampling cycle has been completed. Flow rates must be verified and volume checks must be conducted immediately prior to and immediately after sampling. Sampling equipment must be cleaned prior to each sampling event and stored to prevent cross-contamination. Cleaning of equipment must occur away from the sampling location and sufficient time must be allowed for the evaporation of any cleaning solvents which may interfere with chemical analysis.

Consult NIOSH Method 1501 and the instructions provided by the manufacturer of the sampler device for specific sampling procedures. The following general procedures are recommended to obtain a representative soil gas sample:

- 1. Attach a sufficient length of rubber tubing to the sampling pump to form an air tight seal.
- 2. Break the tip of the sampler tube and fasten the tube securely to the free end of rubber tubing with the arrow of the sampler tube pointing toward the pump.
- 3. Insert the sampler tube into the casing and position it so the inlet of the sampler tube is above, but within 6 inches of, the bottom of the casing.
- 4. Draw a 200 ml volume of soil air through the sampler tube and immediately withdraw it from the borehole casing.
- 5. Disconnect the sampler tube from the rubber tubing and seal the tube using the plastic caps provided by the vendor.

Standard handling and transporting procedures are used for the sampler tubes including the processing of chain-of-custody forms. Samples must be analyzed for benzene and toluene in accordance with NIOSH Method 1501. Analysis of sample blanks for quality assurance is recommended.

# E.2210 GENERAL SITE HOUSEKEEPING

### Last Review or Revision: June 2010

### Objective

To create and maintain an orderly working area that reduces the likelihood of injury and potential for sample contamination due to messy site conditions.

# Equipment

- Equipment specified by project manager.
- Trash bags.
- Disposable chemical-resistant gloves.

#### Procedures

After using disposable equipment, dispose of the materials in general trash bags. Segregate materials if needed as specified by the project manager. Use disposable gloves when handling waste material to reduce the chance for cross-contamination and/or exposure to contaminants. Bags, paper, packaging, and other materials should not be allowed to remain unattended on the site. Dispose of full trash bags using an on-site dumpster, at the Terracon dumpster after returning to the office, in a designated sealed drum on-site, or as specified by the project manager. After decontamination of non-disposable equipment, store in an orderly fashion on the remedial vehicle or trailer to facilitate quick location and reduce the likelihood of injury. Dispose of auger cuttings and cleaning fluids as specified by the Project Manager.

# E.2220 DISPOSAL OF SPENT SUPPLIES

### Last Review or Revision: June 2010

#### Objective

To provide for proper disposal of sampling equipment, personal protective equipment (PPE), etc. in accordance with applicable regulations.

#### Equipment

- Equipment specified by project manager.
- Trash bags.
- Disposable chemical-resistant gloves.

#### Procedures

Collect sampling equipment, spent PPE, cleaning fluids, etc. as specified by the Project Manager. This may include segregating the material and sealing in 55-gallon drums, placing securely on the site, or disposal to a nearby dumpster (non-hazardous). If material is to be containerized for transportation or storage on-site, clearly mark all containers as to their materials, taking care to use proper signage. Transportation of materials off-site may be by Terracon or a hazardous waste hauler. If a hazardous waste hauler is used, collect all documentation provided (i.e., scale tickets, waste manifests, etc.).

# E.2230 HANDLING AND STORAGE OF DRILL CUTTINGS (NON-HAZARDOUS)

# Last Review or Revision: June 2010

# Objective

To dispose of drill cuttings generated by drilling and well installation activities. This procedure is applicable to sites where the cuttings generated can be reasonably be assumed to be non-hazardous in nature. For handling and storage of drill cuttings that can be reasonably be assumed to be hazardous in nature, reference TSOP E.2235.

# Equipment

- Plastic Sheeting.
- Shovel(s).
- Wheelbarrow.
- USDOT-approved 55-gallon drums
- Disposable chemical-resistant gloves.
- Material as specified by Project Manager.

# Procedures

Spread the plastic sheeting over the ground in an area designated by the project manager for drill cutting storage. Using the shovel(s) and wheelbarrow, transport the generated cuttings to the stockpile area, if required. Use disposable gloves to reduce the likelihood of sample cross-contamination and/or exposure to site contaminants. Segregate the stockpiles of soil on the plastic sheeting if instructed by the project manager. After stockpiling soil, cover the soil piles with additional plastic sheeting. Use rocks or other available moderately heavy material to prevent the plastic sheeting from blowing off the pile.

As an alternate procedure, the Project Manager may prefer to containerize the auger cuttings in fifty-five (55) gallon drums for transportation off-site or disposal at a later date. Also as an alternate, the Project Manager may specify that auger cuttings be returned to the borehole (if allowed by state law) as much as possible, and then spread over the ground in the area near the boring.

# E.2235 SITE HOUSEKEEPING HANDLING AND STORAGE OF DRILL CUTTINGS (HAZARDOUS)

# Last Revision or Review: June 2010

# 1. OBJECTIVE

To establish standardized procedures for the identification, handling, storing, documentation and disposal of wastes generated during environmental projects. Although not all wastes are considered hazardous, proper procedures are required to characterize the wastes generated.

# 2. BACKGROUND

The primary regulations governing hazardous waste are Title 40 of the Code of Federal Regulations, Parts 260 through 272. Individual state regulations are also reviewed where applicable.

# 3. PROCEDURES

The project manager will develop the necessary protocols and operating procedures for each site as part of the health and safety plan and the work plan. Technical support for the development of these documents is the responsibility of the following people:

<u>Area</u>	<u>Responsibilities</u>
Health and Safety	Corporate H&S - Mr. Gary Bradley (Corporate)
Regulatory Compliance	Compliance Services - Mr. David Wilson (Dallas)
Site Operations	Project Manager

The corporate H&S officer reviews each project as it is registered to determine the need for technical assistance in hazardous waste operations. The Regulatory Compliance Group will be contacted for assistance and to review each project involving hazardous waste handling or disposal. The project manager is responsible for the hazardous waste operations disposal and documentation at each site. If the site conditions change during field operations, the project manager should be contacted for resolution. Terracon has developed an internal check-list for consulting clients through the RCRA regulations. This same checklist is used by the project managers to review the status of their hazardous waste operations. The checklist may be overly detailed for certain projects. However, the project managers must review those areas that apply to their site and contact the regulatory compliance group for further clarification or assistance.

# HAZARDOUS WASTE CHECK-LIST (Extracted from Terracon Consultants, Inc. Check-Lists - RCRA)

- □ WASTE DETERMINATION AND NOTIFICATION
  - □ Has generator determined whether waste is hazardous or non-hazardous (40 CFR 262.11).
  - Has notification form been submitted and ID numbers obtained (40 CFR 262.12).
- □ MANIFESTS AND LDR FORMS
  - □ If required, has generator properly completed and maintained manifests (40 CFR 262.20-23).
  - □ If required, has generator properly completed and maintained LDR forms (40 CFR 268.7).
- □ STORAGE, ACCUMULATION, AND HANDLING REQUIREMENTS
  - Are wastes packaged, labeled and placarded according to DOT requirements (40CFR 262.30-33).
  - □ Is waste shipped off-site in accordance with applicable accumulation time restrictions (90, 180 days) (40 CFR 262.34).
  - □ Is satellite accumulation in compliance with applicable requirements (40 CFR 262.34(c)).
  - □ Are containers maintained in good condition (40 CFR 265.171).
  - □ Are containers compatible with waste stored (40 CFR 265.172).
  - Are hazardous waste containers closed except when adding or removing waste (40CFR 265.173).
  - □ Are containers and storage areas inspected at least weekly (40 CFR 265.174).
  - Are containers holding ignitable or reactive wastes at least 50 feet from the facility's property line (40 CFR 265.176).
  - □ Are requirements for tank storage met (40 CFR 265.201).

# PREPAREDNESS AND PREVENTION REQUIREMENTS

□ Are communications or alarms systems available (40 CFR 265.32(a)).

- □ Are telephones or radios available for summoning emergency assistance (40 CFR 265.32(b)).
- □ Are fire extinguishers or other fire control equipment available (40 CFR 265.32(c)).
- □ Is water or foam available at adequate volume and pressure (40 CFR 265.32(d)).
- Are all facility communications or alarm systems, fire protection equipment, spill control equipment, and decontamination equipment tested and maintained to assure its proper operation (40 CFR 265.33).
- □ Is there access to internal alarms or emergency communication devices (40 CFR265.34).
- □ Is there adequate aisle space to allow the unobstructed movement of emergency personnel and equipment (40 CFR 265.35).
- □ Have arrangements been made with local authorities for emergency (40 CFR 265.37) response purposes.

#### □ EMERGENCY PROCEDURES AND CONTINGENCY PLAN

- □ Has an Emergency Coordinator been designated (40 CFR 262.34(d)(5)).
- $\Box$  Is emergency information posted next to telephones (40 CFR 262.34(d)(5)).
- Have employees been trained in waste handling and emergency procedures (40 CFR 262.34(d)(5)).
- □ Are emergency coordinators prepared to respond to emergency situations (40 CFR 262.34(d)(5)).
- □ Has a contingency plan been prepared for the facility (40 CFR 265.51).
- Does the plan contain the required contents (40 CFR 265.52).
- □ Is the plan available at the facility and has it been submitted to local fire, police and hospitals (40 CFR 265.53).
- □ Has the contingency plan been amended when required (40 CFR 265.54).
- □ Has an emergency coordinator been designated (40 CFR 265.55).
- □ Are all required emergency procedures addressed in the plan (40 CFR 265.56).

# □ RECORDKEEPING AND REPORTING

- □ Has generator maintained copies of manifests, waste determinations or waste analysis for at least 3 years (40 CFR 262.40).
- □ If LQG, has generator prepared and maintained Biennial reports (40 CFR 262.41).
- □ If applicable, has generator prepared and submitted exception reports (40 CFR 262.42).
- □ EMPLOYEE TRAINING
  - □ Have employees been trained in proper waste handling and emergency procedures relevant to their positions (40 CFR 262.34(d)).
  - □ Have employees received classroom and on-the-job training within six months of employment (40 CFR 265.16(a-b)).
  - □ Has refresher training been provided annually (40 CFR 265.16(c)).
  - □ Are required training documents and records maintained (40 CFR 265.16(d)).

# □ WASTE MINIMIZATION

□ If SQG, has generator made good faith effort at waste minimization (RCRA, 3002(b)).

□ If LQG, has generator prepared a written waste minimization plan (RCRA, 3002(b)).

# HAZARDOUS WASTE MANAGEMENT DOCUMENTS

- NOTIFICATION OF HAZARDOUS WASTE ACTIVITY FORM
- WASTE ANALYSIS RESULTS
- WASTE DETERMINATION DOCUMENTS
- ACCUMULATION AREA INSPECTION RECORDS
- MANIFESTS AND LDRS
- STATE, BIENNIAL, AND EXCEPTION REPORTS
- TRAINING RECORDS
- ENFORCEMENT AGENCY INSPECTION REPORTS
- CONTINGENCY PLAN
- WASTE MINIMIZATION PLAN
- CLOSURE PLAN
- WASTE ANALYSIS PLAN
- ENFORCEMENT ACTIONS

#### Terracon

### □ CONDITIONALLY EXEMPT SMALL QUANTITY GENERATORS

- Does the facility qualify as a conditionally exempt small quantity generator each calendar month by: (40 CFR 261)
  - □ Generating less than 100 kgs and accumulating less than 1,000 kgs of H.W. on site? (40 CFR 261.5[a][g])

or

- □ Generating less than 1 kg of acute H.W., or 100 kgs of acute H.W. contaminated soil or spill residues? (40 CFR 261.5[e][1-2])
- □ Did the quantity determination include all listed and characteristic wastes generated except: (40 CFR 261.5[d])
- □ H.W. removed from on-site storage? (40 CFR 261.5[d][2])
- □ H.W. produced by on-site treatment or reclamation of H.W. that was already counted once? (40 CFR 261.5[d][2])
- □ Spent materials that have already been counted once that are reclaimed, reused, and subsequently generated on-site? (40 CFR 261.5[d][3])

or

- □ H.W. exempted from regulation? (40 CFR 261.5[c])
- □ Has the conditionally exempt small quantity generator treated or disposed of the H.W. in an on-site facility, or ensured delivery of an off-site U.S. TSD either of which is? (40 CFR 261.5[f,g][3])
  - □ Permitted under Part 270? (40 CFR 261.5[f,g][3][i])
  - □ In interim status under 265 and 270? (40 CFR 261.5[f,g][3][ii])
  - □ Authorized by an approved state under Part 271? (40 CFR 261.5[f,g][3][iii])
  - Permitted, licensed, or registered by a state to manager municipal or industrial solid waste? (40 CFR 261.5[f,g][3][iv])

or

□ A facility which? (40 CFR 261.5[f,g][v])

Legitimately uses, reuses, recycles, or reclaims the waste? (40 CFR 261.5[f,g][3][v][A])

#### or

- Treats its waste prior to use, reuse, recycling or reclaiming? (40 CFR 261.5[f,g][3][v][B])
- GENERATORS OF BETWEEN 100 AND 1,000 KG/MONTH
  - □ The 100-1,000 kg/mo. generator may accumulate H.W. on site for up to 180 days without interim status provided they: (40 CFR 262.34[d])
    - □ Accumulate no more than 6,000 kgs of H.W. on site at any time? (40 CFR 262.34[d][1])
    - □ Complied with requirements for storage in containers, 265 Subpart I (except for the 50 foot rule [265.176])? (40 CFR 262.34[d][2])
    - □ Complied with 265.201, storage in tanks (attached)?
    - □ Complied with requirements for Subpart C, preparedness and prevention?
    - □ Clearly marker the date accumulation started on each container?
    - □ Labeled each container and tank with the words "Hazardous Waste"? (40 CFR 262.35[d][3])
  - Does the generator have at least one emergency coordinator (E.C.) on site or immediately available at all times? (40 CFR 262.3[d][4][i])
  - □ E.C.'s name and phone number? (40 CFR 262.3[d][4][ii][A])
  - □ Location of fire extinguisher, spill control material, and any fire alarms? (40 CFR 262.34[d][4][ii][B]
  - □ If no direct alarms, the phone number of the fire department? (40 CFR 262.3[d][4][ii][C])
  - □ Are all employees familiar with their jobs' proper waste handling and emergency procedures? (40 CFR 262.34[d][4][iii])
  - □ Tried to extinguish the fire, or called the fire department? (40 CFR 262.34[d][4][iv][A])

□ In the event of a spill, contained the flow of H.W., and cleaned up as soon as possible? (40 CFR 262.34[d][4][iv][B])

□ Determined if the emergency is threatening human health or surface water outside the facility, and if so, called the National Response Center at (800) 424-8802 and reported: The generator's name, address, and ID No.? Date, time, and type of incident? Quantity

and type of H.W. involved? Extent of any injuries? and Estimated quantity and disposition of any recovered material? (40 CFR 262.34[d][4][iv][C])

- □ If the generator exceeded the applicable storage time or quantity limit without an EPA extension, did they comply with all TSD storage facility regulations? (40 CFR 262.34[e-f])
- □ GENERATOR STANDARDS
  - □ Has the generator made a hazardous waste determination, by determining if the waste is: (40 CFR 262.11)
  - Excluded from regulation under 261.4? (40 CFR 262.11[a])
  - Listed as a hazardous waste in part 261, Subpart D? (40 CFR 262.11[b])
  - Exhibits a characteristic by testing waste of applying knowledge of the waste? (40 CFR 262.11[c])
  - Excluded or restricted under Parts 264, 265, or 268? (40 CFR 262.11[d])
  - □ Is the waste an exempt recyclable material? (40 CFR 261.6[a][3])
  - □ Industrial ethyl alcohol is reclaimed (unless provided otherwise is an international agreement)? (40 CFR 261.6[a][3][i])
  - □ Used batteries or cells returned to the manufacturer for regeneration? (40 CFR 261.6[a][3][ii])
  - □ Used oil not burned for energy recovery? (40 CFR 261.6[a][3][iii])
  - □ Scrap metal? (40 CFR 261.6[a][3][iv])
  - □ Specific steel (K087) and petroleum refinery production waste? (40 CFR 261.6[a][3][v-ix])
  - □ If the waste is any of the following recyclable materials, complete Parts 270 (permits and notifications), and 266 Subparts A-G of the TSD checklists? (40 CFR 261.6[a][2])
    - □ Those used in a manner constituting disposal (Subpart C)? (40 CFR 261.6[a][2][i])
    - □ H.W.'s burned for energy recovery in boilers and industrial furnaces not regulated as an incinerator (Subpart D)? (40 CFR 261.6[a][2][ii])
    - □ H.W. characteristic used oil that is burned as above (Subpart E)? (40 CFR 261.6[a][2][iii])
    - □ Those from which precious metals are reclaimed (Subpart F)? (40 CFR 261.6[a][2][iv])
    - □ Spent lead-acid batteries that are reclaimed (Subpart G)? (40 CFR 561.6[a][2][v])

#### □ GENERATORS

- □ Does the generator prepare a complete manifest according to the instructions (see Appendix) before transporting H.W. off site? (40 CFR 262.20[a])
- Does the generator designate on the manifest one facility which is permitted to handle H.W.? (40 CFR 262.20[b])
- □ Has the facility designated an emergency alternative facility? (40 CFR 262.20[c])
- □ Instructed the transporter to return the waste to the generator in the event an emergency prevents delivery? (40 CFR 262.20[d])
- □ Did the generator use the supplied manifest required by a consignment State? (40 CFR 262.21)
- □ Where the receiving facility is? or, if not provided by the State? (40 CFR 262.21[a])
- □ Where the generating facility is? (40 CFR 262.21[b])
- □ If not provided by either State, the EPA form from another source? (40 CFR 262.21[c])
- □ Did the manifest consist of enough copies? (40 CFR 262.22)
- Did the generator: Sign the manifest by hand? Obtain the signature of initial transporter and date of acceptance on manifest? Keep one copy of the manifest (per 262.40[a])? (40 CFR 262.23[a])
- □ Did the generator give the remaining copies of the manifest to the transporter? If the shipment was sent by water or rail, was 262.23 complied with? (40 CFR 262.23[b])

#### PRE-TRANSPORT REQUIREMENTS

- □ Is waste packaged in accordance with DOT packaging requirements (49 CFR 173, 178-9)? (40 CFR 262.30)
- □ Are waste packages labeled in accordance with DOT regulations (40 CFR 172.101)? (40 CFR 262.30)
- □ Are containers marked in accordance with DOT regulations (49 CFR 172.101)?
- □ Proper shipping name (table column 2)?
- □ Proper ID number (table column 3A)?
- Proper ORM designation for containers of ORM-A, B, C, D, or E wastes? (40 CFR 262.32[a])

#### Terracon

□ Are containers of 110 gallons or less marked with following words?

"HAZARDOUS WASTE - Federal law prohibits improper disposal. If found, contact the nearest police or public safety authority or the U.S. Environmental Protection Agency. Generators Name and Address, Manifest document number. (40 CFR 262.32[b])"

- Does the generator placard or offer the initial transporter the appropriate placards (49 CFR 172, Subpart F): (40 CFR 262.33)
- □ The generator may accumulate at or near the point of initial generation up to 55 gallons of H.W., or one quart of acutely hazardous waste provided? (40 CFR 262.34[c][1])
- □ H.W. from containers not in good condition or leaking were transferred into good containers?
- □ Containers are compatible with the H.W. stored in them?
- □ Containers are stored closed? (40 CFR 262.34[c][1][i])
- □ The containers are marked either with the words "Hazardous Waste" or labels which identify the contents? (40 CFR 262.34[c][1][ii])
- □ If the generator does not have interim status (as a TSD facility), have they accumulated H.W. on site for less than 90 days? (40 CFR 262.34[a])
- □ Are containers visibly marked with date accumulation started? (40 CFR 262.34[a][2])
- □ Is each container or tank clearly marked with the words "Hazardous Waste"? (40 CFR 262.34[a][3])
- Does the generator comply with the requirements of 40 CFR, Part 265?
- □ Subpart I for the use and management of containers?
- □ Subpart J for tanks (except 265.19[c], closure of tanks without secondary containment, and 265.200)?
- □ 265.111 for tank closure performance standards?
- □ 265.114 for tank decontamination after closure?
- □ Subpart C for preparedness and prevention?
- □ Subpart D for contingency plan and emergency procedures?
- □ 265.16 for personnel training? (40 CFR 262.34[a][1]-[4])
- □ If the generator has stored H.W. on-site for more than 90 days, have they:

- □ Been granted an extension from the EPA?
- □ Complied with the 40 CFR Parts 264 and 265 and the permitting requirements in Part 270 of RCRA? (40 CFR 262.3[b])



#### E.2240 SITE SECURITY PROCEDURES

Last Review or Revision: June 2010

#### Objective

To establish procedures for the security of the subject site and remediation systems during hours when employees are not present.

#### Equipment

- a) Keyed-a-like locks.
- b) Site key file box.
- c) Site diagrams.
- d) Operation and Maintenance procedures.
- e) Materials specified by the Project Manager.

#### Procedures

a) Site Security

The Project Manager will determine the specific security requirements for each site. Coordination with the client may be necessary to provide additional keys if needed. Keyeda-like locks should be the same throughout the entire system. A benchstock of commonly used locks is maintained for the field crews to replace broken or damaged locks. If open excavations or sensitive materials are located on-site, the Project Manager may specify the use of access control, such as fencing or barricading.

#### b) Key Control

Keys for each system will be maintained in the central key control box in the Terracon Office. Additional keys may be maintained by field crews and the project manager for routine access. The site keys will be contained on a tab and clearly marked with the site name, job number and location.

#### c) Site Monitoring

The field crew conducting field services is responsible for the security of the site. A final review must be conducted daily prior to leaving the site to check and document site security. The field crew will complete the "Daily Job Report" to indicate that the site is secure or that exceptions were encountered at the site. The project manager will then be able to schedule the necessary corrective action.

#### d) Site Documentation

Documentation must be maintained at each remediation site to assist field crews in routine operation, maintenance, and emergency activities. The following documents are to be maintained on site:

- 1. Site diagram of all wells associated with the site;
- 2. Schematic of the system including free product and water lines;
- 3. Electrical diagram;
- 4. Site Safety Plan;
- 5. Maintenance checklist and schedule;
- 6. Current settings and operational limits of the system (switch settings, and flow rates);
- 7. Summary of system operational characteristics (flow rates, inventory levels,
- 8. Operational and Maintenance manuals for all system components;

All components of the system are to be labeled for identification. This includes pump controls, flow lines, product lines, level probes, and valves.

e) Signs

Each site should have a sign identifying the site and indicating critical information concerning the operation of the system in the event unusual operations are encountered. This could include the client's name and phone number, or Terracon's name and phone number.

f) Vehicles

Secure all vehicles left on-site during non-working hours. Lock all doors and close all windows. Store the keys in the central key control box.

Following is an example of a Daily Job Report. Either this form or another similar form will be utilized to document daily on-site activities.

TSOP E.2240	Terracon Job No
	Test Date
DAILY JOB REPORT	Time on Job
Job Name	Time in Lab
Job Location	Time in Office
Contractor	Travel Time
Client	TOTAL CHARGEABLE HOURS
	MILEAGE
Description of technical and/or engineering field operations (show direction of north), elevations, and other information:	

TERRACON states that the above tests and/or field engineering services have been performed and the results are reported herein. This report, however, does not relieve the contractor of the responsibility to comply with the plans and specifications.

\*Indicate when the field test data is estimated, pending final laboratory test results.

Position: \_\_\_\_\_

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

TERRACON		
By:		
, <u> </u>		

FORM 113 - 6/85

#### E.2405 CLEANING – GENERAL

#### LAST REVIEW OR REVISION: June 2010

#### **OBJECTIVE AND APPLICATION**

To prepare the equipment for field activities in a manner that minimizes the potential for obtaining biased or erroneous data due to contaminant transfer. Cleaning is performed as a quality assurance measure and a safety precaution. It minimizes cross-contamination between samples and also helps to maintain a clean working environment. This procedure provides general guidelines and should be used in conjunction with more specific procedures applicable to the cleaning method used.

#### EQUIPMENT

- As determined by the project manager
- Expendable supplies:
  - Disposable chemical-resistant gloves
  - Garbage bags
  - Aluminum foil or plastic
  - Laboratory glassware detergent such as Alconox or Trisodium Phosphate (TSP)
  - Containers for collection of waste liquids, if necessary
  - Dilute acid, methanol, ethanol, isopropyl alcohol or other cleaning fluid
- Source of potable water without chemicals that would interfere or be identified in chemical analysis of samples. The project manager may require laboratory testing of cleaning water as a background for evaluating chemical analyses.

#### PROCEDURES

Cleaning procedures will vary considerably based on the equipment, type of contaminant, type of sample and detection levels. Initial cleaning should take place at the site prior to demobilizing. This will minimize the spread of contamination. The extent of on-site cleaning will vary based on specific conditions; however, an attempt should be made to decontaminate as thoroughly as possible on site. The more care one applies on keeping the equipment clean, the less energy will be required on cleaning.

All field equipment must be prepared at the laboratory/office prior to use. This will include additional cleaning, inspection, and maintenance.

Equipment such as hand trowels, bailers, mixing bowls, hand augers, etc., should be cleaned and wrapped in aluminum foil (with shiny side out) or plastic, as appropriate, prior to mobilization.

Sampling and monitoring equipment is normally cleaned by washing and rinsing with liquids such as a soap or detergent solution, potable tap water, deionized water (DI), isopropyl alcohol, methanol, or a dilute acid.

The extent and type of contaminant will determine the degree of cleaning. If the level of contamination cannot be readily determined, cleaning should be based on the assumption that the equipment is highly contaminated.

Waste products produced by the cleaning procedures such as waste liquids, solids, gloves, used Chem-wipe® cleaning pads, etc., should be collected, stored in USDOT-approved 55-gallon drums on-site and disposed based on the nature of the contaminant. Specific details for the handling of these wastes should be addressed by the project manager.

#### STANDARD OPERATING PROCEDURE

#### E.2410 CLEANING – MANUAL WASHING

#### LAST REVIEW OR REVISION: June 2010

#### **OBJECTIVE AND APPLICATION**

To prepare the equipment for field activities in a manner that minimizes the potential for obtaining biased or erroneous data due to contaminant transfer between sampling locations. Cleaning is performed as a quality assurance measure and a safety precaution. It minimizes cross-contaminants between samples and also helps to maintain a clean working environment.

#### EQUIPMENT

- As determined by the project manager
- Expendable supplies:
  - Disposable chemical-resistant gloves
  - Chem-wipe® cleaning pads
  - Garbage bags
  - Laboratory glassware detergent such as Alconox or Trisodium Phosphate (TSP)
  - Containers for collection of waste liquids, if necessary
  - Dilute acid, methanol, isopropyl alcohol, ethanol or other cleaning fluid
- Wash rack facility
- Cleaning containers with brushes (plastic, steel or stainless steel buckets)
- Aluminum foil or plastic
- Source of potable water without chemicals that would interfere or be identified in chemical analysis of samples. The project manager may require laboratory testing of cleaning water as a background for evaluating chemical analyses.

#### PROCEDURES

Cleaning procedures will vary considerably based on the equipment, type of contaminant, type of sample and detection levels. Initial cleaning should take place at the site prior to demobilizing. This will minimize the spread of contamination. The extent of on-site cleaning will vary based on specific conditions; however, an attempt should be made to decontaminate as thoroughly as

possible on site. The more care one applies on keeping the equipment clean, the less energy will be required on cleaning.

All field equipment must be prepared at the laboratory/office prior to use. This will include additional cleaning, inspection, and maintenance.

Equipment such as hand trowels, bailers, mixing bowls, hand augers, etc., should be cleaned and wrapped in aluminum foil (with shiny side out) or plastic, as appropriate, prior to mobilization.

Sampling and monitoring equipment is normally cleaned by washing and rinsing with liquids such as a soap or detergent solution, potable tap water, deionized water (DI), methanol, isopropyl alcohol or a dilute acid.

The extent and type of contaminant will determine the degree of cleaning. If the level of contamination cannot be readily determined, cleaning should be based on the assumption that the equipment is highly contaminated.

Listed below is a cleaning procedure which may be employed for field equipment such as a water level indicator at a monitoring well which contains dissolved petroleum hydrocarbons. If different or more elaborate procedures are required, they should be specified by the project manager during the project initiation meeting.

- Remove gross contamination from the equipment using a Chem-wipe® cleaning pad or brush.
- Wash with a soap or detergent solution
- Rinse with D.I. water
- Rinse with methanol or isopropyl alcohol (if method requires) and repeat rinse with D.I. water
- Repeat the entire procedure or any part of the procedure as necessary.

Waste products produced by the cleaning procedures such as waste liquids, solids, gloves, used Chem-wipe® cleaning pads, etc., should be collected and disposed of based on the nature of the contaminant. Specific details for the handling of these wastes should be addressed by the project manager.

#### STANDARD OPERATING PROCEDURE

#### E.2420 CLEANING – HIGH-PRESSURE, HOT-WATER WASHING

Last Review or Revision: June 2010

#### Objective

To prepare the equipment for field activities in a manner which minimizes the potential for obtaining biased or erroneous data due to contaminant transfer. Decontamination is performed as a quality assurance measure and a safety precaution. It minimizes cross-contaminants between samples and also helps to maintain a clean working environment.

#### Equipment

- As determined by the project manager
- High pressure hot/cold water washing device or steam cleaner
- Expendable supplies:
- Disposable chemical-resistant gloves
- Chem-wipes
- Garbage bags
- Detergent (Alconox or TSP)
- Containers for collections of waste liquids, if necessary D.I. water
- Dilute acid, methanol, ethanol or other cleaning fluid
- Wash rack or other approved decontamination fluid collection system
- Cleaning containers with brushes (plastic, steel or stainless steel buckets)
- Aluminum foil or plastic, as appropriate
- Safety monitoring devices as specified in the safety plan.

#### Procedures

Decontamination procedures will vary considerably based on the equipment, type of contaminant, type of sample and detection levels. Initial decontamination should take place at the site prior to demobilizing. This will minimize the spread of contamination. The extent of on-site decontamination will vary based on specific conditions; however, an attempt should be made to decontaminate as thoroughly as possible on site. The more care one applies on keeping the equipment clean, the less energy will be required on decontamination.

All field equipment must be prepared at the laboratory/office prior to use. This will include additional decontamination, inspection, and maintenance.

Equipment such as hand trowels, bailers, mixing bowls, hand augers, etc., should be cleaned and wrapped in aluminum foil (with shiny side out) or plastic, as appropriate, prior to mobilization.

Decontamination of larger objects, such as the working end of the drill rig or the downhole tools is accomplished using a high pressure wash.

Sampling and monitoring equipment is normally cleaned by washing and rinsing with liquids such a soap or detergent solutions, tap water, D.I. water, methanol, or a dilute acid.

The extent and type of contaminant will determine the degree of decontamination. If the level of contamination cannot be readily determined, cleaning should be based on the assumption that the equipment is highly contaminated.

Listed below is a decontamination procedure which may be employed for field equipment such as a water level indicator at a monitoring well which contains dissolved petroleum hydrocarbons. If different or more elaborate procedures are required, they should be specified by the project manager during the project initiation meeting.

- Remove gross contamination from the equipment using a chem-wipe or brush.
- Wash with a soap or detergent solution
- Rinse with D.I. water
- Rinse with methanol or other alternate cleaning agent (if method requires)
- Rinse with D.I. water
- Repeat the entire procedure or any part of the procedure as necessary.

Waste products produced by the decontamination procedures such as waste liquids, solids, gloves, chem-wipes, etc., should be collected, stored in USDOT-approved 55-gallon drums on the site, and disposed based on the nature of the contaminant. Specific details for the handling of these wastes should be addressed by the project manager.

### **TEFFECTION** STANDARD OPERATING PROCEDURE

#### E.3000 BULK SAMPLING OF SUSPECT ASBSETOS-CONTAINING MATERIAL (ACM)

#### Last Review or Revision: June 2010

#### I. PURPOSE

The purpose of this standard operating procedure (SOP) is to provide information on the hazards of asbestos and procedures to follow to sample suspect materials for laboratory analysis. The following guidelines contained in this document apply to Terracon personnel who engage in bulk sampling of suspect ACM and are designed to provide standardization with respect to sample collection. This procedure should ensure that potential asbestos-containing material samples are collected in a manner which allows for accurate analysis of the material and that sampling personnel are protected against potential asbestos fiber releases through controlled sampling techniques or appropriate personal protective equipment.

The objective of bulk sampling building materials and components suspected to contain asbestos is to characterize the items that contain asbestos in quantities equal to or greater than 1% or other contaent limit as specified by local or state guidelines. By characterizing the locations and quantities of asbestos-containing materials (ACM), exposure hazards can be greatly reduced.

#### II. BACKGROUND AND REFERENCE

Asbestos has been a common component used in several building materials because of its strength enhancing and fire resisting properties. However, asbestos has been recognized as a human carcinogen and respiratory hazard. Due to its health hazards, building inspections and asbestos bulk sampling is requested for schools and many public or commercial properties prior to building renovation or demolition activities. Therefore, identifying, locating and quantifying materials containing asbestos is essential in the effort to prevent worker exposure to asbestos and prevent environmental contamination.

As a consequence of inhalation of asbestos fibers, a body of federal and state regulations has been developed. Federal regulations pertaining to asbestos are included in AHERA (Asbestos Hazard Emergency Response Act) US EPA 40 CFR 763, Subparts E, F; NESHAP (National Emissions Standards for Hazardous Air Pollutants (EPA 40 CFR 61); OSHA Asbestos Standards (29 CFR 1910.1001 and 29 CFR 1926.1101), and ASHARA (Asbestos School Hazard Abatement Reauthorization Act). Many states and local

authorities have additional requirements including state-specific licensing and certification. Terracon will comply with applicable federal, state and local regulations when conducting asbestos-related services.

#### III. EQUIPMENT

The minimum equipment necessary to conduct bulk sampling of suspect materials, in addition to the personal protective equipment outlined below in the Health and Safety Section, is listed below.

- Utility Knife, Chisel, Hammer, Screwdriver, Coring Tool
- Duct Tape
- Sample Containers (preferably ziplock-style clear plastic bags)
- Sample Labels and Indelible Marker
- Spray Atomizer containing Detergent Amended Water, Paper Towels/Wet Wipes
- Spray Adhesive
- Roof Patch Kit (if necessary)
- Measuring Wheel
- Camera
- Flashlight
- Field ACM Sample Log

#### IV. CERTIFICATION

Individuals conducting asbestos sampling must have the certifications listed below. Copies of these certifications and licenses should be taken to the site during the sampling event.

- United States Environmental Protection Agency Building Inspector training (and refresher training, if applicable)
- Asbestos Inspector State-license for the state of the project location (where necessary)

In addition, Terracon requires company-based training courses and hands-on experience of employees prior to commencing asbestos-related field services. Each employee must also receive respirator training, be medically monitored and successfully pass a fit-test utilizing issued respirator(s).

#### V. HEALTH AND SAFETY

Asbestos has been recognized to cause asbestosis, cancer of the lungs and digestive tract and mesothelioma. Asbestosis is a lung disorder characterized by a diffuse interstitial (between cell) fibrosis. The onset of asbestosis probably depends upon the asbestos dust concentration, the morphology of the fiber and length of exposure. Cigarette smoking is strongly implicated as a co-carcinogenic among asbestos workers.

Under the OSHA asbestos standards, the employer has an obligation to protect employees against exposure to asbestos fibers in excess of 0.1 fibers per cubic centimeter of air (0.1f/cc). Personnel engaged in asbestos-related activities (including building inspections) must be trained, medically cleared and fit-tested for respiratory protection. Therefore, enrollment in a medical surveillance program in compliance with the OSHA asbestos and respiratory protection standards is mandatory. Terracon employees are not permitted to engage in asbestos-related activities unless they are enrolled in the Terracon medical surveillance program and have been medically cleared for respirator use by a physician.

The following safety and health protocols apply to Terracon personnel who engage in asbestosrelated services. The guidelines contained in this document are based upon potential health hazards from exposure to asbestos fibers and physical hazards which may be encountered on survey project sites. Field activities will be performed in accordance with the procedures outlined in this document and applicable federal/state health and safety regulations.

Terracon personnel will use professional judgment during sample collection to prevent exposure to other building occupants. If unauthorized personnel attempt to enter a sampling area which could reasonably pose a fiber release hazard, the inspector will curtail bulk asbestos sample collection activity and request that the individual(s) leave the work area. If unauthorized personnel refuse to leave the work area, immediately contact the Project Safety Officer and/or a client representative. Sample collection activities should recommence only after unauthorized personnel have left the work area.

In the event that minor amounts of suspect asbestos containing materials such as thermal system insulation, sprayed-on or trowled-on surfacing materials, ceiling texture, etc. are released during the course of sampling, sampling team members will immediately evacuate the area and don Level C personal protective equipment. The area of potential ACM release will then be approached and suspect materials will be thoroughly wetted with amended water, slowly and deliberately swept to a centralized pile, re-wetted, and containerized in heavy mil asbestos disposal bags. Affected surfaces will then be re-wetted and swabbed with clean cloths or paper towels. Used wipes will be disposed of as asbestos-containing waste.

In the event that large quantities of potential ACM is released during sample collection activities, personnel will immediately evacuate the area and notify the Project Safety Officer and the client representative. The Project Safety Officer will request that the area be sealed until a properly attired response team can be mobilized to the area with a high efficiency particulate air filter (HEPA) vacuum and other equipment necessitated by site conditions.

If suspect materials are in deteriorated condition and fiber release appears likely, or if sampling must be conducted overhead and/or above drop ceilings, personnel will upgrade to Level C personal protective equipment as itemized above. Level C personal protective

equipment should be donned before moving drop ceiling panels, attic access panels, etc. where friable fireproofing or thermal system insulation are known to be present.

The indicated personal protective equipment shall be mobilized to asbestos sampling project sites on each day of sample collection and utilized, if necessary:

- Tyvek (standard) protective coveralls
- Half face or full face air purifying respirator equipped with HEPA (P-100) cartridges
- Impermeable gloves (nitrile or latex).
- Tyvek boot covers or washable outer footwear

Additional Health and Safety protocols such as those established by the owner/operator of the project site and Terracon's company policy regarding ladder safety, confined space entry and electrical hazards shall be followed.

#### VI. SAMPLING HAZARDS

#### a. Elevated Surfaces

Asbestos building inspections may include roofing materials and ceiling spaces containing suspect ACM. Appropriate ladders or other suitable devices (e.g., manlifts) will be used for gaining access to elevated sampling locations. Ladders will be inspected prior to use. Spreaders will be fully extended on all step ladders and firmly positioned prior to use. Where footing is uncertain, a sample team member will hold or otherwise secure ladders while in use by another sample team member. Personnel must always face ladders during both ascent and descent. Extension ladders will not be positioned more than one-quarter of their working length from buildings, walls, etc. (4:1 pitch). Sample team personnel will not walk on steeply pitched roof surfaces and will not walk on low pitched roofing surfaces while wet. Remain on designated roof walkways wherever present. Terracon personnel will visually inspect roofs prior to beginning sample collection activities and will avoid all areas which appear to be structurally unsound.

#### b. Confined Space Entry

Terracon asbestos inspectors will not enter any pit, shaft, tunnel, etc. which has limited means of egress, the potential for an oxygen deficient or toxic atmosphere or which was not designed for human occupancy without first developing a written safety plan which includes a confined space entry permit and procedures. Readily accessible spaces such as pipe tunnels in which personnel may stand can be entered to a distance where continuous visual and verbal communication can be maintained with another sample team member.

Adequate portable lighting must be utilized during sample collection in tunnels and similar spaces. No Terracon or sample team member may attempt to walk through a pipe tunnel,

etc. beyond the sight of a stand-by team member unless written confined space entry procedures have been prepared for the project.

#### c. Electrical Contact Hazards

Personnel will remain cognizant of the location and condition of electrical wiring during the collection of bulk asbestos samples. A visual assessment of each work space will be made prior to sample collection and electrical contact hazards will be evaluated. Unguarded junction boxes, exposed wiring, knife switches, etc. will be avoided during the collection of bulk ACM samples, and coring tools will not be used in near proximity to electrical switches or receptacles.

#### VII. PROCEDURES FOR BULK SAMPLING OF SUSPECT ACM

The primary purpose of this section is to identify the methods and techniques of controlled sampling, sampling site control and use of appropriate personal protective equipment to protect Terracon personnel and members of the general public from exposure to asbestos fibers during sampling activities. Adherence to these procedures should enhance personnel safety during sample collection activities and aid in the suitability of samples for analysis. Field activities will be performed in accordance with the procedures outlined in this document and applicable federal/state health and safety regulations.

Protocols for inspection and bulk sampling are defined in AHERA regulations. These are applicable for any type of survey; for example, a school, an area prior to renovation, a building prior to demolition and inspections undertaken to rebut the OSHA presumption that certain materials contain asbestos.

An accredited Building Inspector must perform the inspection. A summary of AHERA sampling protocols is as follows:

1. Visually inspect the building interior and/or exterior and identify locations of suspect ACM. Identify homogenous areas of friable and non-friable suspect ACM. Document locations, condition, classification and estimated quantities of each suspect material. It is recommended to depict locations of materials on a building diagram and take photographs of sampled materials.

2. Touch each suspect ACM to determine its friablity.

3. Collect representative samples of suspect ACM. Terracon recommends a minimum of three (3) samples of each material be collected from each homogeneous area. However, specific materials may require additional samples such as surfacing material and insulation

as described below. Judgment should be used on the need for and quantity of additional sample collection.

*Surfacing materials*: Collect, in a statistically random manner, at least 3 bulk samples from each homogenous area of 1,000 sq. ft. or less, at least 5 bulk samples from each homogenous area that is greater than 1,000 sq. ft. but less than or equal to 5,000 sq. ft., and at least 7 bulk samples of each homogenous area larger than 5,000 sq. ft.

*Thermal System Insulation (TSI)*: Collect, in a randomly distributed manner, at least three bulk samples from each homogeneous area of TSI; collect at least one bulk sample from each homogeneous area of patched TSI if the patched section is less than 6 linear or square feet; collect bulk samples from each insulated mechanical system where cement or plaster is used on tees, elbows, etc. in a manner sufficient to assess whether the material is ACM.

If fiber release appears likely, wet methods will be employed in the collection of suspect ACM samples. Water amended with a minimum of 10% commercially available window cleaning solution or other suitable surfactant should be used to moisten materials prior to sampling. Bulk asbestos samples will not be collected over the heads of, or in near proximity to, non-project personnel. Respiratory protection is not required when sampling non-friable suspect materials or materials below the breathing zone which are adequately wetted with amended water.

4. Bulk ACM samples should be immediately placed in sample containers and sealed while the materials are wet. If collecting samples of friable ACM or normally non-friable materials which are in deteriorated condition, precautions must be taken to prevent the release of fibers to the work area. Precautions include aggressively wetting the surface or potentially isolating the material (e.g. glove bagging) prior to disturbance.

5. Reusable sampling equipment will be gently rinsed with amended water. Dry the equipment with paper towels to be disposed of as contaminated materials.

6. The following decontamination sequence should be used following sample collection activities requiring Level C personal protective equipment:

- Remove coveralls slowly turning the outside inward.
- Place in heavy mil asbestos disposal bag.
- Remove gloves and shoe covers (if utilized)
- Remove respirator and carefully dispose of respirator cartridges in asbestos disposal bag.
- Securely seal protective clothing and any potentially contaminated disposable sampling equipment in heavy mil asbestos disposal bags.
- Do not smoke or eat with soiled hands.

- Wash hands, face and forearms thoroughly before eating, drinking smoking or using toilet facilities.
- Shower thoroughly as soon as possible upon leaving the project site.

7. Bulk sample analysis for asbestos content is performed by polarized light microscopy (PLM). The analytical testing procedure is based on U.S. Environmental Protection Agency (EPA) methods and National Voluntary Laboratory Accreditation Program (NVLAP) requirements. Terracon will use NVLAP accredited and appropriately licensed laboratories for analysis of asbestos bulk samples. Samples will be controlled with the analytical laboratory through chain of custody documentation.

## **TEFFECON** STANDARD OPERATING PROCEDURE

#### E.4000 SAMPLING OF POTENTIAL LEAD-BASED PAINT

Last Review or Revision: June 2010

#### I. PURPOSE

The purpose of this standard operating procedure (SOP) is to provide information on the hazards of lead-based paint (LBP) and describe procedures for identification and proper LBP sampling techniques. The following procedures for sampling potential LBP using a direct reading instrument and paint chip collection are designed to provide standardization with respect to location and number of samples collected and method of labeling sample locations. In addition, this procedure should ensure that potential LBP samples are collected in a manner which allows for accurate analysis of the material. Finally, this procedure will help to ensure that sampling personnel are protected against potential lead dust releases through controlled sampling techniques or appropriate use of personal protective equipment.

The objective of sampling coated (i.e., painted, pigmented or stained) surfaces for LBP (LBP) is to characterize materials and components that contain lead in surface coatings and compare them to established limits such as the Environmental Protection Agency (EPA) or Housing Urban Development (HUD) standards and guidelines such as at quantities greater than 1.0 milligram per square centimeter (mg/cm<sup>2</sup>) or 0.5% by weight or other regulated quantity as specified by state or local authority. By characterizing the locations and quantities of LBPs, exposure hazards can be greatly reduced.

#### II. BACKGROUND AND REFERENCE

Lead is a toxic heavy metal which may cause blood, kidney and nervous system disorders if inhaled or ingested. Metallic lead and lead contained in dusts are not readily absorbed through the skin. However, skin contact with potentially contaminated site materials should be avoided.

The United States EPA and HUD have established an action level for LBP of 1.0 milligram per square centimeter (mg/cm<sup>2</sup>) or 0.5% by weight. Coatings with quantities equal to or greater than these values are considered LBP. The Occupational Safety and Health Administration (OSHA) does not establish a LBP quantity but regulates the amount of lead that can become airborne and either inhaled or ingested by setting limits for air concentrations of 0.05 milligram per cubic meter (mg/m<sup>3</sup>) of air over a work shift and blood values of 50 micrograms per deciliter of blood.

Lead was a common ingredient in paint until 1978 when the Consumer Product Safety Commission (CPSC) banned the sale of LBP for use in residences in quantities greater than 0.06% by weight. However, some industrial paints still contain lead today and are used in several applications. Lead can be introduced into the air by sanding or abrading surfaces containing LBP, and inhalation or ingestion of the dust is possible. Ingestion of lead can occur when children consume deteriorated paint chips, children place exposed toys or body parts into their mouths or when industrial workers exposed to lead-containing dust eat or smoke without washing their hands.

The lead content of paint can be determined with direct-reading instrumentation or by analysis of a bulk paint chip sample. An X-ray fluorescence (XRF) type analyzer is recommended to obtain direct readouts of lead content in coated surfaces. The XRF analyzes for lead by atomic absorption spectroscopy (AAS). Results are in milligrams of lead per square centimeter. Paint chip sample analysis is performed by inductively coupled plasma-atomic emission spectrometry (ICP-AES) in accordance with EPA SW-846 Method 6010B. Results are typically reported as lead percent per paint chip weight.

When sampling for LBP in buildings classified under the United States Department of Housing and Urban Development (HUD) or other child occupied-facilities (i.e., publicly and privately owned-housing, public buildings, daycares, etc.), procedures stated in the most current revision of the Department of Housing and Urban Development *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards Housing*, June 1995 revision will be used.

Lead is also regulated by the Occupational Safety and Health Administration (OSHA) and the EPA. Occupational exposure to lead occurring in the course of construction work, including maintenance activities, painting, renovation and demolition, is subject to OSHA standard (29 CFR 1926.62), Lead Exposure in Construction. Construction work covered by 29 CFR 1926.62 includes any repair or renovation activities or other activities that disturb inplace, lead-containing materials. Employers must assure that no employee will be exposed to lead at concentrations greater than 0.05 mg/m<sup>3</sup> averaged over an eight-hour period without adequate protection.

The Resource Conservation and Recovery Act (RCRA) provides the EPA with the authority to regulate the waste status of demolition or renovation debris, including lead-containing materials. Specific notification and testing requirements must be addressed prior to transporting, treating, storing or disposing of hazardous wastes. Lead containing wastes are considered hazardous waste under RCRA if Toxicity Characteristic Leachate Procedure (TCLP) results exceed 5 milligrams per liter (mg/L). EPA exempts from most RCRA requirements those generators whose combined hazardous waste generation is less than 100 kilograms (kg) per month.

#### III. EQUIPMENT

LBP analysis can be conducted by using a direct reading XRF analyzer or by collecting paint chip samples. Equipment necessary to conduct both methods is listed below.

For direct reading sampling:

- X-ray fluorescence (XRF) analyzer and accessories
- XRF result field log (optional if downloading software is used)

For paint chip sampling:

- Heating tool and extension cord
- Tape measure or template
- Chisel
- Chipping hammer or scraper (lead paint samples from metal structures)
- Face shield or chipping goggles
- Sample containers (preferably sturdy, clear plastic vials)
- Sample labels
- Laboratory chain of custody for paint chip sample analysis

#### IV. CERTIFICATION

Individuals conducting LBP inspection services should have the certifications listed below. Copies of these certifications and licenses should be taken to the site.

- EPA lead inspector and risk assessor certification
- As applicable, local- or State-licensed lead inspector/risk assessor (required for HUD projects)
- Manufacturer training certification for the XRF analyzer

#### V. HEALTH AND SAFETY

The OSHA personal exposure limit (PEL) for lead is 0.05 milligram per cubic meter (mg/m<sup>3</sup>) and the action level is 0.03 mg/m<sup>3</sup>. The primary route of exposure of lead is through inhalation of contaminated dusts or by accidental ingestion; however, collection of small sample volumes required for analysis is not expected to generate significant dust. Although, if painted surfaces are being disturbed and dust is generated in the vicinity, personnel will take protective measures as indicated below. Project activities may be conducted in Level D personal protective equipment modified as specified below.

• Lead sampling activities will be performed in Level D personal protective equipment to include standard work uniform, safety footwear and hard hat if overhead hazards are present.

- Protective goggles or a full face shield will be worn during chipping hammer operations.
- Protective gloves should also be worn during lead chip sampling to prevent abrasion and contact with site materials. Half face disposable dust/fume/mist respirators (3M 9920 "surgical style" masks) or half-face air purifying respirators equipped with HEPA filter cartridges will be worn if dusty conditions develop on-site.

Due to the potential of accidental ingestion when working in areas with lead dust, do not smoke or eat with soiled hands. Wash thoroughly before eating, drinking or smoking. Shower thoroughly as soon as possible upon leaving the site.

The XRF analyzer contains a radioactive source and should be transported and used according to the manufacturer's instructions. Personnel utilizing the equipment shall have the proper training and certifications required for use of the equipment.

#### VI. PROCEDURES FOR XRF ANALYSIS

1. Identify areas with coated or prepared surfaces. This includes building materials, components and fixtures finished with a coating such as paint, stain and varnish. Wallpaper can mask prepared surfaces and should be included in the survey. Some ceramic tiles have a lead-containing glaze and should also be assessed, particularly where required by state or local regulatory agencies.

2. Select appropriate materials and locations to be sampled. When sampling painted interior surfaces, representative samples must be obtained per client instruction or in general compliance with the most current revision of the Department of Housing and Urban Development *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards Housing*, June 1995revision.

3. Calibrate the XRF unit according to the manufacturer's instructions before and after the survey. This involves taking calibration sample readings from a known source provided by the manufacturer.

4. Conduct XRF sampling on selected surfaces. Document descriptions of each surface sampled on the XRF result field log or using the manufacture's software. Record results on the XRF result field log if software is not used.

If results yield 1.0 mg/cm<sup>2</sup> (inconclusive), then a paint chip sample should be collected using the applicable procedures indicated in the following section.

If results indicate the presence of lead in quantities greater than 1.0 mg/cm<sup>2</sup>, it is recommended that a photograph depicting the material and location be taken.

#### VII. PROCEDURES FOR PAINT CHIP SAMPLING

1. Identify areas with prepared surfaces. This includes building materials, components and fixtures finished with a coating such as paint, stain and varnish. Wallpaper can mask prepared surfaces and these areas should be included in the survey.

2. Select appropriate materials and locations to be sampled. When sampling painted interior surfaces, representative samples must be obtained per client instruction or in general compliance with the most current revision of the Department of Housing and Urban Development *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards Housing*, June 1995revision.

3. A heating tool and chisel is recommended to collect representative samples of painted surfaces, but a knife, chipping hammer or paint scraper may also be used. Minimum force should be used to prevent the generation of dusts and particles. Wear protective goggles, abrasion resistant gloves and/or particulate respirator as appropriate to the task.

Check with the analytical laboratory you will use to determine minimum sample size required. A two square inch (2 in<sup>2</sup>) sample is recommended for each sample. The sample size should be documented on the sample log. Some laboratories conducting toxic characteristic leaching procedures (TCLP) analysis may request up to ten grams (10 g) per sample. Lead-based paint samples must be removed down to the bare substrate to ensure each layer of paint has been collected. Use a brush or mini-vacuum to clean up residual material and place it in the sample container.

4. Assign a sample number to each sample collected. Affix a label or mark the sample container indelibly with a sample identification number. Seal the sample container securely. Document descriptions and locations of each surface sampled on a field log. It is recommended that a photograph depicting the material and location be taken.

5. Send results and chain or custody to an American Industrial Hygiene Association (AIHA) accredited laboratory for analysis.



#### APPENDIXC

Terracon's Corporate Quality Program Manual

# Corporate Quality Program Manual

#### **Prepared by:**

Quality Program Committee

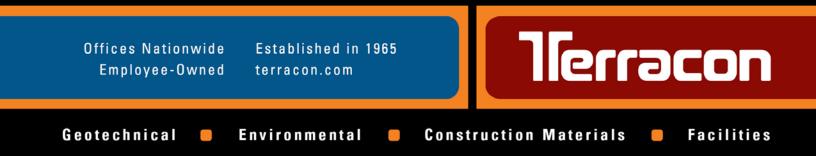
#### Adopted by:

Operations Committee January 1991

#### **Revision:**

Revision 4.1 June 23, 2010







#### TABLE OF CONTENTS

1.	. OVERVIEW		
	1.1	CORPORATE PHILOSOPHY ON QUALITY SERVICE	. 1
	1.2	PROGRAM COMPONENTS	. 1
	1.3	PROGRAM IMPLEMENTATION	.2
2.	DEFI	NITIONS	.2
3.	QUAI	LITY PROGRAM AT THE PROJECT LEVEL	
	3.1	INDIVIDUALS' RESPONSIBILITY	.4
	3.2	PROJECT MANAGER RESPONSIBILITY	.4
	3.3	PROJECT REVIEW	.4
	3.4	SPECIFIC QUALITY CONTROL AND QUALITY ASSURANCE	
		RESPONSIBILITIES	.7
4.	QUAI	LITY PROGRAM AT THE CORPORATE LEVEL	
	4.1	EXECUTIVE VICE PRESIDENT FOR QUALITY AND SERVICE	.9
	4.2	OPERATIONS COMMITTEE	.9
	4.3	QUALITY PROGRAM COMMITTEE	.9
	4.4	TECHNICAL SERVICE LINE COMMITTEES	.9
	4.5	OFFICE PEER REVIEW PROGRAM	.10
	4.6	MANUALS & PRACTICE GUIDELINES	.10
5.	COM	MITMENT TO QUALITY	. 10

# Terracon

#### 1. OVERVIEW

#### 1.1 Corporate Philosophy on Quality Services

Terracon has adopted a Quality Program as described in this Corporate Quality Program Manual (QPM). Terracon's Quality Program is based on a continuous improvement philosophy that involves our employees and, when applicable, subconsultants, vendors, and users of our services. Quality service for our clients is achieved by following the methods and practices that have been developed at Terracon for the services being provided.

Terracon is committed to providing quality services to our clients and assigning qualified and dedicated staff to every project. This commitment is a key component of our "Delivering Success to Clients and Employees" mission statement. Commitment to quality begins with the President and extends to every member of Terracon's staff. Terracon is committed to providing professional services that:

- are consistent with professional practice;
- provide accurate tests and measurements;
- are openly communicated with our clients; and
- adhere to the agreed upon contractual requirements.

Terracon also continuously seeks to find improvements in practices and procedures to better serve our clients needs. Services are supervised by senior professionals and managers who have experience and knowledge in the service being provided. Terracon will not perform work for which we are not adequately experienced or knowledgeable, or if we are unable to provide an appropriate quality of service.

#### 1.2 Program Components

Terracon has established specific operational positions, procedures, practices, and programs to ensure quality service to our clients at the individual project level. They include:

- lines of responsibility, authority, and accountability;
- standard processes and practices;
- quality control of project execution; and
- project quality assurance

Terracon also continually evaluates internal policies at the corporate and office levels by examining individual implementation of these policies. Policy evaluation is accomplished through the following Corporate-level positions, committees, policies, and practices:

- Executive Vice President for Quality and Service
- Operations Committee
- Quality Program Committee
- Service Line Committees
- Office Peer Review Program



Manuals and Practice Guidelines

Each of these positions, committees, policies, and practices combine to help us achieve quality service for our clients. Each is discussed in more detail below.

#### 1.3 Program Implementation

The Executive Vice President of Quality and Service (EVP-QS) is responsible for developing and monitoring the Quality Program. Terracon's Legal Department maintains the QPM and related materials. Program implementation at the corporate level has been assigned to the Service Line Directors. Implementation functions include preparation, distribution, and revision of program documents; staff training; and internal system review.

Responsibility for promoting and enforcing the Quality Program at the operational level lies with the Division Managers. These responsibilities include promoting the program within their divisions; assigning responsibility, authority and accountability to appropriate managers; and enforcing implementation. Office Managers, Project Managers, Authorized Project Reviewers, LEVEL 1 Reviewers, and staff are responsible for day-to-day implementation of the Quality Program for project work.

#### 2. DEFINITIONS

As used in this Manual, the following terms have the following meanings:

<u>Authorized Project Reviewer (APR)</u> is a senior technical professional within Terracon who has been recognized in his/her service line as authorized to review project deliverables and performance as required in our Project Quality Review Manual. An APR is authorized to perform a LEVEL 1, LEVEL 2, or LEVEL 3 review in his/her area of experience.

<u>**LEVEL 1 Reviewer</u>** is approved by the Office Manager and authorized to review project deliverables or tasks relating to field and laboratory observations and test data collection that do not contain opinions, recommendations, or conclusions.</u>

<u>Manuals</u> are documents prepared by Service Line Committees or corporate staff to describe Terracon's operational and administrative procedures and practices to meet quality objectives. Manuals are prepared as needed to cover specific practices or services (e.g., Quality Systems Manual for Construction Material Laboratory, Project Quality Review Manual, etc.).

<u>Office Peer Review</u> is an independent review of an individual office's operations that is conducted by Terracon's Legal Department along with senior managers from outside the Division. The review focuses on quality of services, safety, risk management practices, and business conduct and ethics and is performed in accordance with the Terracon Internal Peer Review Program Manual.



<u>**Practice Alerts</u>** are communications by the various Service Line Committees to notify practitioners of current issues in the practice. The publication's purpose is to communicate timely information and provide technical references to resources on a current topic affecting the service line. This publication is not designed to act as best practice guidelines or establish service line procedures. Publications are prepared as needed.</u>

**<u>Practice Guidelines</u>** are publications prepared by the various Service Line Committees to provide internal best practice guidelines and establish procedures for specific technical services. The publications' purpose is to provide training and guidance for Terracon practitioners. Publications are prepared as needed.

<u>**Project Manager**</u> is a technical professional within Terracon who is responsible for overall quality, product delivery, and client service of projects he/she manages.

<u>Project Quality Assurance Manager (PQAM) is</u> Terracon's assigned technical professional who oversees quality assurance on designated projects that have a project-specific quality assurance program. The Executive Vice President for Quality and Service (EVP-QS) designates specific projects such as nuclear projects, and assigns the PQAM.

<u>Project Quality Review Manual</u> is an internal document where the specific quality control review requirements established by this Manual are set out in detail.

<u>Quality Control</u> is the procedures and practices established for executing our services to achieve the quality objectives. This means establishing those measurements (i.e., standards, specifications, or criteria) and then controlling, monitoring, or measuring the work performed through checks, tests, or confirmations so that quality objectives are met.

<u>Quality Assurance</u> is the assessment process of reviewing, monitoring, and auditing to verify that quality control procedures and practices are fully incorporated and are meeting quality objectives.

<u>Quality Program Committee (QPC)</u> is a committee appointed by the Operations Committee to oversee Terracon's Quality Program. The committee is co-chaired by the EVP-QS and General Counsel, and includes representative members from the respective service lines and operating groups.

<u>Service Line Committee</u> is a group of senior technical professionals within Terracon that is responsible for overseeing the practices and activities of an individual service line.

<u>Service Line Director</u> is the senior technical professional within Terracon appointed to supervise the policies, practices, and activities of our individual service lines.

<u>**TerraNet</u>** is Terracon's electronic intranet, supplying valuable information, policies, and procedures to our employees in an easily accessible format.</u>

<u>Lead Project Auditor (LPA)</u> is Terracon's assigned auditor for quality assurance of project activities and/or testing laboratories for specific projects designated by the EVP-QS.



#### 3. QUALITY PROGRAM AT THE PROJECT LEVEL

Terracon has specific lines of responsibility at all employee levels to ensure quality service. Specific lines of responsibility exist for all phases of each project, and projects are subject to multiple levels of review depending on their size and complexity. These lines of responsibility help to ensure the quality of our service at the project level.

#### 3.1 Individuals' Responsibility

Our primary mechanism for achieving quality on every project lies with the individual performing the work. Each employee of Terracon has responsibilities for professional, technical, or administrative quality, whether on client projects or internal service assignments. Those responsibilities include:

- adhering to the requirements of the Quality Program;
- clearly understanding the needs of the client;
- conducting the assignment in accordance with quality control requirements and documenting quality control where applicable;
- participating in appropriate technical training; and
- supporting and maintaining quality control procedures and initiating system improvements when deficiencies are noted.

#### 3.2 Project Manager Responsibility

Terracon's Project Managers are accountable for the overall quality of projects they manage. Each project is assigned to a technical professional with the necessary skills to manage the project. The assigned professional serves as Project Manager and is responsible for verifying that the applicable quality control criteria, project quality review, and company policies have been followed for all phases of the project. This Project Manager also is responsible for knowing licensing and certification requirements and ensuring that they are met. Checks and reviews are conducted by the Project Manager, as required, as the work progresses.

#### 3.3 Project Review

#### 3.3.1 Levels of Project Review

Terracon provides a wide variety of services to its clients. These services range from simple data acquisition to engineering and project management for large, complex projects. Consequently, the required level of Quality Control varies from project to project. Three Quality Control review levels have been established by this Manual and outlined in specific detail in Terracon's Project Quality Review Manual. Levels 1 through 3 are of increasing intensity.

LEVEL1 review applies to project deliverables or tasks containing field and laboratory observations and test data collection that do not contain opinions, recommendations, or conclusions. It requires the involvement of an approved LEVEL 1 Reviewer.



The remaining levels of review, LEVEL 2 and LEVEL 3, apply to project deliverables that contain opinions, recommendations, and conclusions. They require the involvement of an Authorized Project Reviewer (APR).

- A LEVEL 2 review must be provided by an APR and is required on all project deliverables containing opinions, recommendations, or conclusions regardless of the project size or complexity.
- A LEVEL 3 review is an additional, independent review required for Large/Complex Projects. This is in addition to the required LEVEL 2 APR Review. A LEVEL 3 review must be performed by an APR who works in a different office from the one providing the LEVEL 2 review.

The table on the following page outlines these processes and responsibilities in detail:



#### LEVELS OF REVIEW

Review Level	Project Type	Description and Documentation
LEVEL 1: (Assigned by Project Manager)	Project deliverables or tasks providing field and laboratory observations and test data collection. Do not contain opinions, recommendations, or conclusions.	<ul> <li>(a) Independent check of field and laboratory observations, data and calculations by a LEVEL 1 Reviewer approved by the Office Manager.</li> <li>(b) Documentation by initials and dates on deliverables.</li> </ul>
LEVEL 2: (Assigned by Department Manager)	Projects where opinions, recommendations, or conclusions are provided.	<ul> <li>LEVEL 1 plus:</li> <li>(a) Appointment of APR to review data quality, data interpretation, analytical methods, calculations, results, opinions, recommendations, and conclusions.</li> <li>(b) Document by signature of APR on project deliverables.</li> </ul>
LEVEL 3: (Assigned by Office Manager)	Required on Large/Complex Projects. A large or complex project is one that, because of its size or scope, carries a significant investment by either Terracon or its clients, such as a high-rise structure, large- scale commercial or brownfield development, large industrial manufacturing/processing facility, or an extensive infrastructure/transportation project. NOTE: Certain projects require mandatory LEVEL 3 reviews as outlined in the Project Quality Review Manual.	<ul> <li>LEVEL 2 plus:</li> <li>(a) Additional independent review by an APR in the same discipline outside the local office who is not already involved in the project. Includes proposal review by both LEVEL 2 &amp; 3 APRs.</li> <li>(b) On unique technical projects, an additional independent review by a specialist or consultant from outside the company that is recommended by the LEVEL3 APR and approved by the Office Manager.</li> <li>(c) Dual signatures, with at least one APR, are required on proposal and project deliverables. Written documentation of independent review (form in Appendix J) should be placed in the project file and a copy should be provided to the appropriate Service Line Director. Third signature on project deliverables is not required.</li> </ul>



#### 3.3.2 Project Reviewer Responsibility

All project deliverables or tasks require a secondary review by an additional person who is not the generator of the deliverable or task. The specific level and extent of review depend on the deliverable or task (LEVEL 1, 2, or 3 as described above) and the review must be performed by individuals authorized to perform the required review. LEVEL 1 Reviewers and APRs are selected and approved according to processes outlined in the Project Quality Review Manual.

#### 3.3.3 Quality Assurance for Designated Projects

When appropriate or required by project contract, a Project Quality Assurance Manager (PQAM) will be assigned by the Executive Vice President of Quality and Service (EVP-QS). Such projects include those that are quality affecting of a nuclear facility. It is the duty of the LEVEL 3 Reviewer to notify the EVP-QS of such activities so the PQAM can be assigned. The project manager will prepare a project specific quality program manual approved by the PQAM prior to providing services for such designated projects. The quality control manual should indicate control and verification measures that should be planned, documented and implemented at predetermined points throughout the life cycle of the project. The manual should provide control from initiation of activities through their completion.

Certain types of projects, such as projects involving nuclear facilities, require specific levels of quality assurance. Those projects, which shall be designated by the EVP-QS, will include audits of project activities and/or testing laboratories, scheduled in a manner to provide coverage and coordination with on-going activities based on the importance and status of the activity. The frequency of audits and the selection of the audit team is the responsibility of the EVP-QS. The Lead Project Auditor (LPA) will be designated for each audit by the EVP-QS.

#### 3.4 Specific Quality Control and Quality Assurance Responsibilities

Terracon has established specific Quality Control and Quality Assurance responsibilities for its projects. The Table on the following page outlines and summarizes Terracon's Quality Control and Quality Assurance responsibilities.



#### TERRACON QUALITY CONTROL AND QUALITY ASSURANCE RESPONSIBILITIES

Role	Quality Control (Controlling operations to achieve quality)	Quality Assurance (Quality verification)
Performs project work consistent with company methods, practices, and core values.	Individual responsibility of all staff	
Reviews projects deliverables or tasks relating to field and laboratory observations and test data collection that do not contain opinions, recommendations, or conclusions.	LEVEL 1 Reviewer	
Responsible for overall quality, product delivery, and client service of project.	Project Manager	
Assigns LEVEL 2 APR to projects containing deliverables with opinions, recommendations, or conclusions. Works with the Project Manager to ensure that qualified staff is assigned to the project.	Department Manager	
Provides technical oversight of LEVEL 1 Reviewer. Provides LEVEL 2 review of deliverables containing opinions, conclusions, or recommendations. Provides LEVEL 3 independent review on Large/Complex Projects when required.	Authorized Project Reviewer (APR)	
Responsible for implementing policies and systems necessary to achieve project quality. Determines whether LEVEL 3 review is required on a project and appoints appropriate LEVEL 3 APR.	Office Manager	
Promotes and enforce Quality Program procedures. Assumes responsibility, authority, and accountability for quality of projects within Division.	Division Manager	
Assumes responsibility, authority, and accountability for quality of projects within Operating Group.		Operating Group Manager
Implements Quality Program and recommends continuous improvements.		Service Line Director
Develops and monitors Quality Program.		Executive Vice President of Quality and Service
Sets corporate philosophy regarding quality. Promotes attitudes, processes, and procedures necessary to achieve quality.		President



#### 4. QUALITY PROGRAM AT THE CORPORATE LEVEL

Terracon is committed at a corporate level to maintaining high levels of quality and client service. To that end, Terracon constantly evaluates internal operations to improve the quality of our client service. Various positions, committees, and programs described below have been established to monitor and analyze the performance of our services with an emphasis on improving quality. The President sets corporate policy regarding quality and promotes attitudes, processes, and procedures that are necessary to achieve quality services.

#### 4.1 Executive Vice President of Quality and Service (EVP-QS)

Terracon created a senior officer level position devoted solely to maintaining and improving the quality of our service. The Executive Vice President of Quality and Service (EVP-QS) answers directly to the President. The EVP-QS is charged with continually monitoring the quality of our services and is responsible for developing and monitoring the Quality Program and the Project Quality Review process. This individual works with both practitioners and management to improve our quality of service to our clients, and studies our current operations to determine areas of quality improvement. This position also holds a seat on Terracon's Operations Committee as described below.

#### 4.2 Operations Committee

Terracon's Operations Committee meets regularly to discuss issues and set policy regarding operations and client services. This committee is composed of the President, EVP-QS, and other key senior managers. This committee continuously evaluates the processes and procedures of our operations to find areas of improvement. Client service, product delivery, and achieving quality are top priorities of the Operations Committee.

#### 4.3 Quality Program Committee

Terracon established the Quality Program Committee (QPC) to oversee Terracon's Quality Program. The committee is co-chaired by the EVP-QS and General Counsel, and includes representative members from the respective service lines and operating groups. The QPC meets monthly and continuously evaluates the quality of our services. The QPC's provides oversight to the respective Service Line Committees on quality related issues and designs and implements quality-related improvements to our operations.

#### 4.4 Service Line Committees

Terracon has established specific Service Line Directors and Service Line Committees for the core services we provide to clients. Each committee meets regularly and discusses a variety of issues that relate solely to the performance of the specific service line. These committees seek continual improvement of the quality in the specific service line to better serve our client needs. The committees conduct internal training and publish Manuals, Practice Guidelines, and Practice Alerts to assist employees at all levels.



#### 4.5 Office Peer Review

Independent peer review of office operations is an integral part of Terracon's Quality Program. An attorney with Terracon's Legal Department, along with senior managers from outside the division, reviews the quality, risk management, and loss prevention practices of each office approximately once every five years. For certain smaller or satellite offices, the review is performed solely by an attorney with Terracon's Legal Department. This program includes review of information from and about the offices, an onsite visit, confidential staff interviews and questionnaires, and file review. An oral presentation of the Peer Review Team's findings with the Office and Division Manager is followed by a written report. After the written report is received, the Office Manager prepares a specific action plan report for the President and EVP-QS that addresses necessary measures to correct observed deficiencies. Terracon's General Counsel is responsible for developing and implementing the Office Peer Review with oversight provided by the EVP-QS. The Terracon Internal Peer Review Program Manual describes the program.

#### 4.6 Manuals, Practice Guidelines, and Practice Alerts

Manuals, Practice Guidelines, and Practice Alerts are used to assist staff with the procedures and requirements for specific services. These publications set forth specific practices and procedures for the service line and/or provide timely updates on current technical issues affecting each service line. They are available through individual Service Line Directors or electronically through TerraNet.

#### 5. COMMITMENT TO QUALITY

Terracon is committed to providing quality services with experienced technical and professional staff through a comprehensive Quality Program. As evidence of our commitment to evaluating and improving our quality, we consistently audit our quality service procedures with both internal and external reviews. An officer of the company is responsible for the program and is devoted to improving the quality of our services. As outlined in this Corporate Quality Program Manual, fundamental aspects of the Quality Program include training and experience requirements for various technical and professional positions, senior level deliverable review requirements, and Office Peer Review. Through these programs, our technical and professional staff receives training in technical issues as well as and communication and client service. Quality audits of each office are conducted on a regular basis as part of the Quality Program. With the help of these procedures, we are able to deliver our services on a timely basis with consistently high value and attention to clients needs.

# Terracon

APPENDIX D

Laboratory QA Manuals

# Quality Assurance Manual



12065 LEBANON RD. | MT. JULIET, TN 37122 | (800) 767-5859 | WWW.ESCLABSCIENCES.COM

Version 10.0 4/15/12

## **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.

ESC Lab Sciences Quality Assurance Manual Signatory Approvals

Section: Approvals, Ver. 10.0 Date: April 15, 2012 Page: 1 of 1

#### **COMPREHENSIVE QUALITY ASSURANCE MANUAL**

for

#### ESC LAB SCIENCES 12065 LEBANON ROAD MT. JULIET, TENNESSEE 37207 (615)758-5858

Prepared by

#### ESC LAB SCIENCES 12065 LEBANON ROAD MT. JULIET, TENNESSEE 37207 (615)758-5858

## NOTE: The QAM has been approved by the following people. A signed cover page is available upon request

Peter A. Schulert, CEO 615-773-9660

NCM

Judith R. Morgan, M.S. VP, Director of Technical & Regulatory Affairs 615-773-9657

Eric Jøbrison, VP, Laboratory Director 615-773-9654

Om

Tom Mellette, Client Operations 615-773-9676

arlik 110

Dixie Marlin, B.S., QS Manager, 615-773-9681

Jeff Chandler, B.S., CIO (Information Systems) 615-773-9696

The ESC QAM has been prepared in accordance with the following standards: AIHA (LQAP), A2LA (Env. Prog. Req.), ANSI/ISO 17025-2005, NELAC, DOD QSM.

Section TOC, Ver. 10.0 Date: April 15, 2012 Page: 1 of 8

|--|

ESC/ISO Number	NELAC Ref.	TNI Standard (Vol 1)	Section & SOP references where applicable	Page	Total Pages	SOP Ref
	I		Section 1			
1.0	5.0		General	1	1	
1.1			Index and revision status	1	1	
1.2	5.1		Purpose	1	1	
			Section 2			
2.0			Laboratory Background	1	3	
2.1			Activities	1	3	
2.1.1			Analytical Support and Service Areas	1	3	
2.1.2			Regulatory Compliance and Quality Standards	1	3	
2.1.3	5.4.2.3h		Analytical Capabilities	1	3	
2.2			History	3	3	
			Section 3			
3.0	5.3		Introduction, Scope, and Definitions	1	74	
3.1			Scope of Capabilities	1	74	
3.2			Table Of Contents, References And	1	74	
2.2			Appendices	1	74	
3.3			Definitions and Terminology	1	74 74	
3.4			Abbreviations and Acronyms	40	/4	
1.0			Section 4		20	
4.0	5.4	M2 4.0	Management Requirements	1	30	
4.1	5.4.1	M2 4.1	ORGANIZATION	1	30	
4.1.1	5.4.1.4a	M2 4.1.1	Legal Identity	1	30	
4.1.2	5.4.1.2	M2 4.1.2	Organization	1	30	
4.1.3	5.4.1.3	M2 4.1.3	Facilities Under Management System	1	30	
4.1.4	5.4.1.4b	M2 4.1.4	Independence	1	30	
4.1.5	5.4.1.5	M2 4.1.5	Management Responsibilities and Policies	1	30	
4.1.6		M2 4.1.6	Management System Effectiveness	4	30	
4.2	5.4.2	M2 4.2	MANAGEMENT SYSTEM	6	30	
4.2.1		M2 4.2.1	Management Documentation	6	30	
4.2.2	5.4.1.5h, 5.4.2.2	M2 4.2.2	Quality Management Policy	6	30	
4.2.3	5.4.2.6, 5.4.1.5c	M2 4.2.3	Management System Implementation and Improvement	7	30	Y
4.2.4	5.4.2.2	M2 4.2.4	Commitment to the Client and Regulatory Requirements	7	30	
4.2.4.1	5.4.2.2, 5.4.2.3	M2 4.2.8.3	Quality Manual	8	30	
4.2.4.2		M2 4.2.8.3h	Commitment to the QAM and Related Procedures	8	30	
4.2.5		M2 4.2.5; 4.2.8.5	Procedure List	8	30	
4.2.6	5.4.2.3a	M2 4.2.6	Management Roles and Responsibilities	8	30	
4.2.6.1			Programs	8	30	

Section TOC, Ver. 10.0 Date: April 15, 2012 Page: 2 of 8

ESC/ISO Number	NELAC Ref.	TNI Standard (Vol 1)	Section & SOP references where applicable	Page	Total Pages	SOP Ref
4.2.6.2	5.4.2.4		ESC Policy Manual	9	30	
4.2.7		M2 4.2.7	Management Of System Changes	9	30	
4.3	5.4.3	M2 4.3	DOCUMENT MANAGEMENT	9	30	
4.3.1			Required Documents (SOP #010103 Document Control and Distribution Procedure)	9	30	Y
4.3.2	5.4.2.3d, 5.4.3.2.1	M2 4.3	Document Control	10	30	
4.3.2.1	5.4.3.2.2 b	M2 4.3.2.1	Document Review and Approval	10	30	
4.3.2.2	5.4.3.2.3	M2 4.3.2.2a	Document Distribution	10	30	
4.3.3	5.4.3.3	M2 4.3.3	Changes to Controlled Documents	11	30	
4.3.3.1	5.4.3.3.1	M2 4.3.3.1	Review and Approval of Changes	11	30	
4.3.3.2	5.4.3.3.2	M2 4.3.3.2	Identification of New or Altered Text	11	30	
4.3.3.3	5.4.3.3.3	M2 4.3.3.3	Procedures for Document Revision	11	30	
4.3.3.4	5.4.3.3.4	M2 4.3.3.4	Changes in Electronic Documents	11	30	
4.3.3.5	5.5.4.1.1 5.5.4.1.1 a, 5.5.4.1.1 b, 5.5.4.1.1 c 5.5.4.5.2 5.5.4.1, 5.5.4.4	M2 4.3.3.5	Standard Operating Procedures	12	30	
4.4	5.4.4 5.4.4.1, 5.4.4.1b, 5.4.4.2, 5.4.4.5, 5.4.2.3i	M2 4.4	REVIEW OF REQUESTS, TENDERS AND CONTRACTS (SOP # 020303, <i>Contract</i> <i>Review</i> )	13	30	Y
4.5	5.4.5.1, 5.4.5.2, 5.4.5.4	M2 4.5	SUBCONTRACTING (SOP #030209, Subcontracting)	14	30	Y
4.6	5.4.6 5.4.6.1, 5.4.6.3, 5.4.6.4	M2 4.6	PURCHASING SERVICES AND SUPPLIES (SOP # 030210 Materials Procurement for Analytical Processes)	15	30	Y
4.7	5.4.7	M2 4.7	SERVICE TO THE CLIENT (SOP 010102, Ethics, Data Integrity, and Confidentiality & SOP 020301, TSR Project Management)	16	30	Y
4.8	5.4.8, 5.4.2.3q	M2 4.8	COMPLAINTS (SOP # 020302, Client Complaint Resolution Procedure)	17	30	Y
4.9	5.4.9, 5.4.9.1a	M2 4.9	CONTROL OF NON-CONFORMING WORK (SOP 030208, Corrective and Preventive Action)	18	30	Y

ESC/ISO Number	NELAC Ref.	TNI Standard (Vol 1)	Section & SOP references where applicable	Page	Total Pages	SOP Ref
4.10		M2 4.10	IMPROVEMENT	19	30	
4.11	5.4.9.1d, 5.4.10, 5.4.10.2, 5.4.10.3, 5.4.10.4, 5.4.10.6a 2	M2 4.11	CORRECTIVE ACTIONS (SOP# 030208 Corrective and Preventive Action)	19	30	Y
4.12	5.4.11	M2 4.12	PREVENTIVE ACTIONS (SOP# 030208 Corrective and Preventive Action)	21	30	Y
4.13	5.4.12 5.4.12.2. 4a- 5.4.12.2. 4f	M2 4.13	CONTROL OF RECORDS (SOP #010103, Document Control and Distribution Procedure)	23	30	Y
4.14	5.4.13		AUDITS (SOP # 010104, Internal Audits)	25	30	Y
4.14.1	5.4.10.4 5.4.13.1 5.4.13.2 5.4.13.3 5.4.13.4 5.4.15	M2 4.14	Internal Audits	25	30	
4.14.2			Performance Audits	27	30	
4.14.3	2.4, 2.4.1, 2.5, 2.7, 2.7.3.1, 2.5.1, 2.7.4, 5.4.10.5	M1 5.0	Proficiency Testing	27	30	
4.14.4			External Audits	28	30	
4.15	5.4.14.1, 5.4.14.2	M2 4.15	MANAGEMENT REVIEW (SOP # 010105, Management Review)	29	30	Y
			Section 5			
5.0	5.5	M2 5.0	Technical requirements	1	74	
5.1 5.2	5.5.1	M2 5.1 M2 5.2	General	1	74 74	
5.2.1	5.5.2 5.4.2.3e, 5.4.2.3f, 5.5.2.4, 5.5.2.5	M2 5.2.1	Personnel General Personnel Management	1	74	
5.2.2	5.4.2.3t, 5.5.2.1	M2 5.2.2	Training (SOP# 030205 Technical Training and Personnel Qualifications)	1	74	Y
5.2.2.1	5.5.2.6c1 , 5.4.12, 2.5.4		Corporate Documents	1	74	
5.2.2.2	5.4.12, 2.5.4		Specific Documents	2	74	
5.2.2.3	5.5.2		Routine Training	2	74	

ESC/ISO Number	NELAC Ref.	TNI Standard (Vol 1)	Section & SOP references where applicable	Page	Total Pages	SOP Ref
5.2.2.4			Special Training	2	74	
5.2.2.5			Annual Training	2	74	
5.2.3	5.4.2.4		General Responsibilities (Description of technical positions)	3	74	
5.2.4	5.4.2.3e	M2 5.2.4	Job Descriptions	5	74	
5.2.5	5.4.12, 2.5.4		Training Records	5	74	
5.3	5.5.3 5.4.2.3i & l	M2 5.3	Accommodation & Facility Design	5	74	
5.3.1	5.5.3.1	M2 5.3.1	Laboratory Facilities	5	74	
5.3.2	5.5.3.2	M2 5.3.2	Environmental Conditions	5	74	
5.3.3	5.5.3.2	M2 5.3.3	Separation of Incompatible Activities	6	74	
5.3.4	5.5.3.2	M2 5.3.4	Facilities Access Management	6	74	
5.3.5	5.5.3.3, 5.5.3.4, 5.5.3.5	M2 5.3.5	Good Housekeeping	7	74	
5.4	5.5.4	M2 5.4	TEST METHODS AND VALIDATION	7	74	
5.4.1	5.5.4.1	M2 5.4.1	General	7	74	
5.4.2	5.5.4.2	M2 5.4.2	Selection of Methods	7	74	
5.4.3	5.5.4.3	M2 5.4.3	Laboratory Developed Methods	8	74	
5.4.4	5.5.4.4	M2 5.4.4	Non-Standard Methods	8	74	
5.4.5	5.5.4.5	M2 5.4.5 M4 1.5	Validation of Methods (SOP #030211, Method Validation)	9	74	Y
5.4.5.1	5.5.4.5.1		Validation description	9	74	
5.4.5.2	5.5.4.5.2	M4 1.5.1	Validation summary	9	74	
5.4.5.3	5.5.4.5.3		Validation for client need	9	74	
5.4.5.4		M4 1.5.2	Limits – MDL, RL, PQL, PDL See SOP 030206, <i>Method Detection Limits</i>	9	74	Y
5.4.5.5	5.5.4.2.2	M4 1.6 M4 1.6.1 M4 1.6.2 M4 1.6.3	Demonstration of Capability – IDOC, CDOC (SOP 030205: <i>Technical Training and</i> <i>Personnel Qualifications</i> )	11	74	
5.4.6	5.5.4.6	M4 1.5.3	Measurement Uncertainty (SOP 030221, Measurement of Uncertainty)	13	74	Y
5.4.7	5.5.4.7	M2 5.4.7	Control of Data	14	74	
5.4.7.1	5.5.4.7.1	M2 5.4.7.1	Transfer checks	14	74	
5.4.7.2	5.5.4.7.2, 5.5.5.12	M2 5.4.7.2	Automated acquisition	15	74	
5.4.7.3	5.5.4.7.2 d	M4 5.4.7.2	Commercial software	15	74	
5.4.7.4	5.5.5.12		ESC Software Systems (LIMS & Auxiliary)	15	74	
5.5	5.5.5	M2 5.5	EQUIPMENT	17	74	
5.5.1		M2 5.5.1	Usability	17	74	
5.5.2		M4 1.7.1	Calibration of Equipment	17	74	
5.5.3	5.5.5.7,		Equipment Operation and Maintenance	18	74	

ESC/ISO Number	NELAC Ref.	TNI Standard (Vol 1)	Section & SOP references where applicable	Page	Total Pages	SOP Ref
	5.5.5.8, 5.5.5.9					
5.5.4	5.5.5.9	M2 5.5.4	Identification of Equipment	21	74	
5.5.5	5.5.5.11	M2 5.5.5	Records of Equipment	21	74	
5.5.6	5.5.5.6	M2 5.5.6	Equipment Handling, Storage, Use, and Maintenance	21	74	
5.5.7	5.5.5.7	M2 5.5.7	Equipment Out of Service	23	74	
5.5.8	5.5.5.8	M2 5.5.8	Status of Calibration	23	74	
5.5.9	5.5.5.9	M2 5.5.9	Equipment Returning to Service	23	74	
5.5.10	5.5.5.10	M2 5.5.10	Calibration Checks	23	74	
5.5.11	5.5.5.11	M2 5.5.11	Calibration Factors	23	74	
5.5.12	5.5.5.12	M2 5.5.12	Safeguarding of Equipment Integrity	23	74	
5.6	5.5.6	M2 5.6	MEASUREMENT TRACEABILITY	23	74	
5.6.1	5.5.6.1, 5.5.6.4		Policy (See SOP 030202 Receipt and Records of Stock, Intermediate, and Working Standards)	24	74	Y
5.6.2	5.5.6.2.2		Measurement Traceability (SOP 030212 PT Program)	24	74	Y
5.6.3	5.5.6.3.1	M4 1.7.1	Calibration/Verification	24	74	
5.6.3.1	5.5.6.3.2	M4 1.7.1.1	Standards (calibration)	24	74	
5.6.3.2	5.5.6.3.2	M4 1.7.2	Standards (verification) (SOP 030207 Quality Control Charting and Tracking)	25	74	Y
5.6.3.3	5.5.6		Measuring and Test Equipment	25	74	
5.6.3.4	5.5.6.4 5.5.6.3.4	M4 1.7.3.5	Standard/Reagent Sources, Records, & Preparation (SOP 030210 Materials Procurement for Analytical Processes)	25	74	Y
5.7	5.5.7	M2 5.7	SAMPLING	26	74	
5.7.1	5.5.7.1	M2 5.7.1	Sampling Plan	26	74	
5.7.2	5.5.7.2	M2 5.7.2	Client Requirements	26	74	
5.7.3	5.5.7.3	M2 5.7.3	Sampling Records	27	74	
5.7.4			Field Sampling - General Summary	27	74	
5.7.5			Field Quality Control Checks	29	74	
5.8	5.5.8	M2 5.8	SAMPLE MANAGEMENT (SOP 030605 Sample Receiving)	31	74	Y
5.8.1	5.5.8.1	M2 5.8.1	Sample Management Instructions (SOP 030220, Sample Homogenization and Sub- Sampling)	31	74	Y
5.8.2	5.5.8.2a, 5.5.7.3	M2 5.8.2	Sample Information and Labeling	35	74	
5.8.3	5.5.8.3 5.5.8.3.1, 5.5.8.3.2, 5.5.8.4	M2 5.8.3 M4 1.7.5	Sample Inspection and Receipt	35	74	
5.8.3.1		M2 5.8.4	Sample Objectives	36	74	

Section TOC, Ver. 10.0 Date: April 15, 2012 Page: 6 of 8

ESC/ISO Number	NELAC Ref.	TNI Standard (Vol 1)	Section & SOP references where applicable	Page	Total Pages	SOP Ref
5.8.3.2	5.5.8.3.1 c	M2 5.8.6	Sample Rejection Criteria	36	74	
5.8.3.3	5.5.8.3.1, 5.5.8.3.2	M2 5.8.7.2	Nonconformance Issues	37	74	
5.8.3.4		M2 5.8.7.3	Login Confirmation	37	74	
5.8.4	5.5.8.4	M2 5.8.9	Sample Storage and Handling	37	74	
5.8.5			Special Requirements	38	74	
5.8.6	5.5.8.1		Sample Transportation	38	74	
5.8.7	5.5.8.3.1	M2 5.8.7.5	Sample Custody	39	74	
5.9	5.5.9	M2 5.9	QUALITY CONTROL	49	74	
5.9.1	5.5.9.1	M2 5.9.3 M4 1.7.3	Quality Control Procedures	49	74	
5.9.2	5.5.9.2		Quality Control Activities	49	74	
5.9.2.1	5.5.9.1		General discussion	49	74	
5.9.2.2	5.5.9.2		Laboratory Checks	50	74	
5.9.2.3	5.5.9.2		Batch QC Criteria	51	74	
5.9.2.4	5.5.9.1b		Inter-Laboratory Quality Control	53	74	
5.9.2.5			Procedures for Assessing Data Precision, Accuracy and Completeness	53	74	
5.9.2.6	5.5.9.2		Use and Preparation of QC Samples	53	74	
5.9.2.7			QC Charts	54	74	
5.9.2.8	5.5.9.2	M4 1.5.3	Accuracy	55	74	
5.9.2.9	5.5.9.2	M4 1.5.3	Precision	57	74	
5.9.2.10	5.5.9.2	M4 1.7.4.2	Marginal Exceedence Limits	57	74	
5.10	5.5.10 5.5.10.4	M2 5.10	FINAL REPORTS / CERTIFICATES	60	74	
5.10.1	5.5.10.1	M2 5.10.1	General	60	74	
5.10.2	5.5.10.2	M2 5.10.2	Test Reports	60	74	
5.10.3	5.5.10.3	M2 5.103	Optional Test Report Items	66	74	
5.10.4		M2 5.10.4	Calibration Certificates	66	74	
5.10.5	5.5.10.3 5.5.10.4	M2 5.10.5	Opinions and Interpretations (SOP 030223 Report Revision)	67	74	
5.10.6	5.5.0.5	M2 5.10.6	Results from Subcontractors	67	74	
5.10.7	5.5.10.6	M2 5.10.7	Electronic Transmission of Results	67	74	
5.10.8	5.5.10.7	M2 5.10.8	Format of Reports	67	74	
5.10.9	5.5.10.8	M2 5.10.9	Amendments to Reports	69	74	
5.11	5.4.12.2. 5.3	M4 1.7.3.4	LABORATORY DATA REDUCTION (SOP 030201 Data Handling & Reporting)	69	74	Y
5.11.1	5.5.4.7		Manual Calculations	60	74	
5.11.1		M2 5.4.7.2		69 69	74	
5.11.2		M2 5.4.7.2 M2 5.4.7.2	Computer Processing Data Acquisition	69 69	74	
5.11.5		11/12 J.4.1.2	Analytical Data Records	70	74	
J.11.4	1	1	Anarytical Data Netorus	70	/4	I I

Section TOC, Ver. 10.0 Date: April 15, 2012 Page: 7 of 8

ESC/ISO Number	NELAC Ref.	TNI Standard (Vol 1)	Section & SOP references where applicable	Page	Total Pages	SOP Ref
5.12.1			Chain of Custody Review	71	74	
5.12.2			Field Data	71	74	
5.12.3	5.5.10.3		Laboratory Analysis, QC, and Data Review	72	74	
			Section 6			
6.0			WASTE MINIMIZATION/DISPOSAL	1	7	
6.1			Soil Samples	1	7	
6.2			Mold/Biohazard Sample Disposal	1	7	
6.3			Reagents, Storage & Waste Disposal	2	7	
6.4			Contamination Control	3	7	
			APPENDICES			
Ι			Site Plan			
II			Certifications			
III			Field Sampling			
IV			Inorganic - Wet Lab			
V			Inorganic – Metals & TCLP Lab			
VI			Volatile Organic Laboratory			
VII			Semi-Volatile Organic Laboratory			
VIII			Air Lab			
IX			Aquatic Toxicity			
Х			Microbiology Lab			
XI			Mold Lab			
XII			Protozoan Lab			

Section TOC, Ver. 10.0 Date: April 15, 2012 Page: 8 of 8

# **Tables and Figures**

Туре	#	Section		Page	Pages
Table	3.3a	3.0	Definitions	1	41
Table	3.3b	3.0	Analytical Capabilities	9	41
Figure	4.1	4.0	Corporate Organizational Chart	5	30
Table	5.4.7.4a	5.0	LIMS	15	74
Table	5.4.7.4b	5.0	Auxiliary Software	16	74
Table	5.5.3.3a	5.0	General Equipment Calibration	19	74
Table	5.5.3.3b	5.0	Class 1 Weight Tolerance	21	74
Table	5.5.6	5.0	General Preventive Maintenance	22	74
Table	5.7.5.2	5.0	Equipment Blank Collection Procedure For Each Type Of Sampling Equipment	30	74
Figure	5.8.7a	5.0	Chain of Custody Process	40	74
Figure	5.8.7b	5.0	Individual Container Log Example	41	74
Figure	5.8.7c	5.0	Chain of Custody	42	74
Figure	5.8.7d	5.0	Sample Container Label	43	74
Figure	5.8.7e	5.0	Sample Container Custody Seal	44	74
Figure	5.8.7f	5.0	Sample Login Label	45	74
Figure	5.8.7g	5.0	Example Lab Preparation Sheet	46	74
Figure	5.8.7h	5.0	Example Lab Assignment/Worksheet	47	74
Figure	5.8.9i	5.0	Example Sample Confirmation Report	48	74
Table	5.9.2.2	5.0	Basic Laboratory QC Checks	50	74
Table	5.9.2.6	5.0	Methods Used to Generate Precision and Accuracy Targets	54	74
Table	5.9.2.10	5.0	Precision and Accuracy Charts	59	74
Figure	5.10.2	5.0	Example Final Client Report	62	74
Table	5.10.2	5.0	Data Qualifier Codes	64	74
Table	5.10.8	5.0	Data Package Contents	68	74
Table	5.12.3	5.0	Data Reduction and Validation Slow	74	74

#### 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 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 17025, NELAC, DOD QSM, and 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:

74

- 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

#### 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 17025, NELAC, DOD QSM. The effectiveness of the quality system is measured by internal and external audits, management review meetings, internal error logs and an active preventive and corrective action system.

#### 2.1.3 Analytical Capabilities:

Where mandated, only approved EPA 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:

	<b>Routine Methodology and Programs</b>
PROGRAM	METHOD SOURCE
NPDES	<i>EPA 821/R-93-010-A</i> <i>Methods for the Determination of Nonconventional Pesticides in Municipal and</i> <i>Industrial Wastewater, Volume I. Revision 1, August 1993.</i>
	<i>EPA 821/R-02-019</i> <i>Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry. August 2002.</i>
	40 CFR part 136
	Methods for Chemical Analysis of Water and Wastes (March 1983)
	Standard Methods for the Examination of Water and Wastewater (18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> editions)
AQUATIC TOXICITY	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).
SDWA	40 CFR parts 141
	Methods for Chemical Analysis of Water and Wastes (March 1983)
	Standard Methods for the Examination of Water and Wastewater (18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> editions)
	Methods for the Determination of Organic Compounds in Drinking Water -EPA/600/4- 88/039 - December 1988 (Revised July 1991)
	Methods for the Determination of Organic Compounds in Drinking Water Supplement I, EPA/600/4-90/020 - July 1990
	Methods for the Determination of Organic Compounds in Drinking Water Supplement II, EPA/600/R-92/129 - August 1992
	EPA. Method 1623: Cryptosporidium and Giardia in Water by Filtration/IMS/FA, December 2005.
RCRA	SW-846, Test Methods for Evaluating Solid Wastes $(3^{rd}, 4^{th} and online editions)$
IH	NIOSH Manual of Analytical Methods (4 <sup>th</sup> edition) & OSHA Sampling and Analytical Methods (online)
AIR	Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air
	Emission Measurement Center (Air Emissions Methods)
	NIOSH Manual of Analytical Methods (4 <sup>th</sup> edition)
	Journal of Chromatographic Science, Vol. 36, May 1998.
CLP	USEPA CONTRACT LABORATORY PROGRAM - STATEMENT OF WORK FOR ORGANICS ANALYSIS Multi-Media, Multi-Concentration OLM04.3
	USEPA CONTRACT LABORATORY PROGRAM - STATEMENT OF WORK FOR INORGANIC ANALYSIS Multi-Media, Multi-Concentration ILM05.3

Routine Methodology and Programs			
PROGRAM	METHOD SOURCE		
MOLD	American Industrial Hygiene Association		
	NIOSH Manual of Analytical Methods (4 <sup>th</sup> edition)		
Miscellaneous	American Society for Testing and Materials (ASTM)		
	State Specific Methodologies from the following: <i>Florida, Oregon, Iowa, Washington, Texas, Arizona, Massachusetts, North Carolina, Louisiana, Missouri, Kansas, Wisconsin, Ohio</i>		
Miscellaneous	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)		

#### 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 and is the only laboratory facility certified/approved to operate 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.

Section 3.0, Ver. 10.0 Date: April 15, 2012 Page: 1 of 74

## 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.

## 3.3 **DEFINITIONS AND TERMINOLOGY**

The quality department is responsible for establishing and maintaining a list of definitions and conventions.

	Table 3.3a Definitions			
TERM	DEFINITION			
Acceptance Criteria (Analytical QC Limits)	Specified limits placed on characteristics of an analytical process as defined in analytical methodology or guidance.			
Accuracy	The amount of agreement between an observed value and an accepted reference value. Accuracy is represented as percent recovery.			
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.			
Batch Analysis	Analysis of $1 - 10$ or 20 samples, depending on the published method requirements, including all required QC. When there are 21 or more samples to be analyzed, the QC criteria for the next 20 samples is the same as it is with a single batch.			
Batch	1 - 10 or 20 samples, depending on the published method requirements. A group of samples that behave similarly and are analyzed as a unit.			
Blank	See FIELD, TRIP, METHOD, EQUIPMENT			
Blind Sample	A sample submitted for analysis with a composition known only to the individual requesting the analysis. The analyst/laboratory may know the identity of the sample, but not its composition. It is used to verify the analyst or laboratory's proficiency in the execution of the analytical measurement process.			

Section 3.0, Ver. 10.0 Date: April 15, 2012 Page: 2 of 74

	Table 3.3a Definitions		
Calibration	To determine, by measurement or comparison with a known standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of an instrument control. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.		
Calibration Curve	The graphic representation of the relationship between the known values, such as concentrations of a series of calibration standards and instrument responses.		
Calibration Factor	The ratio of the detector response (peak areas or peak heights) to the amount (mass) of analyte in the calibration standard. $CF = \frac{A_{s}}{C_{s}}$		
	where: $A_s$ - Average Peak Area over the number of peaks used for quantitation $C_s$ – Concentration of the analyte in the standard.		
Continuing Calibration Blank (CCB)	The CCB is used to confirm the absence of contaminants within the analytical system prior to and during the analysis of field samples. The CCB must be <½ RL, concentrations of common laboratory contaminants cannot exceed the reporting limit. The CCB is analyzed on at regular intervals within a batch and is typically utilized in Metals and Wet Chemistry analyses.		
Continuing Calibration Verification (CCV)	A standard, usually near the mid-point of the calibration curve, made from the primary stock used for the calibration curve. The CCV is used to verify the calibration stability of the instrument and must perform within method stated criteria, which is usually $\pm 10$ to 15%. The CCV must be analyzed at regular intervals within a batch.		
Continuing Demonstration of Capability (CDOC)	Continuing Demonstration of Capability – Annual* verification of analyst skill. *unless required more frequently by program or method		
Chain of Custody	A record that documents the possession of the samples from the time of collection to receipt by the laboratory. This record generally includes: the number and types of containers, the mode of collection; collector ID; time of collection; preservation; and requested analysis.		
Corrective Action	An action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.		
Data Quality Objective (DQO)	A statement of the overall level of uncertainty that a data user is willing to accept in results derived from analytical data.		
Duplicate	Second aliquots of field samples carried through the entire preparation and analytical process that are used as an indication of sample precision or consistency in the field sample matrix.		
Equipment Blank	A sample of analyte free water (usually laboratory DI) which has been used to rinse the sampling equipment. It is collected after decontamination procedures but prior to sampling. The purpose is to demonstrate complete decontamination of the equipment.		

Section 3.0, Ver. 10.0 Date: April 15, 2012 Page: 3 of 74

	Table 3.3a Definitions	
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.	
Field Blank	A sample of analyte free water (usually laboratory DI) is poured into the appropriate collection vessel and preserved according to method guidelines. The purpose of the field blank is to serve as a check on reagent and environmental contamination.	
Initial Calibration Verification (ICV) See also SSCV	An independently prepared standard used to verify the accuracy of the initial calibration (for ongoing calibration). The ICV is used to represent the calibration efficiency of the instrument and must perform within method stated criteria, which is usually $\pm 10$ to 15%. For metals analysis, the ICV is a secondary source.	
Initial Demonstration of Capability (IDOC) See also CDOC	A demonstration of capability (DOC) must be made prior to using any analytical method and any time there is a change in instrument type, personnel or testing method. Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated.	
Instrument Detection	IDL is the smallest signal above background noise that an instrument can	
Limit (IDL) Interference Check Sample (ICS)	<ul> <li>reliably detect.</li> <li>A series of two solutions, used in ICP and ICPMS analysis, to verify that interelement interferences are compensated for correctly. This standard is referred to as the Spectra Interference Check (SIC) in EPA Method 200.7</li> <li>ICSA – A solution containing only the interfering analytes at high concentrations.</li> <li>ICSAB – A solution containing interferents plus other method analytes at the level of concern, which corresponds to the project specific action limits.</li> <li>ICSA and ICSAB provide and adequate test of inter-element correction (IEC) factors.</li> </ul>	
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	Analytes not expected to occur naturally in field samples that are spiked to provide a consistent basis for comparison with target analytes. ISTDs are used in internal calibration models.	

**Section 3.0, Ver. 10.0** Date: April 15, 2012 Page: 4 of 74

	T	able 3.3a Definitions
	A known matrix is spiked with known amounts of the analyte(s) of interest used to verify the efficiency of the analytical system without interference from the sample matrix. The LCS provides the best estimate of analytical system accuracy and may also be used to verify the validity of the on-going calibration. The source is usually a secondary source. The LCS matrix must closely represent the matrix of the sample batch and undergo all preparations required by the method prior to analysis. The following list are acceptable matrices for the LCS:	
Laboratory Control Sample (LCS) - 2 <sup>ND</sup> Source	<u>Batch Matrix</u> Water	<u>LCS Matrix</u> Laboratory DI water
	Soil	Spiked Ottawa sand or Glass beads or commercially prepared LCS in a soil matrix
	Paint Chips	Laboratory prepared paint chip/lead mixture Commercially prepared & certified paint chip LCS
	Filters/Sorbent Media/Dust Wipes	Unused Industrial Hygiene sampling media that represents the substrate submitted by the client. Where possible, the media should be the same lot as that of the field samples.
<i>Limit Of Detection</i> ( <i>LOD</i> )	The lowest concentration that can be determined by a single analysis to be statistically different from a blank, within a defined level of confidence. This concentration is recommended to be three standard deviations above the measured average difference between the sample and blank signals, which corresponds to the 99% confidence level. In practice, detection of an analyte by an instrument is often based on the extent to which the analyte signal exceeds peak-to-peak noise (Keith et al. 1983). Samples that do not bear residues at or above the LOD are referred to as non-detects (ND).	
<i>Limit of Quantitation</i> ( <i>LOQ</i> )	The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ may be equal to the RL, MRL, or PQL. Routinely the PQL/LOQ is at least 3-5 times the statistically derived MDL/LOD.	
Linear Dynamic Range (LDR)	In Inorganic analyses, the LDR is defined as the concentration range where absorbance and concentration remain directly proportional to each other. A wide linear dynamic range permits the analysis of a wide range of sample concentrations (optical densities) and reduces sample preparation (dilution)	
	requirements.	

# ESC Lab Sciences Quality Assurance Manual

Scope and Definitions

	Table 3.3a Definitions
	The component, or substrate, which contains the analyte of interest. For purposes of batch determination, the following matrix types are used:
	• <i>Aqueous:</i> Any aqueous sample excluded from the definition of a drinking water matrix or saline/estuarine source. Includes surface water, groundwater, and effluents.
	<ul> <li><i>Drinking Water:</i> Any aqueous sample that has been designated as a potable or potentially potable water source.</li> <li><i>Saline/Estuarine:</i> Any aqueous sample from an ocean or estuary, or other</li> </ul>
	<ul> <li>saltwater source, such as the Great Salt Lake.</li> <li><i>Non-aqueous Liquid:</i> Any organic liquid with &lt;15% settleable solids.</li> </ul>
Matrix	<ul> <li>Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples are grouped according to origin.</li> <li>Solids: Includes soils, sediments, sludge and other matrices with &gt;15% settleable solids.</li> </ul>
	• <i>Chemical Waste</i> : A product or by-product of an industrial process that results in a matrix not previously defined.
	<ul> <li>Air Samples: Media used to retain the analyte of interest from an air</li> </ul>
	sample such as sorbent tubes or summa canisters. Each medium is
	considered as a distinct matrix.
	• <i>Solids (Other than defined above):</i> Includes filters, dust wipes, sorbent tubes, paint chips.
	A separate aliquot of field sample spiked with a known amount of the target
	analyte. Accuracy is determined by comparing the recovery of the spike
	added to the known concentration in the sample divided by the expected
	analyte concentration.
Matrix Spika (MS)	$PercentSpikeRecovery = \frac{O_i - O_s}{T_i} X 100$
Matrix Spike (MS)	$O_i$ = observed sample concentration with the spike added
	$O_s$ = the observed value for the sample without the spike
	$T_{i} = \frac{Spike \ Concentration \ in \ (mg/L) \ X \ Volume \ of \ Spike \ in \ (ml)}{Volume \ of \ Sample \ in \ (ml) + Volume \ of \ Spike \ in \ (ml \ )}}$
	$T_i$ = True value of the spike added
Matrix SpikeThe second aliquot of the field sample spiked as the matrix spike and through all sample preparation/analytical steps. The MS/MSD pair with identical amounts of the target analyte and precision is calculat on the results.	
	The minimum concentration of a substance that can be analyzed with 99%
Method Detection	confidence that the analyte concentration is greater than zero. MDLs are
Limit (MDL)	performed in conjunction with 40CFR 136, Appendix B. The MDL is the
	absolute minimum level of reporting that is allowed. Values reported between the MDL and RL are flagged with a "J" qualifier.

Section 3.0, Ver. 10.0 Date: April 15, 2012 Page: 6 of 74

	Table 3.3a Definitions	
Method Blank	A laboratory produced blank is carried through each step of the analytical procedure for each batch of samples. Blanks are prepared for each preparation method and matrix (i.e., solids assay, dissolved metals, TCLP extraction, etc.). Blanks are used to confirm the absence of contaminants within the preparation and/or analytical system prior to and during the analysis of field samples.	
Negative Control	Measures taken to ensure that an analytical process, its components, or the environment do not cause adverse effects or lead to incorrect quantitation.	
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. and is calculated as follows: $\% \text{ Recovery} = \left[\frac{\text{Observed Value}}{\text{True Value}}\right] X 100$	
Positive Control	Measures taken to ensure that an analysis and/or its components are working properly and producing correct or expected results.	
Post Digestion Spike	In metals analysis, a standard prepared from a previously analyzed spiked sample digestate that yielded reduced recovery for the target analyte due to a suspected matrix interferent.	
Practical Detection Limit (PDL)	An in-house protocol that is used to determine a practical and real number for method detection. This is not a statistically derived number. It is a verified number that is validated using a 20% coefficient of variation.	
Practical Quantitation Limit (PQL) See also Reporting Limit (RL)	Generally, the lowest standard of the calibration curve. The PQL, or RL, is defined as the lowest level that can be reliably achieved within the established limits of precision and accuracy during routine laboratory operating conditions. The PQL is the default reporting limit (RL) when no other limits are required by the project. The PQL is usually a factor of 3-10 times greater than the determined MDL. The value of the PQL changes with subsequent sample dilutions and final volumes. The multiplier (dilution) of the sample is applied to the PQL for reporting. Values reported between the MDL and PQL are flagged with a "J" qualifier.	
Precision	The agreement between 2 or more duplicate measurements. There is no assumption of the true value of the sample. Precision is expressed as RPD (Relative Percent Difference).	
Proficiency Testing	The action of providing controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results in comparison to peer laboratories and the collective demographics and results summary of all participating laboratories.	
Qualifier	A general explanation associated with deviations from established method criteria for a given analyte. The qualifiers are alpha-numeric designations that are related to specific comments. (i.e. J1 - "Surrogate recovery limits have been exceeded, values are outside of upper control limits.")	
Quality Assurance	A plan for laboratory operation that specifies the measures used to produce data of known precision and bias.	
Quality Control	A set of measures within a sample analysis methodology to assure that the process is operating from a controlled analytical system.	

Section 3.0, Ver. 10.0 Date: April 15, 2012 Page: 7 of 74

	Table 3.3a Definitions	
<b>R</b> eference Material	A material or substance in which one or more properties 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.	
<b>Reference</b> Toxicant	The toxicant used in aquatic toxicity analyses to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the procedure correctly and obtain consistent results.	
Replicate Sample	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.	
Reporting Limit (RL) See also PQL	The RL is equal to the PQL unless project specific limits are supplied/required by the client.	
Relative Percent Difference (RPD)	$RPD = \frac{ Dup \ 1 - Dup \ 2 }{\left[\frac{(Dup \ 1 + Dup \ 2)}{2}\right]} X \ 100$ The comparison of two values based on the mean of the two values. It is always reported as a positive number. The result is an assessment of precision. For sample duplication, the RPD is calculated using the actual analytical results of the field sample. LCS & MS calculations are also based on the actual sample result of spiked samples.	
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. $RF = \frac{(Conc{IStd})(Area_{Analyte})}{(Conc{analyte})(Area_{IStd})}$	
	where: $A_s = \text{Response for analyte to be measured}$ $A_{is} = \text{Response for the internal standard}$ $C_{is} = \text{Conc. of the internal std.in ug/L}$ $C_s = \text{Conc. of the analyte to be measured in ug/L}$ .	
Sample 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.	
Selectivity	The capability of an analytical method or instrument to respond to a target substance or constituent in the presence of non-target substances.	
Sensitivity	The capability of an analytical method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a property of interest.	

Section 3.0, Ver. 10.0 Date: April 15, 2012 Page: 8 of 74

	Table 3.3a Definitions	
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.	
Serial Dilution	A subsequent dilution of a high concentration field sample that should agree within 10% of the original undiluted analysis. In metals analysis, a serial dilution is included in each preparation batch if target analyte concentration is at least fifty times the IDL. This is generally used as a test for matrix interferents or matrix effects.	
Standard Operating Procedure (SOP)	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	A certified reference material produced by the U.S. National Institute of Standards and Technology (NIST) and characterized for absolute content independent of analytical method.	
Standards Addition	The process of spiking a known amount of analyte into an extract/digestate to observe the increase in concentration of the analyte in question. This process can be used to confirm analyte identification or suspected matrix interferences.	
Surrogate	A compound that is similar to the target analytes in chemical composition and behavior and not expected to occur naturally in field samples. Surrogates are spiked by preparation/analytical personnel to assess sample preparation and analytical efficiency in each individual field sample.	
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.	
Trip Blank	A sample of analyte-free media (usually laboratory DI) that is taken from the laboratory to the sampling site and then returned unopened to the laboratory. The trip blank is used to ensure that cross contamination does not occur during shipment/storage and is used mainly for VOC analyses.	

Section 3.0, Ver. 10.0 Date: April 15, 2012 Page: 9 of 74

# Table 3.3bAnalytical 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.

Matrix	Method	Parameter
AE	EPA 3C	Carbon Dioxide
AE	EPA 3C	Methane
AE	EPA 3C	Nitrogen
AE	EPA 3C	Oxygen
AE	EPA 0040	Hazardous organics
AE	EPA TO-15	Acetaldehyde
AE	EPA TO-15	Acetone
AE	EPA TO-15	Acetonitrile
AE	EPA TO-15	Allyl 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
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	Chloromethane
AE	EPA TO-15	Chlorotoluene (2-)
AE	EPA TO-15	Cyclohexane
AE	EPA TO-15	Dibromochloromethane
AE	EPA TO-15	Dibromoethane (1,2-) (EDB)
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	Dichlorodifluoromethane
AE	EPA TO-15	Dichloroethane (1,1-)
AE	EPA TO-15	Dichloroethane (1,2-)
AE	EPA TO-15	Dichloroethene (1,1-)
AE	EPA TO-15	Dichloroethene (cis-1,2-)
AE	EPA TO-15	Dichloroethene (trans-1,2-)
AE	EPA TO-15	Dichloropropane (1,2-)
AE	EPA TO-15	Dichloropropene (cis-1,3-)

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 10 of 74

		D:11 (/ 10)
AE	EPA TO-15	Dichloropropene (trans-1,3-)
AE	EPA TO-15	Dichlorotetrafluoroethane (1,2-)
AE	EPA TO-15	Dioxane (1,4-)
AE	EPA TO-15	Ethanol
AE	EPA TO-15	Ethylbenzene
AE	EPA TO-15	Ethyltoluene (4-)
AE	EPA TO-15	Gasoline range organic
AE	EPA TO-15	Hexachlorobutadiene (1,3-)
AE	EPA TO-15	Hexanone (2-)
AE	EPA TO-15	Heptane (n-)
AE	EPA TO-15	Hexane (n-)
AE	EPA TO-15	Isopropanol
AE	EPA TO-15	Isopropylbenzene
AE	EPA TO-15	Methyl alcohol (Methanol)
AE	EPA TO-15	Methyl ethyl ketone
AE	EPA TO-15	Methyl iodide
AE	EPA TO-15	Methyl isobutyl ketone (MIBK)
AE	EPA TO-15	Methyl methacrylate
AE	EPA TO-15	Methyl tert-butyl ether
AE	EPA TO-15	Methylene chloride (Dichloromethane)
AE	EPA TO-15	Naphthalene
AE	EPA TO-15	Propylene
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	Trimethylpentane (2,2,4-)
AE	EPA TO-15	Tert-butyl alcohol
AE	EPA TO-15	Tetrachloroethane (1,1,2,2-)
AE	EPA TO-15	Tetrachloroethene
AE	EPA TO-15	Tetrahydrofuran
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	Trichlorofluoromethane
		Trichloro $(1,1,2)$ trifluoroethane
AE	EPA TO-15	(1,2,2-)
AE	EPA TO-15	Vinyl acetate
AE	EPA TO-15	Vinyl bromide
AE	EPA TO-15	Vinyl chloride
AE	EPA TO-15	Xylene (m-)
AE	EPA TO-15	Xylene (o-)
AE	EPA TO-15	Xylene (p-)
AE	EPA TO-15	Xylenes (total)
DW	SM 9223B	Total coliform / E. coli

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 11 of 74

DW	Other Hach Company	Total coliform / E. coli
DW	EPA 1622	Cryptosporidium
DW	EPA 1623	Cryptosporidium
DW	EPA 180.1	Turbidity
DW	SM 2130 B	Turbidity
DW	EPA 353.2	Nitrate
DW	SM 4500-NO3 F	Nitrate
DW	EPA 300.0	Nitrate
DW	EPA 300.1	Nitrate
DW	SM 4110 B	Nitrate
DW	EPA 353.2	Nitrite
DW	SM 4500-NO3 F	Nitrite
DW	EPA 300.0	Nitrite
DW	EPA 300.1	Nitrite
DW	SM 4110 B	Nitrite
DW	EPA 300.0	Fluoride
DW	EPA 300.1	Fluoride
DW	SM 4110 B	Fluoride
DW	SM 4500-CN C,G	Cyanide
DW	SM 4500-CN C,E	Cyanide
DW	Other Kelada-01	Cyanide
DW	EPA 335.4	Cyanide
DW	EPA 300.0	Sulfate
DW	SM 4110 B	Sulfate
DW	EPA 200.7	Sodium
DW	SM 2540 C	Total dissolved solids (TDS)
DW	EPA 200.7	Calcium
DW	SM 3500-Ca B (20th ed)	Calcium-hardness
DW	SM 3500-Ca D (18/19th ed)	Calcium-hardness
DW	EPA 200.7	Calcium-hardness
DW	EPA 200.7	Total hardness
DW	SM 3120B/3111B or 2340 B	Total hardness
DW	SM 2340 C	Total hardness
DW	SM 2320 B	Alkalinity
DW	EPA 350.1	Ammonia
DW	SM 4500-NH3 G	Ammonia
DW	EPA 300.0	Bromide
DW	SM 4110 B	Bromide
DW	EPA 300.0	Chloride
DW	EPA 300.1	Chloride
DW	SM 4110 B	Chloride
DW	EPA 300.0	Chlorate
DW	EPA 314.0	Perchlorate
DW	EPA 300.0	Chlorite (monthly)
DW	EPA 300.1	Chlorite (monthly)
DW	SM 2120 B	Color

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 12 of 74

DW	SM 5540 C
DW	SM 2150 B
DW	SM 2510 B
DW	SM 4500-P E
DW	SM 5310C
DW	SM 5310 B
DW	SM 5310 C
DW	SM 5320 B
DW	SM 5910B
DW	SM 4500-Cl G
DW	EPA 150.1
DW	SM 4500-H B
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 200.7
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 245.1
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 200.8
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 200.8
DW	EPA 200.7
DW	EPA 200.8
DW	EPA 507

Foaming agents
Odor
Conductivity
Orthophosphate
Dissolved organic carbon (DOC)
Total organic carbon (TOC)
Total organic carbon (TOC)
Total organic halides (TOX)
UV-absorbing compounds
Chlorine - residual
pH
pH
Aluminum
Antimony
Arsenic
Arsenic
Barium
Barium
Beryllium
Beryllium
Cadmium
Cadmium
Chromium
Chromium
Copper
Copper
Iron
Lead
Magnesium
Manganese
Manganese
Mercury
Nickel
Nickel
Selenium
Silver
Silver
Thallium
Zinc
Zinc
Alachlor
Atrazine
Simazine
Butachlor
Metolachlor
Metribuzin

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 13 of 74

DW	EDA 504 1	Dibromosthere (1.2.) (EDD)
DW	EPA 504.1	Dibromoethane (1,2-) (EDB)
DW	EPA 504.1	Dibromo-3-chloropropane (1,2-)
DW	EPA 504.1	Trichloropropane (1,2,3-)
DW	EPA 515.1	D (2,4-)
DW	EPA 515.1	Dalapon
DW	EPA 515.1	Dinoseb
DW	EPA 515.1	TP $(2,4,5-)$ (Silvex)
DW	EPA 515.1	DB (2,4-)
DW DW	EPA 515.1	Dicamba
DW DW	EPA 515.1	Dichlorprop
DW DW	EPA 515.1 EPA 552.2	T (2,4,5-) Bromochloroacetic acid
DW DW	EPA 552.2 EPA 552.2	Dibromoacetic acid
DW DW	EPA 552.2 EPA 552.2	Dichloroacetic acid
DW DW	EPA 552.2	Monobromoacetic acid (MBAA)
DW DW	EPA 552.2	Monochloroacetic acid (MCAA)
DW DW	EPA 552.2 EPA 552.2	Trichloroacetic acid
DW DW	EPA 552.2 EPA 508	Endrin
DW DW		
DW DW	EPA 508 EPA 508	Heptachlor Hantachlor anouide
DW DW	EPA 508	Heptachlor epoxide Hexachlorobenzene
DW DW	EPA 508	Hexachlorocyclopentadiene
DW DW	EPA 508	Lindane (gamma BHC)
DW DW	EPA 508	Methoxychlor
DW	EPA 508	Chlordane (technical)
DW	EPA 508	Chlordane (alpha)
DW	EPA 508	Chlordane (gamma)
DW	EPA 508	Toxaphene
DW	EPA 508	Aldrin
DW	EPA 508	Alpha BHC
DW	EPA 508	Beta BHC
DW	EPA 508	Delta BHC
DW	EPA 508	DDD (4,4'-)
DW	EPA 508	DDE (4,4'-)
DW	EPA 508	DDT (4,4'-)
DW	EPA 508	Dieldrin
DW	EPA 508	Endosulfan I
DW	EPA 508	Endosulfan II
DW	EPA 508	Endosulfan sulfate
DW	EPA 508	Endrin aldehyde
DW	EPA 508	Endrin ketone
DW	EPA 524.2	Bromoform
DW	EPA 524.2	Chloroform
DW	EPA 524.2	Dibromochloromethane
DW	EPA 524.2	Bromodichloromethane

Date: April 15, 2012 Page: 14 of 74

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-)
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	Bromochloromethane
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-)

Date: April 15, 2012 Page: 15 of 74

DW	EDA 524 2	Iconnervillengene
DW DW	EPA 524.2	Isopropylbenzene
	EPA 524.2	Isopropyltoluene (4-)
DW DW	EPA 524.2	Propylbenzene (n-)
	EPA 524.2	Tetrachloroethane $(1,1,1,2-)$
DW	EPA 524.2	Trichlorobenzene (1,2,3-)
DW	EPA 524.2	Trichlorobenzene (1,3,5-)
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 524.2	Acetone
DW	EPA 524.2	Butanone (2-)
DW	EPA 524.2	Carbon disulfide
DW	EPA 524.2	Dichloro-2-butene (trans-1,4-)
DW	User Defined 524.2	Diisopropyl Ether [DIPE]
DW	EPA 524.2	Hexachloroethane
DW	EPA 524.2	Hexanone (2-)
DW	EPA 524.2	Methyl iodide
DW	EPA 524.2	Pentanone (4-methyl-2-) (MIBK)
DW	EPA 524.2	Tetrahydrofuran
NPW	SM 9222D (Class B only) plus EPA 625/R-92/013 Appendix F	Fecal coliform
NPW	SM 9260D plus EPA 625/R-92/013 Appendix F	Salmonella sp. Bacteria
NPW	SW-846 1010	Ignitability
NPW	SW-846 1010A	Ignitability
NPW	User Defined ASTM D93	Ignitability
NPW	SW-846 9040B	Corrosivity - pH waste, >20% water
NPW	SW-846 9040C	Corrosivity - pH waste, >20% water
NPW	SW-846 1110	Corrosivity toward steel
NPW	SW-846 1110A	Corrosivity toward steel
NPW	SW-846 1311	Volatile organics
NPW	SW-846 1311	Semivolatile organics
NPW	SW-846 1311	Metals
NPW	SW-846 1310A	Metals - organics
NPW	SW-846 1310B	Metals - organics
NPW	SW-846 1312	Metals - organics
NPW	SW-846 1320	Metals - organics
NPW	SW-846 9040B	pH
NPW	SW-846 9040C	pH
NPW	SW-846 3005A	Metals, Total Rec and Dissolved
NPW	SW-846 3010A	Metals, Total
NPW	SW-846 3020A	Metals
NPW	SW-846 3015	Metals
NPW	SW-846 3015A	Metals
NPW	SW-846 6010B	Aluminum
111 11	<b>U</b> 0100 0 <del>1</del> 0 <sup>2</sup> 010	

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 16 of 74

NPW	SW-846 6010C
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 7196A
NPW	SW-846 7199
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010D
NPW	SW-846 6010B
NPW	SW-846 6010D
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010D
NPW	SW-846 6010C
INE VV	D W -040 0010D

Aluminum Antimony Antimony Antimony Antimony Arsenic Arsenic Arsenic Arsenic Barium Barium Barium Barium Beryllium Beryllium Beryllium Beryllium Boron Boron Cadmium Cadmium Cadmium Cadmium Calcium Calcium Chromium Chromium Chromium Chromium Chromium (VI) Chromium (VI) Cobalt Cobalt Copper Copper Copper Copper Iron Iron Lead Lead Lead Lead Lithium Lithium Magnesium

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 17 of 74

NPW	SW-846 6010C
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 7470A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A
NPW	SW-846 6010B
NPW	SW-846 6010C
NPW	SW-846 6020
NPW	SW-846 6020A

Magnesium
Manganese
Manganese
Manganese
Manganese
Mercury - liquid waste
Molybdenum
Molybdenum
Molybdenum
•
Molybdenum
Nickel
Nickel
Nickel
Nickel
Potassium
Potassium
1 0000000000000000000000000000000000000
Selenium
Selenium
Selenium
Selenium
Silver
Silver
Silver
Silver
Sodium
Sodium
Strontium
Strontium
Thallium
Thallium
Thallium
Thallium
Tin
Tin
Tin
Tin
Titanium
Titanium
Vanadium
Vanadium
Vanadium
Vanadium
Zinc
Zinc
Zinc
Zinc

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 18 of 74

NPW SW-846 3510C **NPW** SW-846 3511 NPW SW-846 3520C NPW SW-846 5030B NPW User Defined 5030C NPW SW-846 8011 NPW SW-846 8011 NPW SW-846 8015B NPW SW-846 8015D NPW SW-846 8015B Ethyl alcohol **NPW** Ethyl alcohol SW-846 8015D NPW SW-846 8015B SW-846 8015D NPW User Defined MA-DEP-VPH, WI NPW GRO, NW TPH Gx NPW SW-846 8015B NPW SW-846 8015D User Defined MA-DEP-EPH, TN-NPW EPH, WI DRO, NW TPH Dx User Defined NWTPH-Dx, NPW NWTPH-Gx, NWTPHID NPW Other FL - PRO User Defined TX 1005, TX 1006, NPW CT ETPH, NW TPH ID NPW Other IA - OA-1 NPW Other IA - OA-2 NPW User Defined CA LUFT - diesel NPW Other NJ-OQA-QAM-025, Rev. 7 NPW SW-846 8021B Benzene NPW Ethylbenzene SW-846 8021B NPW Toluene SW-846 8021B NPW SW-846 8021B Xylene (o-) NPW SW-846 8021B Xylene (m-) **NPW** SW-846 8021B Xylene (p-) NPW SW-846 8021B Xylenes (total) NPW SW-846 8021B NPW SW-846 8081A Alachlor NPW Alachlor SW-846 8081B NPW SW-846 8081A Aldrin NPW Aldrin SW-846 8081B NPW SW-846 8081A Alpha BHC NPW SW-846 8081B Alpha BHC NPW SW-846 8081A Beta BHC NPW SW-846 8081B Beta BHC NPW SW-846 8081A Delta BHC

Semivolatile organics Semivolatile organics Semivolatile organics Volatile organics Volatile organics Dibromoethane (1,2-) (EDB) Dibromo-3-chloropropane (1,2-) Methyl alcohol (Methanol) Methyl alcohol (Methanol) Gasoline range organic Gasoline range organic Gasoline range organic Diesel range organic Diesel range organic Diesel range organic **Petroleum Organics** Petroleum Organics **Petroleum Organics** Petroleum Organics Petroleum Organics Petroleum Organics Petroleum Organics Methyl tert-butyl ether

Section 3.0, Ver. 10.0		
Date: April 15, 2012		
Page: 19 of 74		

NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A

Delta BHC Lindane (gamma BHC) Lindane (gamma BHC) Chlordane (technical) Chlordane (technical) Chlordane (alpha) Chlordane (alpha) Chlordane (gamma) Chlordane (gamma) Chloroneb Chloroneb Chlorothalonil Chlorothalonil DDD (4,4'-) DDD (4,4'-) DDE (4,4'-) DDE (4,4'-) DDT (4,4'-) DDT (4,4'-) Dieldrin Dieldrin Endosulfan I Endosulfan I Endosulfan II Endosulfan II Endosulfan sulfate Endosulfan sulfate Endrin Endrin Endrin aldehyde Endrin aldehyde Endrin ketone Endrin ketone Etridiazole Etridiazole Heptachlor Heptachlor Heptachlor epoxide Heptachlor epoxide Hexachlorobenzene Hexachlorobenzene Hexachlorocyclopentadiene Hexachlorocyclopentadiene Methoxychlor Methoxychlor Permethrin

NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8081A
NPW	SW-846 8081B
NPW	SW-846 8082
NPW	SW-846 8082A
NPW	SW-846 8082
NPW	SW-846 8082A
NPW	SW-846 8082
NPW	SW-846 8082A
NPW	SW-846 8082
NPW	SW-846 8082A
NPW	SW-846 8082
NPW	SW-846 8082A
NPW	SW-846 8082
NPW	SW-846 8082A
NPW	SW-846 8082
NPW	SW-846 8082A
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A

Permethrin Propachlor Propachlor Toxaphene Toxaphene Trifluralin Trifluralin PCB 1016 PCB 1016 PCB 1221 PCB 1221 PCB 1232 PCB 1232 PCB 1242 PCB 1242 PCB 1248 PCB 1248 PCB 1254 PCB 1254 PCB 1260 PCB 1260 Azinphos methyl Azinphos methyl Bolstar Bolstar Chloropyrifos Chloropyrifos Coumaphos Coumaphos Demeton (o-) Demeton (o-) Demeton (s-) Demeton (s-) Diazinon Diazinon Dichlorvos Dichlorvos Dimethoate Dimethoate Disulfoton Disulfoton EPN EPN Ethoprop Ethoprop Fensulfothion

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 20 of 74

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 21 of 74

NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8141A
NPW	SW-846 8141B
NPW	SW-846 8151A
NPW	Other J. Chrom. Sci. RSK-175
NPW	Other J. Chrom. Sci. RSK-175
NPW	Other J. Chrom. Sci. RSK-175
NPW	SW-846 8310

Fensulfothion Fenthion Fenthion Malathion Malathion Merphos Merphos Mevinphos Mevinphos Naled Naled Parathion Parathion Parathion methyl Parathion methyl Phorate Phorate Ronnel Ronnel Stirofos Stirofos Sulfotepp Sulfotepp TEPP TEPP Tokuthion [Protothiofos] Tokuthion [Protothiofos] Trichloronate Trichloronate Dalapon Dicamba Dichlorprop Dinoseb D (2,4-) DB (2,4-) T (2,4,5-) TP (2,4,5-) (Silvex) MCPA MCPP Ethane Ethene Methane Acenaphthene Acenaphthylene Anthracene Benzo(a)anthracene

Date: April 15, 2012 Page: 22 of 74

NPW	SW-846 8310
NPW	SW-846 8310
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8330
NPW	SW-846 8330A
NPW	SW-846 8260B
NPW	SW-846 8260B

Benzo(a)pyrene Benzo(b)fluoranthene Benzo(ghi)perylene Benzo(k)fluoranthene Chrysene Dibenzo(a,h)anthracene Fluoranthene Fluorene Indeno(1,2,3-cd)pyrene Naphthalene Phenanthrene Pyrene HMX HMX RDX RDX Trinitrobenzene (1,3,5-) Trinitrobenzene (1,3,5-) Dinitrobenzene (1,3-) Dinitrobenzene (1,3-) PETN PETN Tetryl Tetryl Nitrobenzene Nitrobenzene Trinitrotoluene (2,4,6-) Trinitrotoluene (2,4,6-) Dinitrotoluene (4-amino-2,6-) Dinitrotoluene (4-amino-2,6-) Dinitrotoluene (2-amino-4,6-) Dinitrotoluene (2-amino-4,6-) Dinitrotoluene (2,4-) Dinitrotoluene (2,4-) Dinitrotoluene (2,6-) Dinitrotoluene (2,6-) Nitrotoluene (2-) Nitrotoluene (2-) Nitrotoluene (3-) Nitrotoluene (3-) Nitrotoluene (4-) Nitrotoluene (4-) Nitroglycerine Nitroglycerine Safrole Benzene

Date: April 15, 2012 Page: 23 of 74

NPW	SW-846 8260C
NPW	User Defined LUFT
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	User Defined LUFT
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	User Defined LUFT
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	User Defined LUFT
NPW	SW-846 8260B
T 4T 44	5 TI-0TO 0200D

Benzene Bromobenzene Bromobenzene Butyl benzene (n-) Butyl benzene (n-) Sec-butylbenzene Sec-butylbenzene Tert-butylbenzene Tert-butylbenzene Chlorobenzene Chlorobenzene Chlorotoluene (2-) Chlorotoluene (2-) Chlorotoluene (4-) Chlorotoluene (4-) Dichlorobenzene (1,2-) Dichlorobenzene (1,2-) Dichlorobenzene (1,3-) Dichlorobenzene (1,3-) Dichlorobenzene (1,4-) Dichlorobenzene (1,4-) Ethylbenzene Ethylbenzene Ethylbenzene Isopropylbenzene Isopropylbenzene Propylbenzene (n-) Propylbenzene (n-) Toluene Toluene Toluene Isopropyltoluene (4-) Isopropyltoluene (4-) Trichlorobenzene (1,2,3-) Trichlorobenzene (1,2,3-) Trimethylbenzene (1,2,4-) Trimethylbenzene (1,2,4-) Trimethylbenzene (1,3,5-) Trimethylbenzene (1,3,5-) Trimethylbenzene (1,2,3-) Trimethylbenzene (1,2,3-) Xylenes (total) Xylenes (total) Xylenes (total) Xylene (m-)

Benzene

Date: April 15, 2012 Page: 24 of 74

NPW	SW-846 8260C
NPW	User Defined LUFT
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	User Defined LUFT
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	User Defined LUFT
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C

Xylene (m-) Xylene (m-) Xylene (o-) Xylene (o-) Xylene (o-) Xylene (p-) Xylene (p-) Xylene (p-) tert-Amylmethyl ether [TAME] tert-Amylmethyl ether [TAME] Allyl chloride Allyl chloride Bromochloromethane Bromochloromethane Bromodichloromethane Bromodichloromethane Bromoethane Bromoethane Bromoform Bromoform Bromomethane Bromomethane Cyclohexane Cyclohexane Cyclohexanone Cyclohexanone Butadiene (2-chloro-1,3-) Butadiene (2-chloro-1,3-) Dichloro-2-butene (cis-1,4-) Dichloro-2-butene (cis-1,4-) Carbon tetrachloride Carbon tetrachloride Chloroethane Chloroethane Chloroethyl vinyl ether (2-) Chloroethyl vinyl ether (2-) Chloroform Chloroform Chloromethane Chloromethane Diethyl ether (Ethyl ether) Diethyl ether (Ethyl ether) Dichloropropene (trans-1,3-) Dichloropropene (trans-1,3-) Dibromochloromethane Dibromochloromethane

NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NDW	CW 916 9260D

NPW

NPW

SW-846 8260B

SW-846 8260C

Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 25 of 74

Dibromoethane (1,2-) (EDB) Dibromoethane (1,2-) (EDB) Dibromomethane Dibromomethane Dibromo-3-chloropropane (1,2-) Dibromo-3-chloropropane (1,2-) Dichlorodifluoromethane Dichlorodifluoromethane Dichloroethane (1,1-)Dichloroethane (1,1-)Dichloroethane (1,2-)Dichloroethane (1,2-) Dichloroethene (1,1-)Dichloroethene (1,1-) Dichloroethene (trans-1,2-) Dichloroethene (trans-1,2-) Dichloroethene (cis-1,2-) Dichloroethene (cis-1,2-) Dichloropropane (1,2-) Dichloropropane (1,2-) Dichloropropane (1,3-) Dichloropropane (1,3-) Dichloropropane (2,2-) Dichloropropane (2,2-) Dichloropropene (1,1-) Dichloropropene (1,1-) Dichloropropene (cis-1,3-) Dichloropropene (cis-1,3-) Dichloro-2-butene (trans-1,4-) Dichloro-2-butene (trans-1,4-) Diisopropyl Ether [DIPE] Diisopropyl Ether [DIPE] Butanol (1-) Butanol (1-) Ethanol Ethanol Methylene chloride (Dichloromethane) Methylene chloride (Dichloromethane) Nitropropane (2-) Nitropropane (2-) Tetrachloroethane (1,1,2,2-) Tetrachloroethane (1,1,2,2-)Tetrachloroethene Tetrachloroethene Tetrahydrofuran Tetrahydrofuran

NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NIDITI	0111 046 00 600

NPW

SW-846 8260C

Section 3.0, Ver. 10.0 Date: April 15, 2012

Page: 26 of 74

Trichloroethane (1,1,1-) Trichloroethane (1,1,1-)Trichloroethane (1,1,2-)Trichloroethane (1,1,2-) Trichloroethene Trichloroethene Trichlorofluoromethane Trichlorofluoromethane Trichloro (1,1,2-) trifluoroethane (1,2,2-)Trichloro (1,1,2-) trifluoroethane (1,2,2-)Trichloropropane (1,2,3-) Trichloropropane (1,2,3-)Vinyl acetate Vinyl acetate Vinyl chloride Vinyl chloride Acetone Acetone Carbon disulfide Carbon disulfide Butanol (3,3-Dimethyl-1-) Butanol (3,3-Dimethyl-1-) Butanone (2-) Butanone (2-) Butyl formate (t-) Butyl formate (t-) Ethyl-tert-butyl Ether [ETBE] Ethyl-tert-butyl Ether [ETBE] Ethyl acetate Ethyl acetate Ethyl methacrylate Ethyl methacrylate Hexanone (2-) Hexanone (2-) Methacrylonitrile Methacrylonitrile Methyl acrylate Methyl acrylate Methyl methacrylate Methyl methacrylate Methyl acetate Methyl acetate Methyl iodide Methyl iodide

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 27 of 74

NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	User Defined LUFT
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	User Defined 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B
NPW	SW-846 8260C
NPW	SW-846 8260B

Iso-butyl alcohol Iso-butyl alcohol Isopropanol Isopropanol N-Nitroso-di-n-butylamine N-Nitroso-di-n-butylamine Pentachloroethane Pentachloroethane Pentanone (4-methyl-2-) (MIBK) Pentanone (4-methyl-2-) (MIBK) Pentanol (2-Methyl-2-) Pentanol (2-Methyl-2-) Propionitrile Propionitrile Methyl tert-butyl ether Methyl tert-butyl ether Methyl tert-butyl ether Amyl alcohol (t-) Amyl alcohol (t-) Tert-butyl alcohol Tert-butyl alcohol Acetonitrile Acetonitrile Acrolein Acrolein Acrylonitrile Acrylonitrile Hexane (n-) Hexane (n-) Hexachlorobutadiene (1,3-) Hexachlorobutadiene (1,3-) Hexachloroethane Hexachloroethane Methylcyclohexane Methylcyclohexane Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Methylnaphthalene (2-) Naphthalene Naphthalene Octane (-n) Octane (-n) Styrene Styrene Tetrachloroethane (1,1,1,2-)

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 28 of 74

NPW	SW-846 8260C	Tetrachloroethane (1,1,1,2-)
NPW	SW-846 8260B	Trichlorobenzene (1,2,4-)
NPW	SW-846 8260C	Trichlorobenzene (1,2,4-)
NPW	SW-846 8260B	Trimethylpentane (2,2,4-)
NPW	SW-846 8260C	Trimethylpentane (2,2,4-)
NPW	SW-846 8260B	Dioxane (1,4-)
NPW	SW-846 8260C	Dioxane (1,4-)
NPW	User Defined SW846 8260B & 8260C	Gasoline range organic
NPW	SW-846 8270C	Acetophenone
NPW	SW-846 8270D	Acetophenone
NPW	SW-846 8270C	Acetylaminofluorene (2-)
NPW	SW-846 8270D	Acetylaminofluorene (2-)
NPW	SW-846 8270C	Aminobiphenyl (4-)
NPW	SW-846 8270D	Aminobiphenyl (4-)
NPW	SW-846 8270C	Aramite
NPW	SW-846 8270D	Aramite
NPW	SW-846 8270C	Benzal chloride
NPW	SW-846 8270D	Benzal chloride
NPW	SW-846 8270C	Benzo(j)fluoranthene
NPW	SW-846 8270D	Benzo(j)fluoranthene
NPW	SW-846 8270C	Benzotrichloride
NPW	SW-846 8270D	Benzotrichloride
NPW	SW-846 8270C	Benzyl chloride
NPW	SW-846 8270D	Benzyl chloride
NPW	SW-846 8270C	Biphenyl (1,1'-)
NPW	SW-846 8270D	Biphenyl (1,1'-)
NPW	SW-846 8270C	Chlorobenzilate
NPW	SW-846 8270D	Chlorobenzilate
NPW	SW-846 8270C	Chloronaphthalene (1-)
NPW	SW-846 8270D	Chloronaphthalene (1-)
NPW	SW-846 8270C	Diallate (cis)
NPW	SW-846 8270D	Diallate (cis)
NPW	SW-846 8270D	Diallate (trans)
NPW	SW-846 8270D	Diallate (trans)
NPW	SW-846 8270D	Dibenzo(a,e)pyrene
NPW	SW-846 8270C SW-846 8270D	Dibenzo(a,e)pyrene
NPW	SW-846 8270D	Dibenz(a,h)acridine
NPW	SW-846 8270C SW-846 8270D	
		Dibenz(a,h)acridine
NPW	SW-846 8270C	Dibenzo(a,h)pyrene
NPW	SW-846 8270D	Dibenzo(a,h)pyrene
NPW	SW-846 8270C	Dibenz(a,j)acridine
NPW	SW-846 8270D	Dibenz(a,j)acridine
NPW	SW-846 8270C	Dibenzo(a,i)pyrene
NPW	SW-846 8270D	Dibenzo(a,i)pyrene
NPW	SW-846 8270C	Dibenzo(c,g)carbazole (7H-)

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 29 of 74

NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
111 11	

Dibenzo(c,g)carbazole (7H-)
Dichlorophenol (2,6-)
Dichlorophenol (2,6-)
Dimethoate
Dimethoate
Dimethylaminoazobenzene
Dimethylaminoazobenzene
Dimethylbenz(a)anthracene (7,12-)
Dimethylbenz(a)anthracene (7,12-)
Dimethyl benzidine (3,3-)
Dimethyl benzidine (3,3-)
Dinitrobenzene (1,3-)
Dinitrobenzene (1,3-)
Dinoseb
Dinoseb
Disulfoton
Disulfoton
Famphur
Famphur
Hexachlorophene
Hexachlorophene
Hexachloropropene
Isodrin
Isodrin
Isosafrole (cis-)
Isosafrole (cis-)
Isosafrole (trans-)
Isosafrole (trans-)
Kepone
Kepone
Methanesulfonate (Ethyl-)
Methanesulfonate (Ethyl-)
Methanesulfonate (Methyl-)
Methanesulfonate (Methyl-)
Methapyrilene
Methapyrilene
Methylcholanthrene (3-)
Methylcholanthrene (3-)
Napthoquinone (1,4-)
Napthoquinone (1,4-)
Napththylamine (1-)
Napththylamine (1-)
Napththylamine (2-)
Napththylamine (2-)
Nitrodiphenylamine (2-)
Nitrodiphenylamine (2-)

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 30 of 74

NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D

N-Nitroso-di-n-butylamine
N-Nitroso-di-n-butylamine
N-Nitrosomorpholine
N-Nitrosomorpholine
N-Nitrosopiperidine
N-Nitrosopiperidine
Parathion
Parathion
Parathion methyl
Parathion methyl
Pentachlorobenzene
Pentachlorobenzene
Pentachloroethane
Pentachloroethane
Pentachloronitrobenzene
Pentachloronitrobenzene
Phenacetin
Phenacetin
Phenylenediamine (1,4-)
Phenylenediamine (1,4-)
Phenylethylamine (alpha, alpha-
Dimethyl)
Phenylethylamine (alpha, alpha-
Dimethyl)
Phorate
Phorate
Phosphorothioate (O,O,O-triethyl)
Phosphorothioate (O,O,O-triethyl)
Phosphorothioate (O,O-diethyl-O-2-
pyrazinyl) [Thionazin]
Phosphorothioate (O,O-diethyl-O-2-
pyrazinyl) [Thionazin]
Picoline (2-)
Picoline (2-)
Pronamide
Pronamide
Quinoline -1-Oxide (4-Nitro)
Quinoline -1-Oxide (4-Nitro)
Safrole
Safrole
Sulfotepp
Sulfotepp
Tetrachlorobenzene (1,2,3,4-)
Tetrachlorobenzene (1,2,3,4-)
Tetrachlorobenzene (1,2,3,5-)
Tetrachlorobenzene (1,2,3,5-)

NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D

### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 31 of 74

Tetrachlorobenzene (1,2,4,5-) Tetrachlorobenzene (1,2,4,5-)Tetrachlorophenol (2,3,4,6-) Tetrachlorophenol (2,3,4,6-) Toluidine (2-) (2-Methylaniline) Toluidine (2-) (2-Methylaniline) Toluidine (5-nitro-2-) Toluidine (5-nitro-2-) Trinitrobenzene (1,3,5-) Trinitrobenzene (1,3,5-) N-Nitrosodiethylamine N-Nitrosodiethylamine N-Nitrosodimethylamine N-Nitrosodimethylamine N-Nitroso-di-n-propylamine N-Nitroso-di-n-propylamine N-Nitrosodiphenylamine N-Nitrosodiphenylamine N-Nitrosomethylethylamine N-Nitrosomethylethylamine N-Nitrosopyrrolidine N-Nitrosopyrrolidine Diphenylamine Diphenylamine Carbazole Carbazole Benzidine Benzidine Dichlorobenzidine (3,3'-) Dichlorobenzidine (3,3'-) Diphenylhydrazine (1,2-) Diphenylhydrazine (1,2-) Aniline Aniline Chloraniline (4-) Chloraniline (4-) Nitroaniline (2-) Nitroaniline (2-) Nitroaniline (3-) Nitroaniline (3-) Nitroaniline (4-) Nitroaniline (4-) Chloronaphthalene (2-) Chloronaphthalene (2-) Hexachlorobenzene Hexachlorobenzene

NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 32 of 74

Hexachlorobutadiene (1,3-) Hexachlorobutadiene (1,3-) Hexachlorocyclopentadiene Hexachlorocyclopentadiene Hexachloroethane Hexachloroethane Hexachloropropene Hexachloropropene Trichlorobenzene (1,2,4-) Trichlorobenzene (1,2,4-) Bis (2-chloroethoxy) methane Bis (2-chloroethoxy) methane Bis (2-chloroethyl) ether Bis (2-chloroethyl) ether Bis (2-chloroisopropyl) ether Bis (2-chloroisopropyl) ether Chlorophenyl-phenyl ether (4-) Chlorophenyl-phenyl ether (4-) Bromophenyl-phenyl ether (4-) Bromophenyl-phenyl ether (4-) Dinitrotoluene (2,4-) Dinitrotoluene (2,4-) Dinitrotoluene (2,6-) Dinitrotoluene (2,6-) Isophorone Isophorone Nitrobenzene Nitrobenzene Butyl benzyl phthalate Butyl benzyl phthalate Bis (2-ethylhexyl) phthalate Bis (2-ethylhexyl) phthalate Diethyl phthalate Diethyl phthalate Dimethyl phthalate Dimethyl phthalate Di-n-butyl phthalate Di-n-butyl phthalate Di-n-octyl phthalate Di-n-octyl phthalate Acenaphthene Acenaphthene Anthracene Anthracene Acenaphthylene Acenaphthylene

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 33 of 74

NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NP W NPW	SW-846 8270C SW-846 8270D
NPW	SW-846 8270D SW-846 8270C
	SW-846 8270C SW-846 8270D
NPW	
NPW	SW-846 8270C
NPW	SW-846 8270D

Benzo(a)anthracene Benzo(a)pyrene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(ghi)perylene Benzo(ghi)perylene Benzo(k)fluoranthene Benzo(k)fluoranthene Chrysene Chrysene Dibenzo(a,h)anthracene Dibenzo(a,h)anthracene Fluoranthene Fluoranthene Fluorene Fluorene Indeno(1,2,3-cd)pyrene Indeno(1,2,3-cd)pyrene Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Methylnaphthalene (2-) Naphthalene Naphthalene Phenanthrene Phenanthrene Pyrene Pyrene Methyl phenol (4-chloro-3-) Methyl phenol (4-chloro-3-) Chlorophenol (2-) Chlorophenol (2-) Dichlorophenol (2,4-) Dichlorophenol (2,4-) Dimethylphenol (2,4-) Dimethylphenol (2,4-) Dinitrophenol (2,4-) Dinitrophenol (2,4-) Dinitrophenol (2-methyl-4,6-) Dinitrophenol (2-methyl-4,6-) Methylphenol (2-) Methylphenol (2-) Methylphenol (4-) Methylphenol (4-)

Benzo(a)anthracene

Date: April 15, 2012 Page: 34 of 74

NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	User Defined CA LUFT - diesel
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C

Nitrophonol (2)
Nitrophenol (2-)
Nitrophenol (2-)
Nitrophenol (4-)
Nitrophenol (4-)
Pentachlorophenol
Pentachlorophenol
Phenol
Phenol
Trichlorophenol (2,4,5-)
Trichlorophenol (2,4,5-)
Trichlorophenol (2,4,6-)
Trichlorophenol (2,4,6-)
Dibenzofuran
Dibenzofuran
Dichlorobenzene (1,2-)
Dichlorobenzene (1,2-)
Dichlorobenzene (1,3-)
Dichlorobenzene (1,3-)
Dichlorobenzene (1,4-)
Dichlorobenzene (1,4-)
Benzaldehyde
Benzaldehyde
Benzoic acid
Benzoic acid
Benzyl alcohol
Benzyl alcohol
Decane (n-)
Decane (n-)
Octadecane (n-)
Octadecane (n-)
Petroleum Organics
Pyridine
Pyridine
•
Caprolactam
Caprolactam
Atrazine
Atrazine
Acenaphthene
Acenaphthene
Acenaphthylene
Acenaphthylene
Anthracene
Anthracene
Benzo(a)anthracene
Benzo(a)anthracene
Benzo(a)pyrene
· · · · · ·

Section	3.0.	Ver.	10.0
Section	0.0,		10.0

Date: April 15, 2012 Page: 35 of 74

NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 8270C
NPW	SW-846 8270D
NPW	SW-846 9010C
NPW	User Defined 9010B
NPW	SW-846 9010C
NPW NPW	User Defined 9010B SW-846 9012B
NPW	User Defined 9012A
NPW	SW-846 9020B
NPW	SW-846 9020B
NPW	SW-846 9030
NPW	SW-846 9056
NPW	SW-846 9056A
NPW	SW-846 9040C
NPW	SW-846 9050A
NPW	SW-846 9060
NPW	SW-846 9060A
NPW	ASTM F1647-02A
NPW	Other Walkley Black
NPW	Other USDA-LOI (Loss on ignition)
NPW	SW-846 9066

Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(k)fluoranthene Benzo(ghi)perylene Benzo(ghi)perylene Chrysene Chrysene Dibenzo(a,h)anthracene Dibenzo(a,h)anthracene Indeno(1,2,3-cd)pyrene Indeno(1,2,3-cd)pyrene Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Methylnaphthalene (2-) Naphthalene Naphthalene Fluoranthene Fluoranthene Fluorene Fluorene Phenanthrene Phenanthrene Pyrene Pyrene Cyanide Cyanide Cyanide - amenable to Cl2 Cyanide - amenable to Cl2 Cyanide Cyanide Total organic halides (TOX) Sulfides, acid sol. & insol. Sulfides, acid sol. & insol. Sulfate Sulfate pH - waste, >20% water Specific conductance Total organic carbon (TOC) Phenols

Benzo(a)pyrene

# Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 36 of 74

NPW	SW-846 9056	Nitrite
NPW	SW-846 9056A	Nitrite
NPW	SW-846 9056	Nitrate
NPW	SW-846 9056A	Nitrate
NPW	SW-846 9056	Bromide
NPW	SW-846 9056A	Bromide
NPW	SW-846 9056	Chloride
NPW	SW-846 9056A	Chloride
NPW	SW-846 9056	Fluoride
NPW	SW-846 9056A	Fluoride
NPW		Perchlorate
NPW	EPA 300.0	Guanidine nitrate
NPW	User Defined SW-846 8330	Nitroguanidine
NPW	SW-846 8330	Guanidine nitrate
NPW	SW-846 8260B	Trimethylpentane (2,2,4-)
NPW	SW-846 8270C	Nitrodiphenylamine (2-)
NPW	User Defined EPA 353.2 Modified	Nitrocellulose
NPW	SM 9222 D	Fecal coliform
NPW	SM 9222 B	Total coliform
NPW	ASTM D6503	Enterococci
NPW	SM 9215 B	Heterotrophic plate count
NPW	ASTM D1067	Acidity as CaCO3
NPW	SM 2310 B(4A)	Acidity as CaCO3
NPW	SM 2320 B	Alkalinity as CaCO3
NPW	EPA 310.2	Alkalinity as CaCO3
NPW	EPA 350.1	Ammonia
	SM 4500-NH3 B plus G (19/20th	
NPW	ed.)	Ammonia
NPW	SM 5210 B	Biochemical oxygen demand
NPW	EPA 200.7	Boron
NPW	EPA 300.0	Bromide
NPW	EPA 300.1	Bromide
NPW	EPA 200.7	Calcium
NPW	SM 5210 B	Carbonaceous BOD (CBOD)
NPW	EPA 410.4	Chemical oxygen demand
NPW	SM 5220 D	Chemical oxygen demand
NPW	EPA 300.0	Chloride
NPW	EPA 300.1	Chloride
NPW	SM 4110 B	Chloride
NPW	SM 2120 B	Color
NPW	SM 4500-CN C, E	Cyanide
NPW	Other Kelada-01	Cyanide
NPW	EPA 335.4	Cyanide
NPW	SM 4500-CN C,G	Cyanide - amenable to Cl2
NPW	Other Kelada-01	Cyanide - amenable to Cl2
NPW	EPA 300.0	Fluoride

Page: 37 of 74

NPW	EPA 300.1	Fluoride
NPW	SM 4110 B	Fluoride
NPW	EPA 130.1	Hardness - total as CaCO3
NPW	SM 2340 B or C	Hardness - total as CaCO3
NPW	EPA 200.7	Hardness - total as CaCO3
NPW	SM 4500-N Org B or C plus NH3 B plus NH3 C (19/20th ed)	Kjeldahl nitrogen - total
NPW	EPA 351.2	Kjeldahl nitrogen - total
NPW	EPA 200.7	Magnesium
NPW	EPA 300.0	Nitrate
NPW	EPA 300.1	Nitrate
NPW	SM 4110 B	Nitrate
NPW	EPA 353.2	Nitrate - nitrite
NPW	SM 4500-NO3 F	Nitrate - nitrite
NPW	EPA 300.0	Nitrate - nitrite
NPW	EPA 300.1	Nitrate - nitrite
NPW	SM 4110 B	Nitrate - nitrite
NPW	EPA 300.0	Nitrite
NPW	EPA 300.1	Nitrite
NPW	SM 4110 B	Nitrite
NPW	SM 5520 B	Oil & grease - total recov
NPW		Oil & grease - hem-LL
NPW	SM 5520 B	Oil & grease - hem-LL
NPW	EPA 1664A	Oil & grease - hem-SPE
NPW	EPA 1664A	Oil & grease - sgt-non polar
NPW	EPA 1664A	Oil & grease - non polar
NPW	SM 5310 B, C or D	Total organic carbon (TOC)
NPW	SM 5310B, C or D	Dissolved organic carbon (DOC)
NPW	SM 5320 B	Total organic halides (TOX)
NPW	EPA 351.1, .2 - 350.1	Organic nitrogen
NPW	SM 4500-NH3 B, C, D, E, F, G, H	Organic nitrogen
NPW	SM 4500-P, E	Orthophosphate
NPW	EPA 420.1 plus .4	Phenols
NPW	SM 4500-P B5 plus E	Phosphorus (total)
NPW	EPA 200.7	Potassium
NPW	SM 2540 B	Residue - total
NPW	SM 2540 C	Residue - filterable (TDS)
NPW	SM 2540 D	Residue - nonfilterable (TSS)
NPW	SM 2540 F	Residue - settleable
NPW	EPA 160.4	Residue - volatile
		Total, fixed, and volatile solids
NPW	SM 2540 G SM 18th Ed.	(SQAR)
NPW	EPA 200.7	Silica - dissolved
NPW	EPA 200.7	Sodium
NPW	EPA 120.1	Specific conductance
NPW	SM 2510 B	Specific conductance

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 38 of 74

NPW	EPA 300.0
NPW	EPA 300.1
NPW	SM 4110 B
	SM 4500-S D
NPW	SM 5540 C
NPW	EPA 180.1
NPW	SM 2130 B
	SM 4500-Cl G
NPW	SM 4500-Cl G
NPW	SM 4500-O C
NPW	SM 4500-O G
NPW	SM 4500-H B
NPW	SM 4500-SO3 B
NPW	SM 2550 B
NPW	EPA 200.7
NPW	EPA 200.7
NPW	EPA 200.8
NPW	EPA 200.7
NPW	EPA 200.8
NPW	EPA 200.7
NPW	EPA 200.8
NPW	EPA 200.7
NPW	EPA 200.8
NPW	EPA 200.7
NPW	EPA 200.8
NPW	SM 3500-Cr B (20th ed)
NPW	SM 3500-Cr D (18/19th ed)
NPW	EPA 218.6
NPW	SM 3500-Cr C (20th ed)
NPW	EPA 200.7
NPW	
NPW	EPA 200.7
NPW	
NPW	EPA 200.8
NPW	EPA 200.7
NPW	SM 3500-Fe B (20th ed)
NPW	EPA 200.7
NPW	EPA 200.8
NPW	EPA 200.7
NPW	EPA 200.8
NPW	EPA 245.1
NPW	EPA 1631E
NPW	EPA 200.7
NPW	EPA 200.8
NPW	EPA 200.7
NPW	EPA 200.8

Sulfate Sulfate Sulfate Sulfides Surfactants Turbidity Turbidity Chlorine Chlorine Oxygen (dissolved) Oxygen (dissolved) pН Sulfite - SO3 Temperature Aluminum Antimony Antimony Arsenic Arsenic Barium Barium Beryllium Beryllium Cadmium Cadmium Chromium (VI) Chromium (VI) Chromium (VI) Chromium (VI) Chromium Chromium Cobalt Copper Copper Iron Iron, Ferrous Lead Lead Manganese Manganese Mercury Mercury Molybdenum Molybdenum Nickel Nickel

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 39 of 74

NPW	EPA 200.7	Selenium
NPW	EPA 200.8	Selenium
NPW	EPA 200.7	Silver
NPW	EPA 200.8	Silver
NPW	EPA 200.7	Thallium
NPW	EPA 200.8	Thallium
NPW	EPA 200.7	Tin
NPW	EPA 200.8	Tin
NPW	EPA 200.7	Titanium
NPW	EPA 200.7	Vanadium
NPW	EPA 200.8	Vanadium
NPW	EPA 200.7	Zinc
NPW	EPA 200.8	Zinc
NPW	EPA 602	Benzene
NPW	User Defined SM 6200C 20th ED	Benzene
NPW	EPA 602	Ethylbenzene
NPW	User Defined SM 6200C 20th ED	Ethylbenzene
NPW	EPA 602	Methyl tert-butyl ether
NPW	User Defined SM 6200C 20th ED	Methyl tert-butyl ether
NPW	EPA 602	Tert-butyl alcohol
NPW	User Defined SM 6200C 20th ED	Tert-butyl alcohol
NPW	EPA 602	Toluene
NPW	User Defined SM 6200C 20th ED	Toluene
NPW	EPA 602	Xylenes (total)
NPW	User Defined SM 6200C 20th ED	Xylenes (total)
NPW	EPA 608	Aldrin
NPW	SM 6630 B	Aldrin
NPW	SM 6630 C	Aldrin
NPW	EPA 608	Alpha BHC
NPW	SM 6630 B	Alpha BHC
NPW	SM 6630 C	Alpha BHC
NPW	EPA 608	Beta BHC
NPW	SM 6630 C	Beta BHC
NPW	EPA 608	Delta BHC
NPW	SM 6630 C	Delta BHC
NPW	EPA 608	Lindane (gamma BHC)
NPW	SM 6630 B	Lindane (gamma BHC)
NPW	SM 6630 C	Lindane (gamma BHC)
NPW	EPA 608	Chlordane
NPW	SM 6630 B	Chlordane
NPW	SM 6630 C	Chlordane
NPW	EPA 608	Chlordane (alpha)
NPW	User Defined SM 6630C	Chlordane (alpha)
NPW	EPA 608	Chlordane (gamma)
NPW	User Defined SM 6630C	Chlordane (gamma)
NPW	EPA 608	Chloroneb

Section 3	3.0,	Ver.	10.0
-----------	------	------	------

Date: April 15, 2012 Page: 40 of 74

NPW	
NPW	
NPW	
NPW	
NPW	EPA 608
NPW	
NPW	
NPW	
	SM 6630 B
	SM 6630 C
	EPA 608
NPW	
	SM 6630 C
NPW	
	SM 6630 B
	SM 6630 C
NPW	
	SM 6630 B
NPW	
NPW	EPA 608
NPW	User Defined SM 6630C
NPW	
NPW	
NPW	
NPW	
	SM 6630 C
NPW	
NPW	SM 6630 B
NPW	SM 6630 C
NPW	EPA 608
NPW	User Defined SM 6630C
NPW	EPA 608
NPW	SM 6630 B
NPW	SM 6630 C
NPW	EPA 608
NPW	SM 6630 B
NPW	SM 6630 C
NPW	EPA 1658
NPW	EPA 608
NPW	EPA 608
NPW	EPA 608

Chlorothalonil DDD (4,4'-) DDD (4,4'-) DDD (4,4'-) DDE (4,4'-) DDE (4,4'-) DDE (4,4'-) DDT (4,4'-) DDT (4,4'-) DDT (4,4'-) Dieldrin Dieldrin Dieldrin Endosulfan I Endosulfan I Endosulfan I Endosulfan II Endosulfan II Endosulfan II Endosulfan sulfate Endosulfan sulfate Endrin Endrin Endrin Endrin aldehyde Endrin aldehyde Endrin ketone Endrin ketone Heptachlor Heptachlor Heptachlor Heptachlor epoxide Heptachlor epoxide Heptachlor epoxide Hexachlorobenzene Hexachlorobenzene Methoxychlor Methoxychlor Methoxychlor Toxaphene Toxaphene Toxaphene D (2,4-) PCB 1016 PCB 1221 PCB 1232

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 41 of 74

NPW	EPA 608	PCB 1242
NPW	EPA 608	PCB 1248
NPW	EPA 608	PCB 1254
NPW	EPA 608	PCB 1260
NPW	EPA 610	Acenaphthene
NPW	SM 6440 B	Acenaphthene
NPW	EPA 610	Acenaphthylene
NPW	SM 6440 B	Acenaphthylene
NPW	EPA 610	Anthracene
NPW	SM 6440 B	Anthracene
NPW	EPA 610	Benzo(a)anthracene
NPW	SM 6440 B	Benzo(a)anthracene
NPW	EPA 610	Benzo(a)pyrene
NPW	SM 6440 B	Benzo(a)pyrene
NPW	EPA 610	Benzo(b)fluoranthene
NPW	SM 6440 B	Benzo(b)fluoranthene
NPW	EPA 610	Benzo(ghi)perylene
NPW	SM 6440 B	Benzo(ghi)perylene
NPW	EPA 610	Benzo(k)fluoranthene
NPW	SM 6440 B	Benzo(k)fluoranthene
NPW	EPA 610	Chrysene
NPW	SM 6440 B	Chrysene
NPW	EPA 610	Dibenzo(a,h)anthracene
NPW	SM 6440 B	Dibenzo(a,h)anthracene
NPW	EPA 610	Fluoranthene
NPW	SM 6440 B	Fluoranthene
NPW	EPA 610	Fluorene
NPW	SM 6440 B	Fluorene
NPW	EPA 610	Indeno(1,2,3-cd)pyrene
NPW	SM 6440 B	Indeno(1,2,3-cd)pyrene
NPW	EPA 610	Naphthalene
NPW	SM 6440 B	Naphthalene
NPW	EPA 610	Phenanthrene
NPW	SM 6440 B	Phenanthrene
NPW	EPA 610	Pyrene
NPW	SM 6440 B	Pyrene
NPW	Other NJ-OQA-QAM-025, Rev. 5	Petroleum Organics
NPW	Other NJ-OQA-QAM-025, Rev. 7	Petroleum Organics
NPW	EPA 624	Allyl chloride
NPW	SM 6200 B (20th ed)	Allyl chloride
NPW	EPA 624	Amyl alcohol (n-)
NPW	EPA 624	Acetone
NPW	User Defined SM 6200 B	Acetone
NPW	EPA 624	Acrolein
NPW	SM 6200 B (20th ed)	Acrolein
NPW	EPA 624	Acrylonitrile

NPW SM 6200 B (20th ed) **NPW** EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed)

NPW

NPW

NPW

NPW

NPW

EPA 624

EPA 624

EPA 624

SM 6200 B (20th ed)

User Defined SM 6200 B

#### Acrylonitrile Benzene Benzene Bromobenzene Bromobenzene Bromochloromethane Bromochloromethane Bromodichloromethane Bromodichloromethane Bromoethane Bromoethane Bromoform Bromoform Bromomethane Bromomethane Butanone (2-) Butanone (2-) Butadiene (2-chloro-1,3-) Butadiene (2-chloro-1,3-) Butyl benzene (n-) Butyl benzene (n-) Carbon disulfide Carbon disulfide Carbon tetrachloride Carbon tetrachloride Chlorobenzene Chlorobenzene Chloroethane Chloroethane Chloroethyl vinyl ether (2-) Chloroethyl vinyl ether (2-) Chloroform Chloroform Chloromethane Chloromethane Chlorotoluene (2-) Chlorotoluene (2-) Chlorotoluene (4-) Chlorotoluene (4-) Cyclohexanone Cyclohexanone Dibromo-3-chloropropane (1,2-) Dibromo-3-chloropropane (1,2-) Dibromochloromethane Dibromochloromethane

Dibromoethane (1,2-) (EDB)

# Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 42 of 74

NPW User Defined SM 6200 B NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 SM 6200 B (20th ed) NPW NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 User Defined SM 6200 B NPW NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624

### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 43 of 74

Dibromoethane (1,2-) (EDB) Dibromomethane Dibromomethane Dichloro-2-butene (cis-1,4-) Dichloro-2-butene (cis-1,4-) Dichlorobenzene (1,2-) Dichlorobenzene (1,2-) Dichlorobenzene (1,3-) Dichlorobenzene (1,3-) Dichlorobenzene (1,4-) Dichlorobenzene (1,4-) Dichloro-2-butene (trans-1,4-) Dichloro-2-butene (trans-1,4-) Dichlorodifluoromethane Dichlorodifluoromethane Dichloroethane (1,1-) Dichloroethane (1,1-)Dichloroethane (1,2-)Dichloroethane (1,2-)Dichloroethene (1,1-)Dichloroethene (1,1-)Dichloroethene (cis-1,2-) Dichloroethene (cis-1,2-) Dichloroethene (trans-1,2-) Dichloroethene (trans-1,2-) Dichloropropane (1,2-) Dichloropropane (1,2-) Dichloropropane (1,3-) Dichloropropane (1,3-) Dichloropropane (2,2-) Dichloropropane (2,2-) Dichloropropene (1,1-) Dichloropropene (1,1-) Diethyl ether (Ethyl ether) Diethyl ether (Ethyl ether) Dichloropropene (cis-1,3-) Dichloropropene (cis-1,3-) Dichloropropene (trans-1,3-) Dichloropropene (trans-1,3-) Ethyl acetate Ethyl acetate Ethylbenzene Ethylbenzene Hexane (n-) Hexane (n-) Isopropanol

NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 User Defined SM 6200 B NPW NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 User Defined SM 6200 B NPW NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 User Defined SM 6200 B NPW NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW SM 6200 B (20th ed)

NPW

EPA 624

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 44 of 74

Isopropanol Methylene chloride (Dichloromethane) Methylene chloride (Dichloromethane) Methyl tert-butyl ether Methyl tert-butyl ether Methyl isobutyl ketone (MIBK) Methyl isobutyl ketone (MIBK) Tert-butyl alcohol Tert-butyl alcohol Tetrahydrofuran Tetrahydrofuran Styrene Styrene Tetrachloroethane (1,1,2,2-)Tetrachloroethane (1,1,2,2)Tetrachloroethane (1,1,1,2-) Tetrachloroethane (1,1,1,2-) Tetrachloroethene Tetrachloroethene Toluene Toluene Trichloroethane (1,1,1-)Trichloroethane (1,1,1-)Trichloroethane (1,1,2-)Trichloroethane (1,1,2-)Trichloroethene Trichloroethene Trichlorofluoromethane Trichlorofluoromethane Trichloro (1,1,2-) trifluoroethane (1,2,2-)Trichloro (1,1,2-) trifluoroethane (1,2,2-)Vinyl acetate Vinyl acetate Vinyl chloride Vinyl chloride Xylenes (total) Xylenes (total) Xylene (m-) Xylene (m-) Xylene (o-) Xylene (o-) Xylene (p-) Xylene (p-) Acetonitrile

NPW User Defined SM 6200 B NPW EPA 624 NPW EPA 624 User Defined SM 6200 B NPW NPW EPA 624 NPW EPA 624 NPW EPA 624 NPW User Defined SM 6200 B NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW EPA 624 NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 45 of 74

Acetonitrile Cyclohexane Hexanone (2-) Hexanone (2-) Methyl acetate Methylcyclohexane Methyl iodide Methyl iodide Ethyl-tert-butyl Ether [ETBE] Ethyl-tert-butyl Ether [ETBE] Diisopropyl Ether [DIPE] Diisopropyl Ether [DIPE] Dioxane (1,4-)Dioxane (1,4-)Butanol (1-) Ethanol Ethanol Ethyl methacrylate Ethyl methacrylate Hexachlorobutadiene (1,3-) Hexachlorobutadiene (1,3-) Iso-butyl alcohol Iso-butyl alcohol Isopropylbenzene Isopropylbenzene Isopropyltoluene (4-) Isopropyltoluene (4-) Methacrylonitrile Methacrylonitrile Methyl methacrylate Methyl methacrylate Naphthalene Naphthalene Octane (-n) Nitropropane (2-) Propionitrile Propionitrile Pentachloroethane Pentachloroethane Propylbenzene (n-) Propylbenzene (n-) Sec-butylbenzene Sec-butylbenzene tert-Amylmethyl ether [TAME] tert-Amylmethyl ether [TAME] Tert-butylbenzene

NPW SM 6200 B (20th ed) **NPW** EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 624 NPW SM 6200 B (20th ed) NPW EPA 625 NPW SM 6410 B NPW EPA 625

### Section 3.0, Ver. 10.0 Date: April 15, 2012 Page: 46 of 74

Tert-butylbenzene Trichlorobenzene (1,2,3-) Trichlorobenzene (1,2,3-) Trichlorobenzene (1,2,4-) Trichlorobenzene (1,2,4-) Trichloropropane (1,2,3-) Trichloropropane (1,2,3-)Trimethylbenzene (1,2,3-) Trimethylbenzene (1,2,3-) Trimethylbenzene (1,2,4-) Trimethylbenzene (1,2,4-) Trimethylbenzene (1,3,5-) Trimethylbenzene (1,3,5-) Acenaphthene Acenaphthene Acenaphthylene Acenaphthylene Anthracene Anthracene Benzo(a)anthracene Benzo(a)anthracene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Benzo(a)pyrene Benzo(ghi)perylene Benzo(ghi)perylene Butyl benzyl phthalate Butyl benzyl phthalate Bis (2-chloroethyl) ether Bis (2-chloroethyl) ether Bis (2-chloroethoxy) methane Bis (2-chloroethoxy) methane Bis (2-ethylhexyl) phthalate Bis (2-ethylhexyl) phthalate Bis (2-chloroisopropyl) ether Bis (2-chloroisopropyl) ether Bromophenyl-phenyl ether (4-) Bromophenyl-phenyl ether (4-) Biphenylamine (4-) Biphenylamine (4-) Chloronaphthalene (2-) Chloronaphthalene (2-) Chlorophenyl-phenyl ether (4-)

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 47 of 74

NPW	SM 6410 B
	EPA 625
	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
	EPA 625
	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
	EPA 625
	SM 6410 B
NPW	EPA 625
	SM 6410 B
	EPA 625
	SM 6410 B
NPW	EPA 625
	SM 6410 B
	EPA 625
	SM 6410 B
NPW	EPA 625
	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	EPA 625
T J T 11	LI / 1 023

C h low when we low house 1 with $c$ with $c$ $(4)$
Chlorophenyl-phenyl ether (4-)
Chrysene
Chrysene
Chloronaphthalene (1-)
Chloronaphthalene (1-)
Dibenzo(a,h)anthracene
Dibenzo(a,h)anthracene
Dibenzofuran
Di-n-butyl phthalate
Di-n-butyl phthalate
Dichlorobenzidine (3,3'-)
Dichlorobenzidine (3,3'-)
Diethyl phthalate
Diethyl phthalate
Dimethyl phthalate
Dimethyl phthalate
Dinitrotoluene (2,4-)
Dinitrotoluene (2,4-)
Dinitrotoluene (2,6-)
Dinitrotoluene (2,6-)
Di-n-octyl phthalate
Di-n-octyl phthalate
Famphur
Famphur
Fluoranthene
Fluoranthene
Fluorene
Fluorene
Hexachlorobenzene
Hexachlorobenzene
Hexachlorobutadiene (1,3-)
Hexachlorobutadiene (1,3-)
Hexachloroethane
Hexachloroethane
Hexachlorophene
Hexachlorophene
Hexachloropropene
Hexachloropropene
Indeno(1,2,3-cd)pyrene
Indeno(1,2,3-cd)pyrene
Isophorone
Isophorone
Kepone
Kepone
Methylnaphthalene (2-)
Naphthalene

# Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 48 of 74

NPW	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	EPA 625
NPW	EPA 625
	EPA 625
	EPA 625
	SM 6410 B
	EPA 625
	SM 6410 B
	EPA 625
	SM 6410 B
	EPA 625
	SM 6410 B
	EPA 625
	EPA 625
	EPA 625
	SM 6410 B
	EPA 625
	EPA 625
	EPA 625
	SM 6410 B
	EPA 625
	SM 6410 B
	EPA 625
	SM 6410 B
	EPA 625
	SM 6410 B
	EPA 625
	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
	EPA 625
	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
	EPA 625
NPW NPW	SM 6410 B
INP W	EPA 625

Naphthalene
Napththylamine (1-)
Napththylamine (1-)
Napththylamine (2-)
Napththylamine (2-)
Chloroaniline (4-)
Nitroaniline (2-)
Nitroaniline (3-)
Nitroaniline (4-)
Nitrobenzene
Nitrobenzene
N-Nitroso-di-n-propylamine
N-Nitroso-di-n-propylamine
Phenanthrene
Phenanthrene
Pyrene
Pyrene
Pentachlorobenzene
Tetrachlorobenzene (1,2,4,5-)
Trichlorobenzene (1,2,4-)
Trichlorobenzene (1,2,4-)
Methylphenol (2-)
Methylphenol (4-)
Methyl phenol (4-chloro-3-)
Methyl phenol (4-chloro-3-)
Chlorophenol (2-)
Chlorophenol (2-)
Dichlorophenol (2,4-)
Dichlorophenol (2,4-)
Dimethylphenol (2,4-)
Dimethylphenol (2,4-)
Dinitrophenol (2,4-)
Dinitrophenol (2,4-)
Dinitrophenol (2-methyl-4,6-)
Dinitrophenol (2-methyl-4,6-)
Nitrophenol (2-)
Nitrophenol (2-)
Nitrophenol (4-)
Nitrophenol (4-)
Pentachlorophenol
Pentachlorophenol
Phenol
Phenol
Tetrachlorophenol (2,3,4,6-)
Tetrachlorophenol (2,3,4,6-)
Trichlorophenol (2,4,5-)

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 49 of 74

NIDIN	
NPW	
	SM 6410 B
NPW	
	SM 6410 B
NPW	
	SM 6410 B
NPW	
NPW	
	SM 6410 B
NPW	
	SM 6410 B
NPW	
	SM 6410 B
NPW	
	SM 6410 B
NPW NPW	
NPW	EPA 625 SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 625
NPW	SM 6410 B
NPW	EPA 507
NPW	User Defined EPA 1657
NPW	EPA 1657
NPW	EPA 1657
NPW	EPA 1657
NPW	EPA 622

Trichlorophenol (2,4,6-)
Trichlorophenol (2,4,6-)
Benzoic acid
Benzoic acid
Methylphenol (4-)
• 1
Methylphenol (4-)
Acetophenone
Acetophenone
Alpha - terpineol
Alpha - terpineol
Aniline
Aniline
Benzidine
Benzidine
Carbazole
Carbazole
Dichloroaniline (2,3-)
Dichloroaniline (2,3-)
Diphenylhydrazine (1,2-)
Methylphenol (2-)
Methylphenol (2-)
Decane (n-)
Decane (n-)
Hexachlorocyclopentadiene
Hexachlorocyclopentadiene
N-Nitroso-di-n-butylamine
N-Nitrosodiethylamine
N-Nitrosodimethylamine
N-Nitrosodimethylamine
N-Nitrosodiphenylamine
N-Nitrosodiphenylamine
N-Nitrosopyrrolidine
Octadecane (n-)
Octadecane (n-)
Pentachloroethane
Pentachloroethane
Pyridine
Pyridine
Napthoquinone (1,4-)
Napthoquinone (1,4-)
Alachlor
Azinphos methyl
Bolstar
Chloropyrifos
Coumaphos
Coumaphos
Coumaphos

Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 50 of 74

NPW SM 6640 B D (2,4-) DB (2,4-) **NPW** EPA 515.5 NPW SM 6640 B Dalapon NPW EPA 1658 Dalapon NPW Demeton (o-) EPA 622 NPW User Defined EPA 1657 Demeton (o-) NPW **EPA 622** Demeton (s-) NPW User Defined EPA 1657 Demeton (s-) NPW User Defined EPA 1657 Diazinon NPW Dicamba EPA 615 NPW User Defined SM 6640B 18/19th ED Dicamba NPW EPA 1658 Dichlorprop NPW EPA 1657 Dichlorvos NPW Dimethoate EPA 1657 NPW EPA 622 Dimethoate NPW EPA 515.5 Dinoseb NPW Dinoseb User Defined SM 6640B NPW User Defined EPA 1657 Disulfoton NPW **EPN** EPA 1657 NPW EPA 507 Ethoprop NPW SM 6630 C Etridiazole NPW EPA 1657 Fensulfothion NPW Fenthion EPA 1657 User Defined EPA 1657 NPW Malathion NPW EPA 555 MCPA NPW EPA 555 MCPP NPW EPA 507 Merphos NPW EPA 507 Metribuzin NPW EPA 507 Mevinphos NPW EPA 1657 Naled NPW EPA 1657 Parathion NPW EPA 622 Parathion NPW EPA 622 Parathion methyl NPW User Defined EPA 1657 Parathion methyl NPW User Defined EPA 1657 Parathion ethyl NPW EPA 1657 Phorate NPW Ronnel EPA 1657 NPW Stirofos EPA 1657 NPW EPA 622 Stirofos NPW EPA 1657 Sulfotepp NPW EPA 622 Sulfotepp NPW SM 6640 B T (2,4,5-) NPW TEPP EPA 1657 NPW TEPP EPA 622 NPW TP (2,4,5-) (Silvex) SM 6640 B NPW EPA 1657 Tokuthion [Protothiofos]

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 51 of 74

NPW	EPA 622	Toluthion [Protothiofos]
NPW	EPA 022 EPA 1657	Tokuthion [Protothiofos] Trichloronate
NPW	EPA 622	Trichloronate
NPW	SM 6630 B	Trifluralin
NPW	EPA 2002.0	
NPW	EPA 2002.0 EPA 2000.0	Toxicity - acute, FW organism
	EPA 2000.0 EPA 1000.0	Toxicity - acute, FW organism
NPW NPW	EPA 1000.0 EPA 1002.0	Toxicity - chronic, FW organism Toxicity - chronic, FW organism
INF W	SM 9222D (Class B only) plus EPA	Toxicity - chrome, I'w organism
SCM	625/R-92/013 Appendix F	Fecal coliform
SCM	SM 9260D plus EPA 625/R-92/013 Appendix F	Salmonella sp. Bacteria
SCM	SW-846 1010	Ignitability
SCM	SW-846 1010A	Ignitability
SCM	User Defined ASTM D93	Ignitability
SCM	SW-846 1030	Ignitability of solids
SCM	SW-846 9040B	Corrosivity - pH waste, >20% water
SCM	SW-846 9040C	Corrosivity - pH waste, >20% water
SCM	SW-846 1110	Corrosivity toward steel
SCM	SW-846 1110A	Corrosivity toward steel
SCM	SW-846 7.3.3.2	Reactivity
SCM	SW-846 7.3.4.2	Reactivity
SCM	SW-846 1311	Volatile organics
SCM	SW-846 1311	Semivolatile organics
SCM	SW-846 1311	Metals
SCM	SW-846 1310A	Metals - organics
SCM	SW-846 1310B	Metals - organics
SCM	SW-846 1312	Metals - organics
SCM	SW-846 1320	Metals - organics
SCM	SW-846 9040B	pH
SCM	SW-846 9040C	pH
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 6010B	Aluminum
SCM	SW-846 6010C	Aluminum
SCM	SW-846 6010B	Antimony
SCM	SW-846 6010C	Antimony
SCM	SW-846 6020	Antimony
SCM	SW-846 6020A	Antimony
SCM	SW-846 6010B	Arsenic
SCM	SW-846 6010C	Arsenic

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 52 of 74

SCM	SW-846 6020
SCM	SW-846 6020A
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6020
SCM	SW-846 6020A
	SW-846 6010B
	SW-846 6010C
	SW-846 6020
	SW-846 6020A
	SW-846 6010B
	SW-846 6010C
	SW-846 6010B
	SW-846 6010C
	SW-846 6020
	SW-846 6020A
	SW-846 6010B
	SW-846 6010C
	SW-846 6010B
	SW-846 6010C
	SW-846 6020
SCM	SW-846 6020A
	SW-846 7196A
	SW-846 6010B
	SW-846 6010D
SCM	SW-846 6010E
	SW-846 6010D
	SW-846 6020
	SW-846 6020 SW-846 6020A
	SW-846 6010B
	SW-846 6010B
	SW-846 6010C
SCM	
	SW-846 6010C SW-846 6020
SCM	SW-846 6020 SW-846 6020A
SCM	SW-846 6020A SW-846 6010B
SCM	
SCM	SW-846 6010C
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM SCM	SW-846 6010B
	SW-846 6010C
SCM	SW-846 6020
SCM	SW-846 6020A
SCM	SW-846 7470A
SCM	SW-846 7471A
SCM	SW-846 7471B

Arsenic Arsenic Barium Barium Barium Barium Beryllium Beryllium Beryllium Beryllium Boron Boron Cadmium Cadmium Cadmium Cadmium Calcium Calcium Chromium Chromium Chromium Chromium Chromium (VI) Cobalt Cobalt Copper Copper Copper Copper Iron Iron Lead Lead Lead Lead Lithium Lithium Magnesium Magnesium Manganese Manganese Manganese Manganese Mercury - liquid waste Mercury - solid waste Mercury - solid waste

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 53 of 74

SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6020
SCM	SW-846 6020A
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6020
SCM	SW-846 6020A
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6020
SCM	SW-846 6020A
SCM	SW-846 6010B
	SW-846 6010C
SCM	SW-846 6020
SCM	SW-846 6020A
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6020
SCM	SW-846 6020A
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6020
SCM	SW-846 6020A
SCM	SW-846 6010B
	SW-846 6010C
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6020
SCM	SW-846 6020A
SCM	SW-846 6010B
SCM	SW-846 6010C
SCM	SW-846 6020
SCM	SW-846 6020A
SCM	SW-846 3540C
SCM	SW-846 3550B
SCM	SW-846 3550C
SCM	SW-846 3546
SCM	SW-846 3580A
SCM	SW-846 3585

Molybdenum Molybdenum Molybdenum Molybdenum Nickel Nickel Nickel Nickel Potassium Potassium Selenium Selenium Selenium Selenium Silver Silver Silver Silver Sodium Sodium Strontium Strontium Thallium Thallium Thallium Thallium Tin Tin Tin Tin Titanium Titanium Vanadium Vanadium Vanadium Vanadium Zinc Zinc Zinc Zinc Semivolatile organics Semivolatile organics Semivolatile organics Semivolatile organics Organics Organics

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 54 of 74

SCM	SW 946 5025 A	Valatile angening law some
SCM	SW-846 5035A	Volatile organics - low conc.
SCM	SW-846 5035L	Volatile organics - low conc.
SCM	SW-846 5035A	Volatile organics - high conc.
SCM	SW-846 5035H	Volatile organics - high conc.
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 8011	Dibromoethane (1,2-) (EDB)
SCM	SW-846 8011	Dibromo-3-chloropropane (1,2-)
SCM	SW-846 8015B	Methyl alcohol (Methanol)
SCM	SW-846 8015D	Methyl alcohol (Methanol)
SCM	SW-846 8015B	Ethyl alcohol
SCM	SW-846 8015D	Ethyl alcohol
SCM	SW-846 8015B	Gasoline range organic
SCM	SW-846 8015D	Gasoline range organic
SCM	User Defined MA-DEP-VPH, WI	Gasoline range organic
SCIVI	GRO, NW TPH Gx	Gasonne range of game
SCM	SW-846 8015B	Diesel range organic
SCM	SW-846 8015D	Diesel range organic
SCM	User Defined MA-DEP-EPH, TN-	Diesel range organic
SCIVI	EPH, WI DRO, NW TPH Dx	Dieser range organie
SCM	User Defined NWTPH-Dx,	Petroleum Organics
SCIM	NWTPH-Gx, NWTPHID	r eu oleum Organics
SCM	Other FL - PRO	Petroleum Organics
SCM	User Defined TX 1005, TX 1006,	Petroleum Organics
SCIVI	CT ETPH, NW TPH ID	Tenoleum Organies
SCM	Other IA - OA-1	Petroleum Organics
SCM	Other IA - OA-2	Petroleum Organics
SCM	User Defined CA LUFT - diesel	Petroleum Organics
SCM	Other NJ-OQA-QAM-025, Rev. 7	Petroleum Organics
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 8021B	Xylenes (total)
SCM	SW-846 8021B	Methyl tert-butyl ether
SCM	SW-846 8081A	Alachlor
SCM	SW-846 8081B	Alachlor
SCM	SW-846 8081A	Aldrin

## Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 55 of 74

SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A

Aldrin Alpha BHC Alpha BHC Beta BHC Beta BHC Delta BHC Delta BHC Lindane (gamma BHC) Lindane (gamma BHC) Chlordane (technical) Chlordane (technical) Chlordane (alpha) Chlordane (alpha) Chlordane (gamma) Chlordane (gamma) Chloroneb Chloroneb Chlorothalonil Chlorothalonil DDD (4,4'-) DDD (4,4'-) DDE (4,4'-) DDE (4,4'-) DDT (4,4'-) DDT (4,4'-) Dieldrin Dieldrin Endosulfan I Endosulfan I Endosulfan II Endosulfan II Endosulfan sulfate Endosulfan sulfate Endrin Endrin Endrin aldehyde Endrin aldehyde Endrin ketone Endrin ketone Etridiazole Etridiazole Heptachlor Heptachlor Heptachlor epoxide Heptachlor epoxide Hexachlorobenzene

Castian	9.0	Van	10.0	
Section	J.U,	ver.	10.0	

Date: April 15, 2012 Page: 56 of 74

SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
SCM	SW-846 8081B
SCM	SW-846 8081A
	SW-846 8081B
SCM	SW-846 8081A
	SW-846 8081B
	SW-846 8081A
	SW-846 8081B
SCM	SW-846 8081A
	SW-846 8081B
	SW-846 8081B
	SW-846 8082A
SCM	SW-846 8082
	SW-846 8082A
	SW-846 8082
	SW-846 8082A
SCM	SW-846 8082
	SW-846 8082A
	SW-846 8082
SCM	SW-846 8082A
SCM	SW-846 8082
SCM	SW-846 8082A
SCM	SW-846 8082
SCM	SW-846 8082A
SCM	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141B SW-846 8141A
SCM	SW-846 8141A SW-846 8141B
SCM	SW-846 8141B SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A

Hexachlorobenzene Hexachlorocyclopentadiene Hexachlorocyclopentadiene Methoxychlor Methoxychlor Permethrin Permethrin Propachlor Propachlor Toxaphene Toxaphene Trifluralin Trifluralin PCB 1016 PCB 1016 PCB 1221 PCB 1221 PCB 1232 PCB 1232 PCB 1242 PCB 1242 PCB 1248 PCB 1248 PCB 1254 PCB 1254 PCB 1260 PCB 1260 Azinphos methyl Azinphos methyl Bolstar **Bolstar** Chloropyrifos Chloropyrifos Coumaphos Coumaphos Demeton (o-) Demeton (o-) Demeton (s-) Demeton (s-) Diazinon Diazinon Dichlorvos Dichlorvos Dimethoate Dimethoate Disulfoton

# Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 57 of 74

SCM	SW-846 8141B
SCM	SW-846 8141A
SCM	SW-846 8141B
	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
	SW-846 8141B
	SW-846 8141A
SCM	SW-846 8141B
	SW-846 8141A
	SW-846 8141B
	SW-846 8141A
	SW-846 8141B
SCM	SW-846 8141A
	SW-846 8141B
	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
	SW-846 8141B
	SW-846 8141A
	SW-846 8141B
SCM	SW-846 8141A
	SW-846 8141B
	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
	SW-846 8141B
	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
	SW-846 8141B
	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8141A
SCM	SW-846 8141B
SCM	SW-846 8151A
SCM	SW-846 8310

Disulfoton
EPN
EPN
Ethoprop
Ethoprop
Fensulfothion
Fensulfothion
Fenthion
Fenthion
Malathion
Malathion
Merphos
Merphos
-
Mevinphos
Mevinphos
Naled
Naled
Parathion
Parathion
Parathion methyl
Parathion methyl
Phorate
Phorate
Ronnel
Ronnel
Stirofos
Stirofos
Sulfotepp
Sulfotepp
TEPP
TEPP
Tokuthion [Protothiofos]
Tokuthion [Protothiofos]
Trichloronate
Trichloronate
Dalapon
Dicamba
Dichlorprop
Dinoseb
D (2,4-)
DB (2,4-)
T (2,4,5-)
TP (2,4,5-) (Silvex)
MCPA
MCPP
Acenaphthene

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 58 of 74

SCM	SW-846 8310
SCM	SW-846 8310
	SW-846 8310
	SW-846 8310
	SW-846 8310
SCM	SW-846 8310
	SW-846 8330
	SW-846 8330A
	SW-846 8330
	SW-846 8330A
	SW-846 8330
SCM	SW-846 8330A
SCM	SW-846 8330
SCM	SW-846 8330A
SCM	SW-846 8330
SCM	SW-846 8330A
	SW-846 8330
	SW-846 8330A
	SW-846 8330
	SW-846 8330A
	SW-846 8330
	SW-846 8330A
	SW-846 8330
SCM	SW-846 8330A
SCM	SW-846 8330
SCM	SW-846 8330A
SCM	SW-846 8330
SCM	SW-846 8330A
SCM	SW-846 8330
SCM	SW-846 8330A
SCM	SW-846 8330
SCM	SW-846 8330A
SCM	SW-846 8330
SCM	SW-846 8330A
SCM	SW-846 8330
SCM	SW-846 8330A
SCM	SW-846 8330

Acenaphthylene Anthracene Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(ghi)perylene Benzo(k)fluoranthene Chrysene Dibenzo(a,h)anthracene Fluoranthene Fluorene Indeno(1,2,3-cd)pyrene Naphthalene Phenanthrene Pyrene HMX HMX RDX RDX Trinitrobenzene (1,3,5-) Trinitrobenzene (1,3,5-) Dinitrobenzene (1,3-) Dinitrobenzene (1,3-) PETN PETN Tetryl Tetryl Nitrobenzene Nitrobenzene Trinitrotoluene (2,4,6-) Trinitrotoluene (2,4,6-) Dinitrotoluene (4-amino-2,6-) Dinitrotoluene (4-amino-2,6-) Dinitrotoluene (2-amino-4,6-) Dinitrotoluene (2-amino-4,6-) Dinitrotoluene (2,4-) Dinitrotoluene (2,4-) Dinitrotoluene (2,6-) Dinitrotoluene (2,6-) Nitrotoluene (2-) Nitrotoluene (2-) Nitrotoluene (3-) Nitrotoluene (3-) Nitrotoluene (4-) Nitrotoluene (4-) Nitroglycerine

# Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 59 of 74

SCM	SW-846 8330A
SCM	SW-846 8260B
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	User Defined LUFT
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	User Defined LUFT
SCM	SW-846 8260B
SCM	SW-846 8260C
	SW-846 8260B
	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	User Defined LUFT
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260C SW-846 8260B
SCM	SW-846 8260B SW-846 8260C
SCM SCM	SW-846 8260C SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B

Nitroglycerine Safrole Benzene Benzene Benzene Bromobenzene Bromobenzene Butyl benzene (n-) Butyl benzene (n-) Sec-butylbenzene Sec-butylbenzene Tert-butylbenzene Tert-butylbenzene Chlorobenzene Chlorobenzene Chlorotoluene (2-) Chlorotoluene (2-) Chlorotoluene (4-) Chlorotoluene (4-) Dichlorobenzene (1,2-) Dichlorobenzene (1,2-) Dichlorobenzene (1,3-) Dichlorobenzene (1,3-) Dichlorobenzene (1,4-) Dichlorobenzene (1,4-) Ethylbenzene Ethylbenzene Ethylbenzene Isopropylbenzene Isopropylbenzene Propylbenzene (n-) Propylbenzene (n-) Toluene Toluene Toluene Isopropyltoluene (4-) Isopropyltoluene (4-) Trichlorobenzene (1,2,3-) Trichlorobenzene (1,2,3-) Trimethylbenzene (1,2,4-) Trimethylbenzene (1,2,4-) Trimethylbenzene (1,3,5-) Trimethylbenzene (1,3,5-) Trimethylbenzene (1,2,3-) Trimethylbenzene (1,2,3-) Xylenes (total)

Section 3.0, V	Ver. 10.0
----------------	-----------

Date: April 15, 2012 Page: 60 of 74

SCM SW-846 8260C SCM User Defined LUFT SCM SW-846 8260B SCM SW-846 8260C SCM User Defined LUFT SCM SW-846 8260B SCM SW-846 8260C SCM User Defined LUFT SCM SW-846 8260B SCM SW-846 8260C SCM User Defined LUFT SCM SW-846 8260B SW-846 8260C SCM SCM SW-846 8260B SCM SW-846 8260C SCM SW-846 8260B SW-846 8260C SCM SCM SW-846 8260B SCM SW-846 8260C SW-846 8260B SCM SCM SW-846 8260C SCM SW-846 8260B SCM SW-846 8260C SCM SW-846 8260B

Xylenes (total) Xylene (m-) Xylene (m-) Xylene (m-) Xylene (o-) Xylene (o-) Xylene (o-) Xylene (p-) Xylene (p-) Xylene (p-) tert-Amylmethyl ether [TAME] tert-Amylmethyl ether [TAME] Allyl chloride Allyl chloride Bromochloromethane Bromochloromethane Bromodichloromethane Bromodichloromethane Bromoethane Bromoethane Bromoform Bromoform Bromomethane Bromomethane Cyclohexane Cyclohexane Cyclohexanone Cyclohexanone Butadiene (2-chloro-1,3-) Butadiene (2-chloro-1,3-) Dichloro-2-butene (cis-1,4-) Dichloro-2-butene (cis-1,4-) Carbon tetrachloride Carbon tetrachloride Chloroethane Chloroethane Chloroethyl vinyl ether (2-) Chloroethyl vinyl ether (2-) Chloroform Chloroform Chloromethane Chloromethane Diethyl ether (Ethyl ether) Diethyl ether (Ethyl ether) Dichloropropene (trans-1,3-)

Xylenes (total)

SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C

SCM

SW-846 8260B

# Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 61 of 74

Dichloropropene (trans-1,3-) Dibromochloromethane Dibromochloromethane Dibromoethane (1,2-) (EDB) Dibromoethane (1,2-) (EDB) Dibromomethane Dibromomethane Dibromo-3-chloropropane (1,2-) Dibromo-3-chloropropane (1,2-) Dichlorodifluoromethane Dichlorodifluoromethane Dichloroethane (1,1-)Dichloroethane (1,1-)Dichloroethane (1,2-)Dichloroethane (1,2-)Dichloroethene (1,1-) Dichloroethene (1,1-)Dichloroethene (trans-1,2-) Dichloroethene (trans-1.2-) Dichloroethene (cis-1,2-) Dichloroethene (cis-1,2-) Dichloropropane (1,2-) Dichloropropane (1,2-) Dichloropropane (1,3-) Dichloropropane (1,3-) Dichloropropane (2,2-) Dichloropropane (2,2-) Dichloropropene (1,1-) Dichloropropene (1,1-) Dichloropropene (cis-1,3-) Dichloropropene (cis-1,3-) Dichloro-2-butene (trans-1,4-) Dichloro-2-butene (trans-1,4-) Diisopropyl Ether [DIPE] Diisopropyl Ether [DIPE] Butanol (1-) Butanol (1-) Ethanol Ethanol Methylene chloride (Dichloromethane) Methylene chloride (Dichloromethane) Nitropropane (2-) Nitropropane (2-) Tetrachloroethane (1,1,2,2-) Tetrachloroethane (1,1,2,2-) Tetrachloroethene

Date: April 15, 2012 Page: 62 of 74

SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
	SW-846 8260C
SCM	
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	
	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCIVI	SW-040 8200B

Tetrachloroethene Tetrahydrofuran Tetrahydrofuran Trichloroethane (1,1,1-)Trichloroethane (1,1,1-) Trichloroethane (1,1,2-)Trichloroethane (1,1,2-)Trichloroethene Trichloroethene Trichlorofluoromethane Trichlorofluoromethane Trichloro (1,1,2-) trifluoroethane (1,2,2-)Trichloro (1,1,2-) trifluoroethane (1,2,2-) Trichloropropane (1,2,3-) Trichloropropane (1,2,3-)Vinyl acetate Vinyl acetate Vinyl chloride Vinyl chloride Acetone Acetone Carbon disulfide Carbon disulfide Butanol (3,3-Dimethyl-1-) Butanol (3,3-Dimethyl-1-) Butanone (2-) Butanone (2-) Butyl formate (t-) Butyl formate (t-) Ethyl-tert-butyl Ether [ETBE] Ethyl-tert-butyl Ether [ETBE] Ethyl acetate Ethyl acetate Ethyl methacrylate Ethyl methacrylate Hexanone (2-) Hexanone (2-) Methacrylonitrile Methacrylonitrile Methyl acrylate Methyl acrylate Methyl methacrylate Methyl methacrylate Methyl acetate

# Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 63 of 74

SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
	SW-846 8260B
	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
	SW-846 8260B
	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
	SW-846 8260B
	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	User Defined LUFT
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	User Defined 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
JUNI	5 TI-0TO 0200C

Methyl acetate Methyl iodide Methyl iodide Iso-butyl alcohol Iso-butyl alcohol Isopropanol Isopropanol N-Nitroso-di-n-butylamine N-Nitroso-di-n-butylamine Pentachloroethane Pentachloroethane Pentanone (4-methyl-2-) (MIBK) Pentanone (4-methyl-2-) (MIBK) Pentanol (2-Methyl-2-) Pentanol (2-Methyl-2-) Propionitrile Propionitrile Methyl tert-butyl ether Methyl tert-butyl ether Methyl tert-butyl ether Amyl alcohol (t-) Amyl alcohol (t-) Tert-butyl alcohol Tert-butyl alcohol Acetonitrile Acetonitrile Acrolein Acrolein Acrylonitrile Acrylonitrile Hexane (n-) Hexane (n-) Hexachlorobutadiene (1,3-) Hexachlorobutadiene (1,3-) Hexachloroethane Hexachloroethane Methylcyclohexane Methylcyclohexane Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Methylnaphthalene (2-) Naphthalene Naphthalene Octane (-n) Octane (-n)

Section 3.0, Ver. 10.0	
Date: April 15, 2012	
Page: 64 of 74	

SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
SCM	SW-846 8260B
SCM	SW-846 8260C
COM	User Defined SW846 8260B &
SCM	8260C
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D

Styrene Styrene Tetrachloroethane (1,1,1,2- Tetrachloroethane (1,1,1,2- Trichlorobenzene (1,2,4-) Trichlorobenzene (1,2,4-) Trimethylpentane (2,2,4-) Trimethylpentane (2,2,4-) Dioxane (1,4-)	
Dioxane (1,4-)	
Gasoline range organic	
Dibenzo(a,e)pyrene Dibenzo(a,e)pyrene Dibenz(a,h)acridine Dibenz(a,h)acridine Dibenzo(a,h)pyrene Dibenzo(a,h)pyrene Dibenz(a,j)acridine Dibenz(a,j)acridine	

scope a	na Definitions
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C

SCM

SW-846 8270D

SW-846 8270C

Date: April 15, 2012 Page: 65 of 74

Dibenzo(a,i)pyrene Dibenzo(a,i)pyrene Dibenzo(c,g)carbazole (7H-) Dibenzo(c,g)carbazole (7H-) Dichlorophenol (2,6-) Dichlorophenol (2,6-) Dimethoate Dimethoate Dimethylaminoazobenzene Dimethylaminoazobenzene Dimethylbenz(a)anthracene (7,12-) Dimethylbenz(a)anthracene (7,12-) Dimethyl benzidine (3,3-) Dimethyl benzidine (3,3-) Dinitrobenzene (1,3-) Dinitrobenzene (1,3-) Dinoseb Dinoseb Disulfoton Disulfoton Famphur Famphur Hexachlorophene Hexachlorophene Hexachloropropene Isodrin Isodrin Isosafrole (cis-) Isosafrole (cis-) Isosafrole (trans-) Isosafrole (trans-) Kepone Kepone Methanesulfonate (Ethyl-) Methanesulfonate (Ethyl-) Methanesulfonate (Methyl-) Methanesulfonate (Methyl-) Methapyrilene Methapyrilene Methylcholanthrene (3-) Methylcholanthrene (3-) Napthoquinone (1,4-) Napthoquinone (1,4-) Napththylamine (1-) Napththylamine (1-) Napththylamine (2-)

# Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 66 of 74

SCM	SW-846 8270D
SCM	SW-846 8270C
	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270D
	SW-846 8270C
SCM	
	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
DCIVI	5W-0400270D
SCM	SW-846 8270C
SCM SCM	SW-846 8270C SW-846 8270D
SCM	SW-846 8270D
SCM SCM	SW-846 8270D SW-846 8270C
SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D
SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C
SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D
SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C
SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D
SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D
SCM SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D
SCM SCM SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D
SCM SCM SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D SW-846 8270C SW-846 8270D
SCM SCM SCM SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D SW-846 8270D SW-846 8270D SW-846 8270D SW-846 8270D SW-846 8270D
SCM SCM SCM SCM SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D SW-846 8270D SW-846 8270C SW-846 8270C SW-846 8270D
SCM SCM SCM SCM SCM SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D SW-846 8270D SW-846 8270D SW-846 8270D SW-846 8270D SW-846 8270D
SCM SCM SCM SCM SCM SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D
SCM SCM SCM SCM SCM SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D
SCM SCM SCM SCM SCM SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D
SCM SCM SCM SCM SCM SCM SCM SCM SCM SCM	SW-846 8270D SW-846 8270C SW-846 8270D SW-846 8270D

(upunitylumite (2)
Nitrodiphenylamine (2-)
Nitrodiphenylamine (2-)
N-Nitroso-di-n-butylamine
N-Nitroso-di-n-butylamine
N-Nitrosomorpholine
N-Nitrosomorpholine
N-Nitrosopiperidine
N-Nitrosopiperidine
Parathion
Parathion
Parathion methyl
Parathion methyl
Pentachlorobenzene
Pentachlorobenzene
Pentachloroethane
Pentachloroethane
Pentachloronitrobenzene
Pentachloronitrobenzene
Phenacetin
Phenacetin
Phenylenediamine (1,4-)
Phenylenediamine (1,4-)
Phenylethylamine (alpha, alpha-
Dimethyl)
Phenylethylamine (alpha, alpha-
Dimethyl)
Phorate
Phorate
Phosphorothioate (O,O,O-triethyl)
Phosphorothioate (O,O,O-triethyl)
Phosphorothioate (O,O-diethyl-O-2-
pyrazinyl) [Thionazin]
Phosphorothioate (O,O-diethyl-O-2-
pyrazinyl) [Thionazin]
Picoline (2-)
Picoline (2-)
Pronamide
Pronamide
Quinoline -1-Oxide (4-Nitro)
Quinoline -1-Oxide (4-Nitro)
Safrole
Safrole
Sulfotepp
Sulfotepp
Tetrachlorobenzene (1,2,3,4-)

Napththylamine (2-)

SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C

Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 67 of 74

Tetrachlorobenzene (1,2,3,4-) Tetrachlorobenzene (1,2,3,5-) Tetrachlorobenzene (1,2,3,5-) Tetrachlorobenzene (1,2,4,5-) Tetrachlorobenzene (1,2,4,5-) Tetrachlorophenol (2,3,4,6-) Tetrachlorophenol (2,3,4,6-) Toluidine (2-) (2-Methylaniline) Toluidine (2-) (2-Methylaniline) Toluidine (5-nitro-2-) Toluidine (5-nitro-2-) Trinitrobenzene (1,3,5-) Trinitrobenzene (1,3,5-) N-Nitrosodiethylamine N-Nitrosodiethylamine N-Nitrosodimethylamine N-Nitrosodimethylamine N-Nitroso-di-n-propylamine N-Nitroso-di-n-propylamine N-Nitrosodiphenylamine N-Nitrosodiphenylamine N-Nitrosomethylethylamine N-Nitrosomethylethylamine N-Nitrosopyrrolidine N-Nitrosopyrrolidine Diphenylamine Diphenylamine Carbazole Carbazole Benzidine Benzidine Dichlorobenzidine (3,3'-) Dichlorobenzidine (3,3'-) Diphenylhydrazine (1,2-) Diphenylhydrazine (1,2-) Aniline Aniline Chloraniline (4-) Chloraniline (4-) Nitroaniline (2-) Nitroaniline (2-) Nitroaniline (3-) Nitroaniline (3-) Nitroaniline (4-) Nitroaniline (4-) Chloronaphthalene (2-)

#### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 68 of 74

SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270D
SCM	SW-846 8270C
	SW-846 8270D SW-846 8270C
SCM	
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C

Chloronaphthalene (2-) Hexachlorobenzene Hexachlorobenzene Hexachlorobutadiene (1,3-) Hexachlorobutadiene (1,3-) Hexachlorocyclopentadiene Hexachlorocyclopentadiene Hexachloroethane Hexachloroethane Hexachloropropene Hexachloropropene Trichlorobenzene (1,2,4-) Trichlorobenzene (1,2,4-) Bis (2-chloroethoxy) methane Bis (2-chloroethoxy) methane Bis (2-chloroethyl) ether Bis (2-chloroethyl) ether Bis (2-chloroisopropyl) ether Bis (2-chloroisopropyl) ether Chlorophenyl-phenyl ether (4-) Chlorophenyl-phenyl ether (4-) Bromophenyl-phenyl ether (4-) Bromophenyl-phenyl ether (4-) Dinitrotoluene (2,4-) Dinitrotoluene (2,4-) Dinitrotoluene (2,6-) Dinitrotoluene (2,6-) Isophorone Isophorone Nitrobenzene Nitrobenzene Butyl benzyl phthalate Butyl benzyl phthalate Bis (2-ethylhexyl) phthalate Bis (2-ethylhexyl) phthalate Diethyl phthalate Diethyl phthalate Dimethyl phthalate Dimethyl phthalate Di-n-butyl phthalate Di-n-butyl phthalate Di-n-octyl phthalate Di-n-octyl phthalate Acenaphthene Acenaphthene Anthracene

# Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 69 of 74

SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270D
SCM	SW-846 8270C
	SW-846 8270D
SCM	
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
	SW-846 8270D
SCM	
SCM SCM	SW-846 8270D
	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C

Anthracene
Acenaphthylene
Acenaphthylene
Benzo(a)anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(b)fluoranthene
Benzo(ghi)perylene
Benzo(ghi)perylene
Benzo(k)fluoranthene
Benzo(k)fluoranthene
Chrysene
Chrysene
Dibenzo(a,h)anthracene
Dibenzo(a,h)anthracene
Fluoranthene
Fluoranthene
Fluorene
Fluorene
Indeno(1,2,3-cd)pyrene
Indeno(1,2,3-cd)pyrene
Methylnaphthalene (1-)
Methylnaphthalene (1-)
Methylnaphthalene (2-)
Methylnaphthalene (2-)
Naphthalene
Naphthalene
Phenanthrene
Phenanthrene
Pyrene
Pyrene
Methyl phenol (4-chloro-3-)
Methyl phenol (4-chloro-3-)
Chlorophenol (2-)
Chlorophenol (2-)
Dichlorophenol (2,4-)
Dichlorophenol (2,4-)
Dimethylphenol (2,4-)
Dimethylphenol (2,4-)
Dinitrophenol (2,4-)
Dinitrophenol (2,4-)
Dinitrophenol (2,4-) Dinitrophenol (2-methyl-4,6-)
Dinitrophenol (2-methyl-4,6-)
Methylphenol (2-)
Wethylphenol (2-)

# Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 70 of 74

~ ~ ~ ~	
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	User Defined CA LUFT - diesel
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D

Methylphenol (2-)
Methylphenol (4-)
Methylphenol (4-)
Nitrophenol (2-)
Nitrophenol (2-)
Nitrophenol (4-)
Nitrophenol (4-)
_
Pentachlorophenol
Pentachlorophenol
Phenol
Phenol
Trichlorophenol (2,4,5-)
Trichlorophenol (2,4,5-)
Trichlorophenol (2,4,6-)
Trichlorophenol (2,4,6-)
Dibenzofuran
Dibenzofuran
Dichlorobenzene (1,2-)
Dichlorobenzene (1,2-)
Dichlorobenzene (1,3-)
Dichlorobenzene (1,3-)
Dichlorobenzene (1,4-)
Dichlorobenzene (1,4-)
Benzaldehyde
Benzaldehyde
Benzoic acid
Benzoic acid
Benzyl alcohol
Benzyl alcohol
Decane (n-)
Decane (n-)
Octadecane (n-)
Octadecane (n-)
Petroleum Organics
Pyridine
Pyridine
Caprolactam
Caprolactam
Atrazine
Atrazine
Acenaphthene
Acenaphthene
Acenaphthylene
Acenaphthylene
Anthracene
Anthracene

### Section 3.0, Ver. 10.0

Date: April 15, 2012 Page: 71 of 74

SCM	SW-846 8270C
	SW-846 8270D
SCM	SW-846 8270C
	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
	SW-846 8270D
SCM	SW-846 8270C
	SW-846 8270D
	SW-846 8270C
SCM	SW-846 8270D
SCM	SW-846 8270C
	SW-846 8270D
	SW-846 8270C
	SW-846 8270D
	SW-846 8270C
	SW-846 8270D
	SW-846 8270C
	SW-846 8270D
	SW-846 8440
	SW-846 9010C
SCM	
SCM	SW-846 9010C
SCM	User Defined 9010B
SCM	SW-846 9013
SCM	User Defined 9013A
SCM	SW-846 9012B
SCM	User Defined 9012A
SCM	SW-846 9023
SCM	SW-846 9030B
SCM	SW-846 9034
SCM	SW-846 9056
SCM	SW-846 9056A
SCM	SW-846 9040C
SCM	SW-846 9045C

Benzo(a)anthracene Benzo(a)anthracene Benzo(a)pyrene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(k)fluoranthene Benzo(ghi)perylene Benzo(ghi)perylene Chrysene Chrysene Dibenzo(a,h)anthracene Dibenzo(a,h)anthracene Indeno(1,2,3-cd)pyrene Indeno(1,2,3-cd)pyrene Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Methylnaphthalene (2-) Naphthalene Naphthalene Fluoranthene Fluoranthene Fluorene Fluorene Phenanthrene Phenanthrene Pyrene Pyrene Total rec. petroleum hydrocarbons Cyanide Cyanide Cyanide - amenable to Cl2 Cyanide - amenable to Cl2 Cyanide Cyanide Cyanide Cyanide Extractable organic halides (EOX) Sulfides, acid sol. & insol. Sulfides, acid sol. & insol. Sulfate Sulfate pH - waste, >20% water pH - soil and waste

Section 3.0, Ver. 10.0	
Date: April 15, 2012	

SCM	SW-846 9045D	pH - soil and waste
SCM	SW-846 9060	Total organic carbon (TOC)
SCM	SW-846 9060A	Total organic carbon (TOC)
SCM	ASTM F1647-02A	Total organic carbon (TOC)
SCM	Other Walkley Black	Total organic carbon (TOC)
SCM	Other USDA-LOI (Loss on ignition)	Total organic carbon (TOC)
SCM	SW-846 9071 B	Oil & grease - sludge-hem
SCM	SW-846 9071 B	Oil & grease - sludge-hem-npm
SCM	ASTM D5468 and D482	% ash
SCM	ASTM D240	Heat of combustion (BTU)
SCM	SW-846 9095	Free liquid
SCM	SW-846 9095B	Free liquid
SCM	User Defined 9095A	Free liquid
SCM	SW-846 9056	Nitrite
SCM	SW-846 9056A	Nitrite
SCM	SW-846 9056	Nitrate
SCM	SW-846 9056A	Nitrate
SCM	SW-846 9056	Bromide
SCM	SW-846 9056A	Bromide
SCM	SW-846 9056	Chloride
SCM	SW-846 9056A	Chloride
SCM	SW-846 9056	Fluoride
SCM	SW-846 9056A	Fluoride
SCM		Perchlorate
SCM	SW-846 9056	Orthophosphate
SCM	SW-846 9056A	Orthophosphate
SCM	EPA 300.0	Guanidine nitrate
SCM	User Defined SW-846 8330	Nitroguanidine
SCM	SW-846 8330	Guanidine nitrate
SCM	SW-846 8260B	Trimethylpentane (2,2,4-)
SCM	SW-846 8270C	Nitrodiphenylamine (2-)
SCM	SM 2540 G SM 18th Ed.	Total, fixed, and volatile solids (SQAR)

Page: 72 of 74

# 3.4 ABBREVIATIONS/ACRONYMS

The quality department is responsible for setting up and maintaining a list of abbreviations used in the quality manual.

ABBREVIATION	DESCRIPTION
A2LA	AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION
AIHA	AMERICAN INDUSTRIAL HYGIENE ASSOCIATION
BLANK	See FIELD, TRIP, METHOD, EQUIPMENT
CAL	CALIBRATION
ССВ	CONTINUING CALIBRATION BLANK
CCV	CONTINUING CALIBRATION VERIFICATION
CDOC	CONTINUING DEMONSTRATION OF CAPABILITY
COC	CHAIN OF CUSTODY
СА	CORRECTIVE ACTION
DQO	DATA QUALITY OBJECTIVES
DUP	DUPLICATE
EB	EQUIPMENT BLANK
FB	FIELD BLANK
GC	GAS CHROMATOGRAPHY
GCMS	GAS CHROMATOGRAPHY MASS SPECTROMETRY
HPLC	HIGH PRESSURE LIQUID CHROMATOGRAPHY
IC	ION CHROMATOGRAPHY
ICP	INDUCTIVELY COUPLED PLASMA
ICPMS	INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY
ICS	INTERFERENCE CHECK SAMPLE
ICV – See SSCV	INITIAL CALIBRATION VERIFICATION
IDOC	INITIAL DEMONSTRATION OF CAPABILITY (SEE ALSO CDOC)
IDL	INSTRUMENT DETECTION LIMIT
IS	INTERNAL STANDARD
LCS	LABORATORY CONTROL SAMPLE (Typically 2 <sup>ND</sup> Source)
LOD	LIMIT OF DETECTION
LDR	LINEAR DYNAMIC RANGE
MAT	MATRIX
MS	MATRIX SPIKE
MSD	MATRIX SPIKE DUPLICATE
MDL	METHOD DETECTION LIMIT
MB	METHOD BLANK
NC	NEGATIVE CONTROL
NELAP	NATIONAL ENVIRONMENTAL LABORATORY ACCREDITATION
% Rec	PERCENT RECOVERY

Date: April 15, 2012 Page: 74 of 74

ABBREVIATION	DESCRIPTION
PC	POSITIVE CONTROL
PDL	PRACTICAL DETECTION LIMIT
PQL	PRACTICAL QUANTITATION LIMIT also See Reporting Limit (RL)
PT	PROFICIENCY TEST SAMPLE
QUAL	QUALIFIER
QA	QUALITY ASSURANCE
QAM	QUALITY ASSURANCE MANUAL
QAO	QUALITY ASSURANCE OFFICER
QC	QUALITY CONTROL
RL	REPORTING LIMIT
RPD	RELATIVE PERCENT DIFFERENCE
RF	RESPONSE FACTOR
SSCV	SECONDARY SOURCE CALIBRATION VERIFICAION
SOP	STANDARD OPERATING PROCEDURE
SRM	STANDARD REFERENCE MATERIAL
SURR	SURROGATE
UV	ULTRAVIOLET
VOC	VOLATILE ORGANIC COMPOUND

## 4.0 MANAGEMENT REQUIREMENTS

### 4.1 ORGANIZATION

#### 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 at 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 maintain 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 Manager, Technical Director or the Quality Manager. The organizational structure indicated in this section is designed to minimize the potential for conflicting or undue stresses that might influence the technical judgment of analytical personnel. Additionally, it provides adequate management for consistent supervision of laboratory practices 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.

### 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. Mr. Schulert is responsible for developing and executing ESC's strategic plan. 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 operational responsibilities are delegated to the Chief Financial Officer, Laboratory Director, Director of Technical & Regulatory Affairs, and the Chief Information Officer.

## 4.1.5.2 Director of Technical & Regulatory Affairs

Judith R. Morgan, Master of Science in Analytical Chemistry and Registered Environmental Manager, is the Laboratory Director of Technical & Regulatory Affairs and serves as the laboratory Quality Assurance Officer (QAO). She has been serving the environmental industry since 1986 and is a respected expert witness. The majority of her experience is specific to quality and regulatory matters; however, she does have previous experience as an analyst in both organic and inorganic methods. In matters of laboratory QA/QC, she reports directly to Peter Schulert, CEO, thus making her QAO functions separate from laboratory operations. Her primary responsibility is the oversight of administrative and technical operations of the laboratory. She specifies and/or approves all methodologies used in the laboratory and ensures continued accreditation of the laboratory. She is responsible for maintaining the laboratory QA manual, initiating and overseeing audits, activating corrective measures (when necessary), implementing numerous international quality standards and preparing internal QA/QC reports. Additionally, she oversees the Technical Specialist group, which includes personnel who are considered to be experts in one or more facets of the laboratory. The Technical group maintains specific regulatory information that impacts quality, client relations, and strategic marketing. Dixie Marlin assumes responsibility for all QA functions, in the absence of the director.

#### 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 CEO. In his absence, all operations responsibilities are delegated to Tom Mellette and then to individual department managers.

#### 4.1.5.4 Quality Control Manager

Dixie Marlin, B.S. in Biology, is the laboratory Quality Control Manager. She has more than 20 years of combined laboratory experience in research, regulatory, and production lab environments. This experience has spanned the environmental lab in both privately owned, university facilities, and Federal Superfund sectors, with additional experience gained in state regulatory agencies. Her primary function is to assist production chemists/technicians regarding quality assurance/control measures, ensure compliance with method requirements and procedures, and perform audits of internal laboratory functions. Where necessary, she identifies, develops, and implements improvement of the laboratory measurement capability to meet the requirements of governing authorities, department programs, and laboratory clients. She is responsible for the supervision of the laboratory QC group and technical specialists. Judith Morgan assumes responsibility for these functions in her absence.

## 4.1.5.5 Chief Information Officer

Jeff Chandler, B.S. in Computer Science, is the ESC CIO. His responsibilities include direction of laboratory computer systems, internal and external software development, database management, records management system and control of ESC's laboratory information management system. Prior to joining ESC, Mr. Chandler served as VP of eCommerce for a large internet retailer for seven years, preceded by three years in management within a major consulting company. He has over twenty-three years experience in information technology disciplines, including project management, software development, hardware infrastructure planning /deployment, and voice/data analysis. Tom White is responsible for the department in Mr. Chandler's absence.

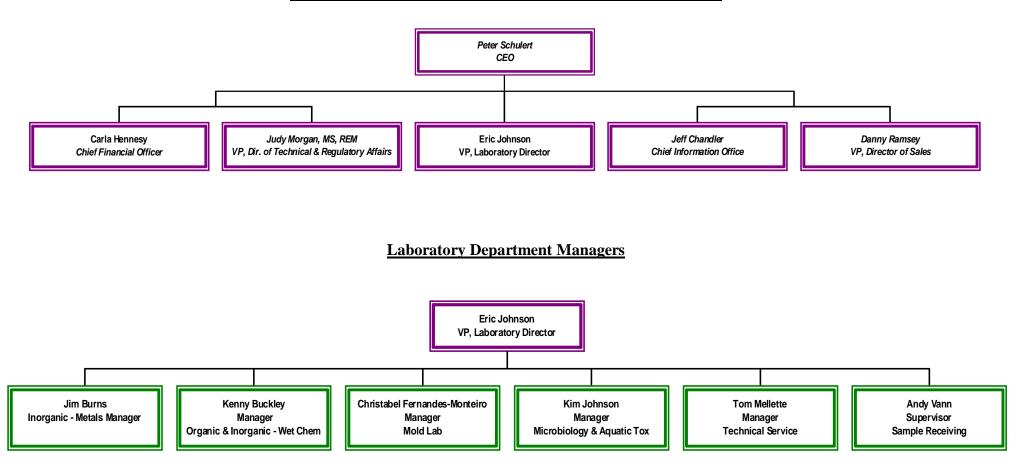
#### 4.1.6 Management System Effectiveness

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 corporate organizational chart, which lists key individuals and relevant departmental structure.

ESC Lab Sciences Quality Assurance Manual Organization And Responsibility Section 4.0, Ver. 10.0 Date: April 15, 2012 Page: 5 of 30

#### Figure 4.1 Corporate Organizational Chart (Subject to change)



# 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 quarterly 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 federal organizations. The certification program represents greater than 48 separate state and national certifications. 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.

Section 4.0, Ver. 10.0 Date: April 15, 2012 Page: 7 of 30

#### 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 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 Manual*, including the quality policy.

#### 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 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 Department and is available via the ESC intranet.

#### 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, NELAP, 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.

# 4.3 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 Procedure*.

#### 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 managers and QAO, or designee, prior to issue.

Documents are reviewed at least annually or sooner, as deemed necessary to ensure their contents are suitable, 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 QA 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 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. Pending changes in each revision 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*. Suggested revisions to electronic documents are presented to management for review and approval. Changes to electronic documents can only be made by the QAO, 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 and date 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 QA 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 most recent 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 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 QA Department and Department Manager/Laboratory Director. Department Manager 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 QAO, 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. 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 **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*. On receipt of a request or invitation to tender, 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 SUBCONTRACTING

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.

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.

Section 4.0, Ver. 10.0 Date: April 15, 2012 Page: 15 of 30

#### 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 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 managers are responsible for ordering supplies/chemicals that meet the method 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 analyses produced by the laboratory.

Section 4.0, Ver. 10.0 Date: April 15, 2012 Page: 16 of 30

# 4.7 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 limits, 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 surveys. 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

ESC provides reasonable access, as needed by outside parties, to relevant areas of the lab for witnessing tests.

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 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 QAO (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 Procedure*.

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, retesting is conducted.

4.8.2 Causes and Corrective Actions

The personnel in the quality department examine all documents and records associated with complaints and the department manager 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 **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 QAO, or designee, and documented.

Types of non-conformances are:

- § Deviations from written procedures that were not pre-approved by QA.
- 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 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 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 Non-conforming Work

The correction 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

Section 4.0, Ver. 10.0 Date: April 15, 2012 Page: 19 of 30

• Informing the client (if necessary)

### 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 defendable. The Regulatory Affairs 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 QAO, or designee, and Department Managers administer corrective action approval. It is the Manager'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 QA Department for processing.

#### 4.11.1 General

The initiation, management, tracking, and closure of corrective actions is 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 non-conformities 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.

#### <u>4.11.6</u> Cessation and Restarting of Work

All technical 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:

- Laboratory Manager/Director
- QA Department
- Technical Director/Supervisor
- Technical Service Representative

Technical directors 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 Release of Non-conforming Work

The laboratory allows the release of nonconforming data only with approval on a case-by-case basis by the appropriate Technical Director, or their designee. Planned departures from procedures or policies do not require audits or investigations. Permitted departures for nonconformances, such as QC failures,

Section 4.0, Ver. 10.0 Date: April 15, 2012 Page: 21 of 30

are fully documented and include the reason for the deviation and the impact of the departure on the data.

4.11.8 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 QA/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, quarterly purchased round robin samples, client submitted QC samples and periodic internal blind samples.

4.11.9 Corrective Action Documents

In general, corrective action documents are maintained by the Regulatory Affairs Department. These documents include the following: corrective action resulting from both internal and external audits, corrective action resulting from performance evaluation testing, corrective action as deemed necessary by the QA Department.

Corrective action resulting from analytical failure is kept with the analytical data and is recorded on the bench sheet or raw data. The Department Manager is responsible for making sure that suitable measures have been taken to ensure that the problem is identified and corrected.

Corrective action involving sample receiving is recorded on a Nonconformance form and is then filed with the original Chain of Custody.

# 4.12 **PREVENTIVE ACTIONS**

Preventive Action, rather than corrective action, aims at minimizing or eliminating inferior data quality or other nonconformance 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,

Section 4.0, Ver. 10.0 Date: April 15, 2012 Page: 22 of 30

measures are taken to identify the problem and formulate a plan to implement the defined change needed. The QAO, 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, annual managerial reviews, scheduled column trimming, running a new LIMS system in tandem with the old system to assure at least one working system, 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 a subset of documents, 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.

A documented records procedure SOP# 010103, *Document Control and Distribution Procedure*, and SOP# 020304, *Protection and Transfer of Records*, is 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.

Data Storage Criteria			
Data Type	Storage Criteria		
Manual Data Wet Chemistry	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		
Manual Data Prep Labs	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.		
Manual Data Env. Micro, Mold	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.		
All Data Aquatic Toxicity	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.		
Computerized Data - Organic Dept.	Injection logs are printed and kept in a notebook with the instrument. 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		
Computerized Data – Inorganic Metals Dept.	and kept a minimum of ten years. Original raw data files cannot be edited.All data produced by metals instrumentation is backed up to a secure drive, nightly, utilizing a seven-day rotation cycle. Hard copies are printed and filed by date and instrument. 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.		
Final Report Storage - LIMS	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.		
Final Report Storage - PDF	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.		
Misc. Data Storage	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.		

Section 4.0, Ver. 10.0 Date: April 15, 2012 Page: 25 of 30

4.13.3 Records Disposal

Records that have exceeded the required storage requirement are disposed of through the use of professional records destruction firm. ESC retains the manifest of documents destroyed and files the verification receipt that is generated at the time of destruction.

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 a separate SOP.

# **4.14 AUDITS**

4.14.1 Internal Audits

SOP# 010104, *Internal Audits*, addresses the implementation and maintenance procedure for a comprehensive system of internal audits at planned intervals to verify the on-going effectiveness of the management system.

- 4.14.1.1 The QA 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 are 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.

Section 4.0, Ver. 10.0 Date: April 15, 2012 Page: 26 of 30

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 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/ISO 17025, NELAC, and DOD QSM.

*Method Specific Criteria* – Technique, analytical method, standard operating procedures, and effectiveness are also 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 ethics violations result in a confidential investigation involving only the QAO, or designee, Director of Technical & Regulatory Affairs, 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. Clients are notified promptly when audit findings cast doubt on the validity of the data.

*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.

#### 4.14.2 Performance Audits

Performance audits require evaluation of control and blind results. On a quarterly basis, documentation of results and corrective actions are evaluated as part of the management review process.

## 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.

Study	Frequency	Vendor
WP (Water Pollution)	Semi-annually	Environmental Resource Associates
WS (Water Supply)	Semi-annually	Environmental Resource Associates
Matrix – Soil RCRA	Semi-annually	Environmental Resource Associates
Matrix – UST Soil/Water	Semi-annually	Environmental Resource Associates
Matrix – Air Canisters	Semi-annually	Environmental Resource Associates
DMRQA – Chemistry	Annually	Environmental Resource Associates
DMRQA – Aquatic Tox.	Annually	Environmental Resource Associates
ELLAP	Quarterly	AIHA
IHLAP	Quarterly	AIHA
EMLAP	Quarterly	AIHA
EMLAP – Direct Exam	Quarterly	AIHA
EMLAP – Fungal /	Triannually	AIHA
Bacterial		
Cryptosporidium /	Quarterly	US EPA
Giardia		
Aquatic Toxicity	Annually	North Carolina
Performance Evaluation		

## <u>Annual Studies</u>

- <u>Blind Field Duplicates</u> ESC collects blind duplicates periodically to evaluate field collection and laboratory precision. ESC routinely receives unmarked field duplicates from clients to evaluate sample batches.
- <u>Split Samples</u> 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

ESC agrees to host on-site system audits from external organizations and currently participates in various system and performance 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).

# <u>SDWA</u>

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. ESC participates in WS PE studies in support of drinking water certifications.

# 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. In addition to Water Pollution or Non-Potable water studies, ESC is required to analyze additional blind samples for West Virginia. The laboratory is also periodically audited by the Metropolitan Government of Nashville and Davidson County and certified for wastewater sampling and analysis. ESC participates in WP Studies, DMR QA program, and Solid Matrix PE studies.

# INDUSTRIAL HYGIENE

The American Industrial Hygiene Association routinely audits ESC to maintain certification for analytical support of organic chemical exposure monitoring, microbiological testing and metals exposure activities. ESC currently participates in the required performance testing studies and maintains the quality systems to satisfy the requirements necessary for certification in the following: Environmental Lead (air, soil, paint and wipes), Industrial Hygiene (air filters, diffusive samplers, and sorbent tubes), 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.

ESC is subject to several external audits on an annual basis. The audits cover all disciplines, SDWA, CWA, CAA and RCRA/UST. In addition, the laboratory also participates in additional performance testing, where required by individual clients and for new method development purposes.

# 4.15 MANAGEMENT REVIEW

## 4.15.1 Items in Management Review

Regular management review meetings take place quarterly during the months of January, April, July and October and cover the events from the preceding quarter. The Quality Assurance Officer (QAO), the Laboratory Director, and all Department Managers 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 Director of Technical & Regulatory Affairs and 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*.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 1 of 74

# 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. Approved signatories for support documents and final reports are kept by the Regulatory Affairs Department and, likewise, documents are maintained within each analytical department for the analysts. 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 and Laboratory Safety 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 and Laboratory Safety Plan
- SOPs (As specified/required for work area)

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 2 of 74

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, is 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. The plan is maintained by the Regulatory Affairs Department, which specifies details of the training to be carried out in each department to permit effective implementation of the management system. Managers ensure that the plan is implemented within their areas of responsibility.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 3 of 74

## 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
- 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

## Laboratory Director:

The Laboratory Director is responsible for all operational laboratory activities. The Laboratory Director must approve the *Quality Manual*.

## Laboratory Group Leader, Department Manager:

Day to day supervision of technical laboratory operations 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, corroborating the analysis performed, and approving demonstrations of capability. Additionally they certify that personnel with appropriate educational and/or technical background 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.

# Laboratory OA Officer (Also called QA Manager)

The QAO has the authority and responsibility for ensuring that the quality system is implemented and followed. The QAO has direct access to the Laboratory Director and is independent of operations.

The QAO routinely reviews QA/QC policies for all analyses to ensure that the data is evaluated within method requirements. The QAO is also responsible for assessing data that is out of compliance and ensuring that necessary corrective action measures are taken and are effective.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 4 of 74

# Laboratory OC Manager (Also called QC Officer, QCO)

The QCO shares the authority and responsibility for ensuring that the quality system is implemented and followed. The QCO has direct access to the Laboratory Director and is independent of operations.

The QCO routinely reviews QC policies for all analyses to ensure that the data is evaluated within method requirements. The QCO is also responsible for data review and is responsible for ensuring method/program compliance and that necessary corrective action measures are taken, completed, and remain effective.

# **<u>QC</u>** Specialist (QCS)</u>

Each ESC Analytical Division employs the use of a QC Specialist (QCS). This individual has analytical experience in their assigned area and reports to the QCO. Working knowledge of the instrumentation, printouts, and processes is key to successful approval of data being generated in that area. The QCS gives final approval of the initial raw data. The QCS is responsible for the review of data for method compliance. In addition, the application of qualifiers is verified and approved. If the QCS determines a result to be questionable, the data is given to the Department Manager to initiate appropriate action based on the severity of the problem.

# **Technical Specialist**

Technical Specialists are a part of the Regulatory Affairs Department. These individuals have comprehensive experience in their areas of expertise. The Technical Specialist may be called upon for data interpretation, where compliance issues arise. In addition, these individuals often interface with the clients where questions arise concerning methods, data interpretation, and recommendations concerning alternate analyses.

# 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 completeness and any outstanding anomalies. If an error is suspected, the report is delayed until the appropriate Department Managers can be contacted to resolve the question. Each TSR has laboratory experience in one or more departments.

# LIMS Specialist

The LIMS Specialist tracks internal sample custody, computerizes data, and stores it in the LIMS system.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 5 of 74

## 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 Manager determines specific education and experience requirements for individual positions based on the particular department need.

## 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. Training on new or revised Standard Operating Procedures is maintained with the Master copy of the procedure by the Regulatory Affairs Department.

# 5.3 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 dust, electromagnetic interference, humidity, line voltage, temperature, sound and vibration levels are appropriately controlled for specific measurement results and do not adversely affect accuracy or increase the uncertainty of each measurement.

Environmental conditions are recorded on all data sheets, 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 6 of 74

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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 7 of 74

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. Some areas are periodically monitored to detect and resolve specific contamination and/or 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 be made readily available to personnel (see 4.3).
- 5.4.1.3 Deviation 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 8 of 74

- 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 confirmation 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 covered 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,
    - o 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
    - o 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 9 of 74

#### 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, 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 QAM Appendices.

# <u>Method Detection Limits (MDLs) – 40CFR, Part 136, Appendix B</u> - SOP# 030206, Method Detection Limits

All detection limits 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. The standard deviation of, at least, seven 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:

#### MDL = TS

where: S is the Standard Deviation of replicate measurements and T is the value of Student's T for n-1.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 10 of 74

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).

## **Reporting Limits (RLs)**

Reporting Limits (RLs) are typically set 3 - 10 times the standard deviation calculated in the MDL process listed 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 not set at less than 3 times the MDL. 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.

## ESC – Practical Detection Limit

Where necessary, ESC uses in-house protocol to determine a practical and real number for method detection. This is not a statistically derived number. It is a verified number that is validated using a 20% coefficient of variation. Signal to noise ratios and baseline behaviors are assessed and considered for each instrument type. Instrument performance is assessed based on the lowest possible detectable concentration that is 3X above the noise level. A series of samples are prepared at the determined level, using the method protocol. The samples must perform within a 20% coefficient of variation. The lowest concentration that meets the criteria is the Practical Detection Limit. This determination either confirms or replaces the MDL as determined using 40CFR Part 136.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 11 of 74

5.4.5.5 Demonstration of Capability

## <u>Initial and Continuing Demonstration of Capability (IDOC & CDOC)</u> (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 is recorded on the form found in the NELAP Standard 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*

# <u>IDOC</u>

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:

- A quality control sample is obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.
- 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 laboratorycalculated 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 12 of 74

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 analysis of actual samples may begin. 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.

# <u>CDOC</u>

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)

# <u>IDOC</u>

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 not be sufficient to provide adequate demonstration of performance.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 13 of 74

# <u>CDOC</u>

Annual demonstrations are performed by Analysts/Technicians involved in Lead (Pb) analyses to showed 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 runs.

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 on conformance are based

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 14 of 74

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, where applicable, and is performed using a population of 50 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 matrix. Data set must be 50 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 are considered in this determination.

## 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 15 of 74

## 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 functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of data.

## 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

Table 5.4.7.4a LIMS		
System	Description	
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 and QA. 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	• Login summary - includes all information on sample and requested analyses	
Records	• Lab preparation preview and benchsheets for digestions, extractions	
	• Lab assignment/benchsheets to generate work assignments and record data	
	Data approval reports	
	Final reports for clients	
	QA summary	
Hardcopy	All paper records are retained by ESC. As the pages become historical (prior to	
Records	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. The Lab Support Supervisor maintains a log of numbered boxes	
	and their contents. They are cross-referenced by sample log number, date and	

Table 5.4.7.4a LIMS			
System	Description		
	storage number.		
Data	Data is available on electronic media. Revisions to the LIMS software are		
Records	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.		
Manual	• The section supervisor first approves raw data.		
Data Entry	• The data entry portion of the LIMS can only be accessed by authorized		
(verified by	individuals, therefore allowing limited access to protect the integrity and		
4-step	maintain the confidentiality of the data.		
system)	• The data entry person and a qualified laboratory analyst then proofread each		
	group of entered data.		
	• When all results for a sample are complete, a report is printed and examined by		
	a Technical Service Representative for final approval.		
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 is transferred electronically from instrumentation directly to the LIMS. Once		
Data	the data has been transferred, it undergoes a screen review. The data is then		
Transfer	printed and reviewed again. If data needs to be changed, a data entry specialist		
	changes it and a hardcopy is printed of the final data.		

Table 5.4.7.4b AUXILIARY SOFTWARE			
System	Description		
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 group leader. The lab 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 group leader.		
Supplier Info All purchased software that is used in conjunction with software specific instruments is guaranteed by the supplier to function as required. The sup 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 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 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 NELAP 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 NELAP 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).

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 18 of 74

- 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 Department Manager 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 Manager 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 19 of 74

Table 5.5.3.3a General Equipment Calibration			
Equipment	Activity	Frequency	Record Type
Balances	Verified with Class I NIST traceable weights when used	Before use	Logbook – Located in each respective lab
Balances	<ul> <li>Clean</li> <li>Check alignment</li> <li>Service Contract</li> <li>Top-loading balances are allowed a tolerance of ±1%, while analytical balances are allowed a tolerance of ±0.1%.</li> </ul>	Semi-annually under a service contract.	Certificates from contractor.
Weights – Class I	<ul> <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, annually, or sooner if necessary.	Certificates from contractor.
pH meters and probes	<ul> <li>Calibration:</li> <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.</li> </ul>	Before use	Calibrations are recorded in a logbook.
Automatic pipettes	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.
Refrigerators, Freezers, Hot plates and BOD incubators	<ul> <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> </ul>	Temperatures are recorded each day in use	Logbook
Ovens	<ul> <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

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 20 of 74

Table 5.5.3.3a General Equipment Calibration			
Equipment Activity		Frequency Record Type	
	ESC NIST-certified thermometers	Calibrated annually by a NIST calibration service, accredited to	Calibration certificates from the calibration service.
Thermometers	All working thermometers	ISO/IEC 17025 and ANSI/NCSL Z540-1.	"Accuracy Assurance Program Test Data Sheets" provided by the servicer. All
		Verified semi- annually against NIST-certified thermometers by an outside	thermometers are tagged with current tolerances. Internal daily
DO Meter	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.
Specific Conductivity Meter	<ul> <li>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.</li> <li>Biomonitoring, potassium chloride with a conductivity value of 100 and 1000 µmhos /cm is used as the calibration standard.</li> <li>Wet Lab, potassium chloride with a value of 1413 µmhos/cm is purchased from NSI for calibration purposes.</li> </ul>	Before use	Calibration of each meter is recorded in separate daily logbooks.
Fume Hoods	Check semi-annually and must meet the OSHA minimum recommended face velocity of 60 – 100 fpm.	Semi-annually	Recorded in Logbook

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 21 of 74

	Table 5.5.3.3b Class 1 Weight Tolerance			
Value	ASTM Class 1 Tolerance	Unit	ASTM Class 1 Tolerance	Unit
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.

Tε	able 5.5.6 - GENERAL PREVENTATIVE MAINTENANCE
Туре	Description
Glassware	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 cont.
Logbooks	<ul> <li>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. All preventive maintenance is noted either in the maintenance logbook or in the runlog notebook.</li> <li>At a minimum, all maintenance logs contain the following: <ul> <li>All entries in the maintenance logs must be initialed and dated by the person performing the maintenance.</li> <li>All maintenance logs must be bound and paginated.</li> <li>All pages of the maintenance logs must have "ESC" at the top of page.</li> <li>The instrument ID number or serial number.</li> <li>Make and model of the instrument.</li> <li>Date of installation or the date the instrument was put in service (if available).</li> <li>Condition of the instrument when installed (new or used)</li> <li>A unique number for each notebook</li> </ul> </li> </ul>
Service Records	Maintenance that requires a service call from the vendor should contain the following:
	<ul> <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> <li>To verify that the instrument is running properly after service has been performed, recalibrate and analyze QC samples before the service engineer leaves.</li> </ul>
Additional Records – Misc. Monitoring	<ul> <li>Additional records are kept, updated and signed when technicians are assigned to perform the following tasks:</li> <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> </ul>
	• 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.

#### 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 Manager, 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.

5.5.10 Calibration Checks

Analytical instruments are calibrated per method requirements. Balances and temperature-indicating devices are verified semiannually. Records are maintained as quality assurance documents.

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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 24 of 74

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# 030202, *Receipt and Records of Stock, Intermediate, and Working Standards)*
- 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 25 of 74

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 for the equipment.
- 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 Laboratory Director 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 26 of 74

## Standard/Reagent Preparation

When standards are prepared in-house, they are weighed on an analytical balance, calibrated against Class "I" 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 with each analysis book, 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. Where appropriate, an electronic LIMS Standard Logger is used in lieu of handwritten logbooks.

# 5.7 SAMPLING

## 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 27 of 74

## 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. All field information can be reviewed in the field notebook by date and client.

## 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 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.)

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 28 of 74

- 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:
  - Ø Well casing composition and diameter
  - Ø Water table depth
  - Ø Well depth
  - Ø Calculations to determine the volume of water to be purged
  - Ø The total volume of water purged and how accomplished
  - Ø The date and time well was purged, beginning to end
  - Ø 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:
  - Ø Collection beginning and ending time and date
  - Ø Specific equipment used (manual or automatic)
  - Ø Abnormal conditions of the sampling location
  - Ø 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 29 of 74

## 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. 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 shown in Table 5.7.5. Equipment blanks are prepared by rinsing the equipment with analyte-free water 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 30 of 74

## 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 <u>not</u> 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.

# TABLE 5.7.5.2 EQUIPMENT BLANK COLLECTION PROCEDUREFOR EACH TYPE OF SAMPLING EQUIPMENT

No. of Samples	Precleaned Equipment Blank Per Parameter Group Prior to Sample Collection	Field-Cleaned Equipment Blanks Per Parameter Group		
Less than 10	1 equipment blank if no field cleaning on site; OR	1 equipment blank for field- cleaned equipment		
Greater than 10	1, or 5% of equipment sets, whichever is greater	1, or 5% of equipment sets cleaned, whichever is greater		

## **NOTE:** Equipment blanks must accompany samples in the same container used for transportation.

## 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 SAMPLE MANAGEMENT

5.8.1 Sample Management Instructions

Clients supply environmental samples from various sources/programs for analysis. ESC utilizes method SOPs and contract requirements as the instructions to properly handle and process these samples.

## 5.8.1.1 Holding Time Verification

- The Login Technicians are trained to recognize analyses with immediate, 24-hour, and 48-hour holding times. 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 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 32 of 74

5.8.1.2 Sample container and Sub-Sampling

- Each container displays the following information once it has been released from sample login to the laboratory: the original sample container label and the sample login label showing the sample log number.
- 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.
- When a sample is obtained for analysis the chemist records in the appropriate prep book or benchsheet the log number, the date removed, his initials, and the volume or mass of sample removed.
- 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*.

## 5.8.1.3 Sample 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.

- ESC currently maintains the following prep information: wet chemistry, metal digestions, organic extractions (by method), and GC and GC/MS injection logs.
- The chemist 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 prep book 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: date prepped, amount prepared (volume/weight), dilutions, etc.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 33 of 74

• The various prep books, workbooks, and injection logs document every manipulation of the sample through receipt, preparation, and analysis.

5.8.1.4 Analysis & Analysts

- 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 benchsheet. 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 benchsheet.
- The benchsheet 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, a QC Specialist, 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.
- For data that cannot be transferred electronically, a Data Entry Specialist enters the results into the LIMS. The entered results are reviewed for transcription errors against the original worksheet by a chemist. If the lab supervisor or senior chemist rejects the work, he discusses the corrective action measures with the analyst.
- 5.8.1.5 Laboratory Documentation
  - Laboratory notebooks and related documentation are an essential part of the analytical procedure. Every detail concerning the sample analysis must be documented.
  - All documentation must be written with permanent/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 date and initials of the person making the change. The correct result is clearly recorded adjacent to the incorrect result.

5.8.1.6 Sample Storage and Transportation

- When a Chemist completes the preparation or analysis of a sample, he returns the sample container to the Sample Custodian.
- Samples transported under the responsibility of the laboratory are done so safely and according to storage conditions.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 34 of 74

• Specific safety operations are addressed outside of this document.

## 5.8.1.7 Final Reporting

- When all analyses on a sample number have been completed, the LIMS prints the final report.
- The TSR reviews the final report for discrepancies. If discrepancies are found, re-analysis may be requested.
- The TSR gives the final approval on the report and indicates approval by signature.
- Routinely, data reports are transmitted to the client through email as a PDF file. Reports are sent as PDFs to prevent alteration of the document. The hardcopy report can be mailed to the client, when necessary. Reports may also be sent to the client by fax, or via secure access through the ESC website.
- Reports that are sent electronically are protected using the latest technology available to protect the confidentiality of the results and the client.

## 5.8.1.8 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 400 degrees Fahrenheit for two minutes and disposed of in a commercial waste container.
- All other waste is disposed of according to Section 6.

## 5.8.1.9 Sample Subcontracting

- When samples are transferred to subcontracted facility, a COC accompanies the samples. The COC contains the following required information: collection date and time, ESC login ID number, quantity and type of container, date of sample collection, 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 35 of 74

## 5.8.2 Sample Information and Labeling

A unique sample identification number is generated for each sample and is used throughout the analytical and disposal cycle. A record of all client-supplied samples is established and maintained. The samples are stored according to published method requirements and determinative SOP.. While in storage, the client 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.
- 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

Any sample supplied by the client is verified upon receipt as meeting its description and being free from damage. In the event of a client 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 36 of 74

The Login Technician is 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 preservation at 4°C must arrive at the laboratory above freezing but  $\leq 6^{\circ}$ C. If the samples are not appropriately preserved, the problem is noted on a sample nonconformance form, the sampler 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. If extractions are necessary, the laboratory supervisor is notified, via daily management reports, to ensure that holding times are not exceeded for samples, extracts, or digestates.

#### 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 placed onto 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
- 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 filled out 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 though he has 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 Handling

Client samples remain in their original packaging until analysis. Any samples that need to be dispensed or removed from their original packaging are stored in conditions that provide the same degree of protection.

Sample/Extract Storage:

- Samples, extracts, distillates and digestates have specific storage locations arranged in log number order unless rush analysis is required.
- Access to these areas is limited to authorized personnel.
- Samples are stored either in the cooler or in ambient-temperature storage, according to method preservation requirements

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 38 of 74

- Extracts, digestates, and standards are stored separately from calibration and other QC Standards in dedicated areas as follows:
  - Organic extractions for pesticides and PCBs are stored in glass vials in a designated refrigerator in the SVOC GC lab.
  - Organic extractions for SVOCs are stored in glass vials in a designated refrigerator in the semivolatile GC/MS lab.
  - TCLP extracts for metals only and metal digestates are stored in the metals lab.
  - TCLP extracts for SVOCs, pesticide, and herbicide analysis are stored on designated sample shelves in the cooler. After the extraction, the extract is stored in a designated refrigerator in the semivolatile GC/MS lab.
  - Zero headspace extracts and samples for volatiles are stored in VOC vials and segregated in a designated cooler. Where necessary, samples collected by Method 5035 are frozen.
  - Volatile standards are stored in a designated freezer in the VOC lab.
  - Pesticide and PCB standards are stored in a designated refrigerator in the SVOC GC lab.
  - SVOC standards are stored in a designated freezer in the SVOC GC/MS lab.
- 5.8.5 Special Requirements

The following entities mandate any required needs for special handling, storage, packaging, preservation, shipping, and marking provisions:

- EPA Approved Methods 29 CFR (OSHA) IATA (Dangerous Goods)
- 40CFR Part 136.3 •
- 49 CFR (DOT)
- 5.8.6 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 client 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.

## 5.8.7 Sample Custody

## Chain of Custody

An important part of any sampling/analytical plan is ensuring sample integrity from collection to data reporting. Figure 5.8.7a 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 log sheets, sample extraction and digestion prep books, analytical workbooks and instrument logs, QC data associated with the sample set, and the final report. Examples of these documents are presented in Figures 5.8.7b through 5.8.7k.

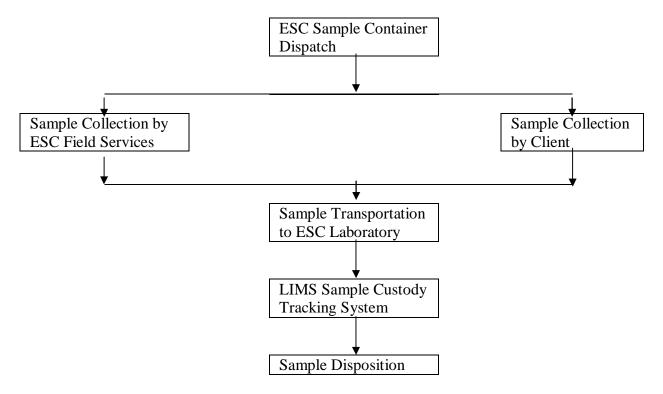
## Legal Chain of Custody

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 vender as certified clean per EPA protocols. When a kit is prepared for delivery to the field a Shipping Batch Detail Report is filled out 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 that is sealed with the COC/Shipping BDR inside by the person involved with 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 40 of 74

## FIGURE 5.8.7a CHAIN OF CUSTODY PROCESS



Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 41 of 74

#### FIGURE 5.8.7b INDIVIDUAL CONTAINER LOG EXAMPLE (Contents varies depending on client kit requirements)

12065 Lebanon Rd. Mt. Juliar, TM 37122 (415) 758-5818 1-900-767-5950 Fax (415) 758-5859 ENVIRONMENTAL SCIENCE CORP. Tax I.D. 62-0814289 Egt. 1970 Shipping Batch Detail Report Batch ID: Dates 04/18/07 Active: Y Client: ATHEUS TER: Claudia G. Himeman Athens UE-Fretreatment Program Order# Frequency TYDe Description Due Dt <u>#Kit Templat</u>e P207532 As Needed Standing 05/18/07 N 1 T40592 Proj.Desc.: ESC Key : ATHE03-CRYPTO Project No: CRYPTO-FS Site ID: Comments: Please include LT2 paper work with order Client ID: Sample No: P207532-01 Packing List: Analysis Required QTY Container/Preservative cryptosporidium 1 10LCarboy Total Cntrs: 1 Outbound Method of Shipment FedEX Ground Return Method of Shipment FedEX Priority Paid By Client Carrier: Shipping Audit Trail Date Shipped: # Pieces: Color: Cooler: Size: Initials:

Ship To:

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 42 of 74

## FIGURE 5.8.7c CHAIN OF CUSTODY GENERAL EXAMPLE (Required Analysis is printed by ESC or Client)

		1	demate biling	information:			A	<u>y</u> nalys	is/Co	ntainer	r/Pres	ervati	<u>/8</u>	c	hain of Custody	٦.
Emerald Manufactu	iring														age of	
12065 Lebanon Road Mount Juliet,TN 37122														Prepared by:	ONMENTAL	
Report to: Mr. Tom White			Emai:	hite@envsi	ni aom		開閉							SCIEN	CE CORP.	
Project			City/St	ne	alcom		183		11a	SV8310	1.0				onnon Road TN 37122	
Description: Demo report	1.6		Collect				慶	-83		S					0) 767-5859	
Phone: (615) 758-5858	Cient Project #		Lab Project #				Metals 250mlHDPE-HN03	250mlAmb-No Pres	p	s			業重		15) 758-5859	
FAX: (615) 758-5859 Collected by (print):	UPPB Site/Facility IDA		P.O.:	MERALD-U			+5	Ž	SV8270 IL-Amb-NoPres	IL-Amb NoPres	Ð	-	æ			
Palitated by Jalassinsky	12345		99999999999				- G	Am	1-90	A da	l-du	AMB	AMB	Acctnum: EMERALD (lab use only) Template/Prelogin T33311/ P158345		8
Collected by (signature): Rush? ( Lab MUST Same Day							HIM	J	-An	Ψ	lAr		13.317			
4				Email?	No X_Yes	No.	250	125	E	H	6	E	Ξ	Cooler #	3511 1 138343	
Packed on Ice N Y	Two Day			60% EAX? No Yes		of	tals	SV8081	827	SVOCS	V8260 40mlAmb-HCI	SV8141	SV8151 IL	Shipped Via: FedEX Ground		
Sample ID	Comp/Grab	Matrix*	Depth	Date	Time	0.08	Me	SV	SV	SV	V8	SVS	SV	Remarks/Contaminant	Sample # (lab only)	]
MW-1		GW				6	X	X	X	X	X	х	X		1219795-0	01
MW-2		GW				6	X	X	X	X	X	Х	X		0	<u>ي</u> کر
MW-3		GW				6	X	X	X	X	X	X	X		-0	
MW-4		GW				6	X	x	X	X	X	X	X		-6	Y
						-	235	-		-			10.1		all see the statement of	
				_			133		6.5	-		-		-		
					-		12161		- 21		1.2				Rice Sciences	
						1			131		1.25				用的数量量量	2.6
"Matrix SS-Soil GW-Groundwater WW	- WesteWater	W - Drinking V	Neler OT - Ot	har										Town		-
Remarks:												p	n	Temp		
Invitients.												FI	ow	Other	and the transmission of "	
					Δ.											

			10	
Relinquished by: (Signature)	Date:	Time:	Received by (Signature)	Samples setufned via: LI UPS Condition: (lab use only)
			and	B FedEx Courier
Reinquished by (Signature)	Date:	Time:	Received by: (Signature)	Temp Jan Bottles Received:
				Ye C
Reinquished by (Signature)	Date:	Time:	Received for lab by: (Signature)	Date:// Time: pH Checked: NCF;
				15/25

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 43 of 74

#### FIGURE 5.8.7d

#### SAMPLE CONTAINER LABEL

## ABC WASTEWATER PLANT

Prepared by Environmental Science Corp.

Project: Annual Sludge - SOUR/Class "B" Fecal

Proj #:<u>57243</u>

Sample Location/ID: Sludge Digester

Analysis Req'd: Class "B" Fecal Coliform

NaThio Preservative Included

Date:\_\_\_\_\_ Time:\_\_\_\_\_

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 44 of 74

#### FIGURE 5.8.7e

#### SAMPLE CONTAINER CUSTODY SEAL

CUSTODY SEAL
--------------

Date:----

## I-CHEM

**Chemists In The Container Business**<sup>TM</sup>

Signature:

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 45 of 74

## FIGURE 5.8.7f

## SAMPLE LOGIN LABEL

Sanple #1 SV625	1L=Anb- NoPres <b>9999999</b>	<b>L99999</b> - 01
Coll. Date/Time:		<b>EN</b>
EMERMFG Enerald Manufactu Outfall Manhole-g	L99999-01 ring Corp. parterly	"BARCODE HERE"

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 46 of 74

#### FIGURE 5.8.7g

#### **EXAMPLE LAB PREPARATION SHEET**

## ENVIRONMENTAL. SCIENCE CORP. Laboratory Sample Prep Sheet Date Created: 4/13/2007 Analyst: 196 Metind: Tig Matrix: Solid Samples

		Bam	pies		
Account	Sample Name	Method	Weight(g)	Volume(mL)	Sample Description
	L288458-01	7471A	0.58	30	Brewn slodge
	L288518-01	7471A	0.58	30	Brewn clay
	L288519 01	747LA	0.60	30	Brown clay
	L288868 10	7471A	0.58	30	dark brown clay
	L288920 08	7171A	0.55	90	Pingle palit.
	1.268435-01	2471A	Fa-L	30	lkown a ay
	1.26%633-02	2471A	I-n?	30	Ikown clay
	T.098935-05	24712	6.57	<u>10</u>	Brianciay
	L05893S-04	7471A	6.63	50	Boownelay
	L288439 05	7.71.2	6:03	30	Brown clay
	L288965-01	7.71A	0.55	30	Bisoz studze
	L285795-05	74712	1:52	90	Boo wa sedencal, seess
	L(26x497.00	2:71A	1-57	30	Macy sectioner process
	L2Ex497 21	2471A	3557	30	Daty brown solutiont, rec-
	L069003-01	7471A	0.59	30	Boown shidge
	1.159030-01	347.1 A	6.61	50	Geey pluy
	1.259232-03	747]A	0.59	30	Gery day
	L289075-01	7.71A	0.00	30	Brown sand, reesa
	LLE9075-02	7471A	0.60	30	stad, speks
	1.259095-01	2171A	1-55	30	Mulacolored, recks

## QC Samples

Black	BLKW-G255550	71714	6.66	30	Brown sand
328	13 899 (295555	2471A	3546	30	Bown sol-
200	L259375-01DDP	7-71A	l el	30	Roown sand, rooks
VS.	L259075-02MS	2/71A	1 el·	30	Roown sand, news
VSD	LC5907S-0EMSD	7471A	0.60	30	Booten smill, reside

F13-2007 11:06 SI-PM

1 ef 1

#### FIGURE 5.8.7h

## **EXAMPLE LAB ASSIGNMENT/WORKSHEETS**

ENVIRONMENTAL SCIENCE CORP.	Laboratory Bench Sheet	
Date Created: 4/2/2007 Analyst: 156	TOTAL PHENOL BY 4AAP	Workgroup: WG293708 Calibration Date: 03/15/07
Method: 4AAP	Instrument: Lachat5	Calib. Corr.: 0.999990
Matrix: Water		Units: mg/I
Prep Date: 4/2/2007	PropStart: 11:20 PM PropEnd: 1:00 PM	
	Reagents	
Reagent Name	Standard Number	Expiration Date
1 AAP	7D02049	04/03/07
	7C28009	04/04/07

#### Samples

Sample Name	Workgroup	Results	Dilution	Report Value	Qualifiers	
L285470-02	WG293700	0.076	1	0.076**		
1.285297-02	WG293700	0.0208	1	40.04		
L286321-02	WG293700	-0.0079	1	<0C.C4		
L286335-02	WG293700	0.0274	1	<0.04		
1.286401-02	WG293700	0.0076	1	<0.04		
L286618-02	WG293700	0.0125	1	<0.04		
L286703-01	WG293700	0.042	1	0.042**		
L286703 02	WG293700	0.005	1	<0.04		
L286788-01	WG293700	0.0066	1	~00.04		
L286788-02	WG293700	0.0087	1	<0.04		
L286788-03	WG293700	0.0128	1	<0.04		
L286807-01	WG293700	0.162	1	0.162**		
L286807-02	WG293700	1.16	1	1.16**		
L286807-03	WG293700	0.069	1	0.069**		
L286807-04	WG293700	0.149	1	0.149**		

4/2/2007 5:56:34 PM

## FIGURE 5.8.7i EXAMPLE SAMPLE CONFIRMATION REPORT

Environmental Science Corp.

					Apr Login Number:	Confirmation Rep 17 2007, 06:17 p L3547 Template CALD Emerald Man	n Number: N/A					
leport To: Ton White : 12065 Leb : : Hount Jul: telephone #: 615-758- Tax #: -758-5859 mail: twhite@envaci roject/Account Comm	enon Ro iet, TN, -5858 .com;ton	37122		Project Des POS: 1234 POS Require Lab Project								
ab Sample # Test 3547-01 W P AP1	Sample MW-1	ID Desc. Appendix I I	01-Nov-04	ate & Time , 12:00	Collected By Tom White	Site TN56383752	Receive Date 02-NOV-04	PR QR	Est.DueDate(1 09-NOV-04	) Method	\$	Unit Price
C         AGICP           C         BAICP           R         C		Silver Barium Beryllium Cobalt Cobalt Chromium Copper Mercury Nickel Lead Selenium EDS/DDCP Thallium by App I Volati Zinc	ICIMS		4625010 4625010 4625010 4625010 4625010 4625010 4625010 4625010 4625010 4625010 4625010 4625011 4625011 4625011 4625011 4625011	250n1HDFE-HBO 250n1HDFE-HBO 250n1HDFE-HBO 250n1HDFE-HBO 250n1HDFE-HBO 250n1HDFE-HBO 250n1HDFE-HBO 250n1HDFE-HBO 250n1HDFE-HBO 250n1HDFE-HBO 40m1Am5-HC1 250n1HDFE-HBO 40m1Am5-HC1 250n1HDFE-HBO	3 DEFRULT 3 DEFRULT			60108 60108 60108 60108 60108 60108 60108 60108 60108 60108 60108 60108 60118 60208 60108 60108		0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0
547-02 oundwater project	HW-2		01-Nov-04	, 12:00	Ton White	TN56383752	02-NOV-04	QR	09-NOV-04			
<ul> <li>P AP1</li> <li>C AGICP</li> <li>C BAICP</li> <li>C BAICP</li> <li>C CAICP</li> <li>C CDICP</li> <li>C CAICP</li> <li>C CAICP</li> <li>C CAICP</li> <li>C CAICP</li> <li>C FBICP</li> <li>C SV8011</li> <li>C TAICP</li> <li>C V9250AP1</li> <li>C V9250AP1</li> <li>C XICP</li> </ul>		Appendix I I Silver Barium Beryllium Cobalt Chronium Copper Nercury Nickel Lead Selenium EC0/DBCP Thallum by App I Volati Zinc	ICIMS				DE FAULT DE FAULT		1 Hottles 1 Hottles 1 Hottles 1 Hottles 1 Hottles 1 Hottles 1 Hottles 1 Hottles 1 Hottles 2 Hottles 2 Hottles 2 Hottles 1 Hottles	60108 60108 60108 60108 60108 60108 60108 60108 60108 60108 60108 80108 8011 6020 82608	***	250.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0
3547=03	NW-3		AL 11 AL	, 12:00	Ton White	TN56383752	02-NOV-04	OR	09-NOV-04			

Entered 28-JUN-02 By SEEDPAK

Page 1 of 2

(1) Due Date listed is an estimate based on average workloads. Please communicate required due dates to your TSR.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 49 of 74

## 5.9 QUALITY 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.

5.9.2 Quality Control Activities

Monitoring of quality may include the following:

- regular use of certified reference materials and/or internal quality control using secondary reference materials;
- participation in interlaboratory comparison or proficiency testing programs;
- replicate analyses
- re-testing or re-calibration
- logic check or correlation of results from related analyses
- 5.9.2.1 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.

## 5.9.2.2 Laboratory Checks

See Section 3 for a description of QC samples and related definitions.

Table 5.9.2.2 BASIC LABORATORY QC CHECKS								
QC Check Sample	Source	Prep Required						
Method/reagent blanks - One blank is carried through each step of the analytical procedure for each batch of samples. Blanks are prepared for each preparation method and matrix (i.e., solids assay, dissolved metals, TCLP extraction, etc.). Blanks are used to confirm the absence of contaminants within the preparation and/or analytical system prior to and during the analysis of field samples.	Lab DI	Yes						
<b>Initial Calibration Verification (ICV)</b> – An independently prepared standard used to verify the accuracy of the initial calibration (for ongoing calibration)	Primary or Secondary	No *						
<b>Laboratory Control Sample (LCS)</b> – A known clean matrix is spiked with known amounts of the analyte(s) of interest used to verify the efficiency of the analytical system without interference from the field sample matrix. The LCS provides the best estimate of analytical system performance and may also be used to verify the validity of the on-going calibration.	Secondary	Yes						
<b>Continuing Reference Standard Checks</b> – Metals and Organics; *Also called SSCV (Secondary Source Calibration Verification) – An independently prepared standard used to verify the accuracy of the existing calibration.	Secondary	No						
<b>Continuing Calibration Verification (CCV)</b> - A standard, usually near the midpoint of the calibration curve, made from the primary or same standard stock used for the calibration curve. The CCV is used to represent the ongoing calibration stability of the instrument and must perform within method stated criteria.	Primary	No *						
<b>Sample Matrix Spikes and Spike Duplicates (MS/MSD)</b> –Prepared field samples spiked with known quantities of target analyte and carried through the entire preparation and analytical process concurrently with unspiked field samples to assess the effect of the sample matrix on the target analytes present and to provide an estimate of analytical precision. For analyses where field sample type does not allow for MS/MSD preparation (i.e. lead wipes, air samples on charcoal tubes, etc.) an LCS/LCSD pair may be substituted.	Primary or Secondary	Yes						
<b>Post Digestion Spike</b> – (used in metals analysis) A standard prepared from a previously analyzed spiked sample digestate that yielded reduced recovery for the target analyte due to a suspected matrix interferent.	Primary	No						
<b>Sample Duplicates</b> – Second aliquots of field samples carried through the entire preparation and analytical process that used as an indication of sample precision or consistency in the field sample matrix.	Client Sample	Yes						
<b>Surrogate Standards</b> – Analytes not expected to occur naturally in field samples that are spiked by preparation/analytical personnel to assess sample preparation and analytical efficiency in each individual field sample.	NA	Yes						
<b>Internal Standards</b> – Analytes not expected to occur naturally in field samples that are spiked to provide a consistent basis for comparison with target analyte concentrations. ISTDs are used in internal calibration models.	NA	No						

\* Preparation requirements can vary depending on method. Requirements are listed in each individual determinative SOP.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 51 of 74

#### 5.9.2.3 Batch QC Criteria

#### 5.9.2.3.1 Environmental Samples

Sample Batch - Defined as a set of 20 or fewer samples of a similar matrix prepared and/or analyzed concurrently. The maximum number of samples possible per batch is dependent on the determinative method allowance.

Required Instrument QC per batch:

- Calibration Blank (CB or CCB)
- Initial Calibration Verification (ICV)
- (1) Continuing Calibration Verification (CCV) every 10-20 samples where and as required.
- (1) CCV at end of run where required.
- (1) Post-Digestion Spike Metals analysis
- (1) Serial Dilution Metals analysis
- **NOTE:** The CCV is typically a mid-point concentration. In addition to the mid-point, where required, the CCV is run at a concentration that varies from the mid-point by +/-25% during each analytical run. The varied CCV must meet the same acceptance criteria as the mid-point.

Required Method QC per batch (Must include internal standards and surrogates, where required by the method):

- (1)Method/prep Blank
- (1) Laboratory Control Sample Duplicate Pair, LCS/LCSD must be analyzed for analytes where spiking procedures are not practical, such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, oil& grease, temperature, dissolved oxygen or turbidity
- Matrix Spike/Spike Duplicate (MS/MSD) Pair, MS/MSD must be analyzed except for analytes where spiking procedures are not practical, such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, oil& grease, temperature, dissolved oxygen or turbidity
- (1) Sample Duplicate (where sufficient field sample is available and where required by determinative method)

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 52 of 74

5.9.2.3.2 Industrial Hygiene Analyses, Including Environmental Lead

Sample Batch - Defined as a set of 20 or fewer samples of a similar matrix prepared and/or analyzed concurrently.

Required Instrument QC per batch:

- Calibration Blank (CB or CCB)
- Initial Calibration Verification (ICV)
- (1) Continuing Calibration Verification (CCV) every 10 samples
- (1) CCV at end of run.
- (1) Post-Digestion Spike Metals analysis

**NOTE:** The CCV is typically a mid-point concentration. In addition to the mid-point, the CCV is run at a concentration that varies from the mid-point by +/- 25% during each analytical run. The varied CCV must meet the same acceptance criteria as the mid-point.

Required Method QC per batch:

- (1) method/prep blank
- (1)Laboratory Control Sample/Laboratory Control Sample Duplicate Pair, LCS/LCSD
- Matrix Spike/Spike Duplicate (MS/MSD) pair, where matrix permits
- (1) Sample Duplicate (where sufficient sample is available)

5.9.2.3.3 Batch QC Protocols

If more stringent QC protocols are required than those outlined above for any method or project, then the more stringent method protocols are followed.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 53 of 74

#### 5.9.2.4 Inter-Laboratory Quality Control

- Reference samples are ordered from Environmental Resource Associates or similar provider. Samples are purchased to evaluate the following method types: Air, Water Supply, Water Pollution, and Solid Waste.
- Blind QC check samples are purchased at least semi-annually from Environmental Resource Associates or similar provider as an external source for performance evaluation samples. These samples are supplied to ESC without the true concentration values. For specific state water pollution programs, two levels are analyzed. The laboratory may perform additional studies as required by contract, regulatory agency or accreditation. ESC reviews the results as an overall check on internal QC procedures. If blind QC check sample results are unacceptable and such information impacts certification the laboratory immediately initiates corrective action and orders another check sample to ensure ongoing proficiency of that analyte.
- Blind field duplicates are collected at least annually to evaluate field collection and laboratory precision. Client field duplicates are collected based on project requirements. The field duplicates are logged in as regular samples and laboratory personnel are unaware of sample origin.
- Split samples are periodically sent to outside laboratories to confirm analytical results.

#### 5.9.2.5 Procedures for Assessing Data Precision, Accuracy and Completeness

The following procedures apply to all analytes measured, unless more stringent QC has been specified. All field measurements must meet the same QC criteria as those run in the lab.

#### 5.9.2.6 Use and Preparation of QC Samples

Certified standards, generated from reference materials, are used to check calibration throughout the analytical run. The standards are obtained from suppliers who are NIST recognized and ISO compliant. A Certificate of Analysis or other documentation verifying purity accompanies the standards.

Sample matrix spikes are prepared using actual samples prior to digestion, extraction, etc. Separate matrix spike limits are calculated for each type of sample (i.e., water, solid, TCLP extract, personnel filter, etc.). Sample duplicate analyses are also initiated prior to digestion, extraction, etc. Duplicate spikes and duplicate laboratory control samples are used to generate precision data.

TABLE 5.9.2.6         METHODS USED TO GENERATE PRECISION									
AND ACCURACY TARGETS									
Method	Purpose	Method References							
Reference Standards (Laboratory Control Sample - LCS)	Accuracy	All analyses							
Reference Standards (Dup. Laboratory Control Sample – LCSD)	Precision and Accuracy	All analyses							
Matrix Spikes	Accuracy	All quantitative Wet Chemistry analyses. All Metals and Organics.							
Duplicate Matrix Spikes	Precision and Accuracy	All quantitative Wet Chemistry analyses. All Metals and Organics.							
Sample Duplicates	Precision	All analyses							

#### Table 5.9.2 lists methods used to generate precision and accuracy targets.

#### 5.9.2.7 QC Charts

When an analyst completes a reference standard check, a duplicate, or a matrix spike, the result is calculated and compared to the appropriate QC chart and evaluated against the established limits. A rough x-bar or duplicate QC graph, with mean, warning and control limits, is available. If the results are out of control limits, the analyst notes this problem for appropriate corrective action. Corrective action is taken, based on an established list of identified corrective action procedures.

#### Outliers

Control limits are calculated at least annually, where required. The data are evaluated using  $\pm 4$  times the standard deviation or  $4\sigma$  criteria for outliers. Data that falls outside of  $\pm 4$  times the standard deviation are eliminated from the calculation. Data points are not eliminated otherwise, unless an obvious system failure has occurred and the error can be documented and identified.

#### Control Data Entry

For non-data transfer results, the data entry specialist gathers data directly from the benchsheet and enters it into the computer LIMS or Excel, depending on the origin of the data. For instrumentation with data transfer, the data is obtained directly from LIMS. The data is then brought into a ESC Lab Sciences Quality Assurance Manual *Technical Requirements*  Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 55 of 74

spreadsheet and the charts can be plotted and evaluated by the computer software.

5.9.2.8 Accuracy

Laboratory Control Standards (LCS)

- Laboratory Control Standards are run with every analytical batch.
- X-bar control charts are generated using a minimum of the last 20 data points, based upon percent recovery.
- Warning limits are set at the 95% confidence interval and are plus/minus two standard deviations from the arithmetic mean.
- Control limits are set at the 99% confidence interval and are plus/minus three standard deviations.
- LCS limits are calculated at least annually where necessary. See the individual laboratory appendices for the list of established limits. Method stated limits override in-house calculated limits.

Percent Recovery:

Percent Recovery = 
$$\frac{Observed Concentrat ion}{True Concentrat ion} \times 100$$

Standard Deviation for Percent Recovery:

$$Sp = \sqrt{\frac{(P_1^2 + P_2^2 + P_3^2 + ...)(P_1 + P_2 + P_3 + ...)^2}{num \ of \ entries}}{num \ of \ entries - 1}}$$

Where: Sp = Standard deviation for percent recovery  $P_{1,2,3,..} =$  Percent recovery results

#### Matrix Spiked Samples

Spiked samples are typically ten percent of all samples, where matrix and sampling permits. Spiked samples are entered onto similar QC charts with the percent recovery. The target spike concentration routinely used is one to five times the initial concentration of the unspiked sample. This basis for the spike target provides analyte concentrations that do not exceed the range of the analysis and are not too small to be significantly affected by normal data variability. One exception for higher ratios is if an MS is spiked at one to five times the client sample concentration based on historical data but the client sample concentration turns out to be much lower or non-detect, the MS/MSD recovery results would still be usable.

- Matrix spiked samples are run with every analytical batch of samples.
- X-bar control charts are generated using a minimum of the last 20 data points, based upon percent recovery.
- Warning limits are set at the 95% confidence interval and are plus/minus two standard deviations from the arithmetic mean.
- Control limits are set at the 99% confidence interval and are plus/minus three standard deviations.
- MS limits are calculated at least annually or sooner where necessary. See the individual laboratory appendices for the list of established limits.
- Method stated limits supercede in-house calculated limits.

MS/MSD Percent Recovery:

% Spike Recovery=  $\frac{Spiked \ sample \ value \ - \ initial \ sample \ value}{Concentration \ of \ spike} X \ 100$ 

Standard Deviation for Percent Recovery:

Calculate using the same formula provided in the previous LCS section.

#### 5.9.2.9 Precision

Precision is assessed through the use of duplicate client and/or QC samples, which constitute approximately 10% of all samples run. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|Duplicate \ 1 - Duplicate \ 2|}{\left[\frac{(Duplicate \ 1 + Duplicate \ 2)}{2}\right]} X \ 100$$

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 57 of 74

- **§** Duplicates are analyzed with every analytical batch.
- **§** X-bar control charts are generated using a minimum of the last 20 data points, based upon percent recovery.
- **§** Warning limits (WL) are set at the 95% confidence interval using

 $WL = Mean Value + (2.456 \bullet SD)$ 

**§** Control limits are set at the 99% confidence interval and are plus three standard deviations.

$$CL = Mean Value + (3.268 \bullet SD)$$

- Limits are calculated at least annually or sooner where necessary. See the individual laboratory Appendices for the list of established limits.
- **§** For Laboratory Control Samples and Matrix Spikes Calculate RPD using the actual analytical result.
- **§** For Sample Duplicates Calculate RPD using the actual analytical result.
- **§** Calculate the standard deviation, separately for LCS, MS and Sample Duplicates by matrix, where appropriate.
- **§** Method stated limits override in-house calculated limits.

#### 5.9.2.3.10 5.9.2.10 Marginal Excedence Limits

Due to the large number of compounds analyzed using some analytical methods, it is statistically likely that accuracy and precision failures occur. Failures that occur on a random basis are deemed as marginal excedences and must meet the criteria below. Not all regulatory programs allow for the use of marginal excedence limits. In addition, not all analytical methods meet the requirements for the use of ME limits. Refer to the specific determinative SOP for more guidance regarding use and limitations.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 58 of 74

Marginal excedences must be random events. If failures can demonstrate a pattern or occur with the same target analyte in a trend, the failure is actionable and not considered to be marginally exceeding the method requirements.

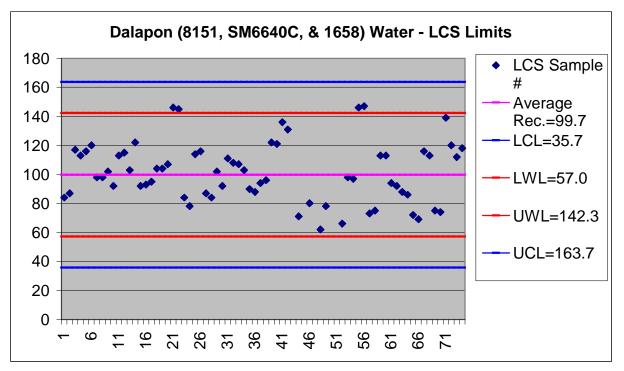
In addition, ME limits are utilized for methods with large numbers of target analytes being analyzed concurrently, as in the 8270/625 determinative method.

For example, the normal compound list for 8270/625 typically contains 90+ analytes; therefore, per the criteria listed below, only 5 analytes can be considered as marginally exceeding the acceptance criteria. If more than 5 failures occur or if the failures demonstrate a pattern that is causing the outliers, the entire sample batch with associated QC must be re-extracted and re-analyzed.

Upper and lower marginal excedence (ME) limits are established by +/four times the standard deviation of historical accuracy data and the number of marginal excedences allowed is based on the number of analytes spiked in the LCS.

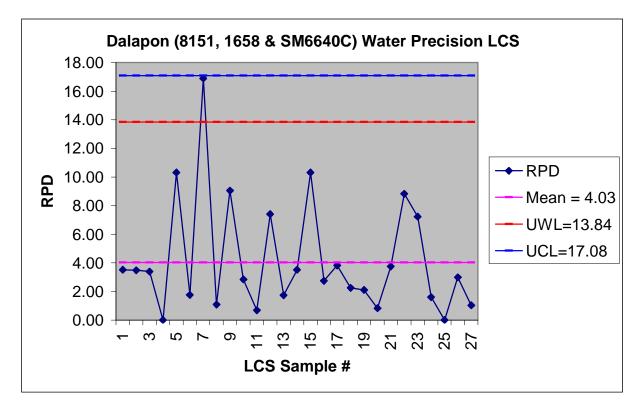
Number of	Allowable Marginal				
Target Analytes	<b>Excedence Outliers</b>				
90+	5 analytes allowed in the ME limit				
71-90	4 analytes allowed in the ME limit				
51-70	3 analytes allowed in the ME limit				
31-50	2 analytes allowed in the ME limit				
11-30	1 analytes allowed in the ME limit				
<10	0 analytes allowed in the ME limit				

## FIGURE 5.9.2.10 PRECISION AND ACCURACY CHARTS



Dalapon LCS Duplicate Accuracy - Example

Dalapon LCS Duplicate Precision - Example



Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 60 of 74

## 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 work, includes, 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.

Section 5.0, Ver. 10.0 Date: April 15, 2012 Page: 61 of 74

- 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 below.

ESC Lab Sciences Quality Assurance Manual Technical Requirements

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 62 of 74

## Figure 5.10.2 Example Final Client Report

No. Lon Teres State Sound Ling State Sound Lin	Environmental Science Corp.					Loss exercit Ro. House House the Const of St Cost Ress Loss Part Ress Carlot Cost Ress Cost Job 78 (1914)80	
Parts Received () Kana 12, 1007 Receive () () () 100 Strate () () () 100 Strate () () () 100 Strate () () () 100 Strate () () () () () () () () () () () () ()	A25 Converting NGN Primestory Primes	DT N.	NT OF REALIZED		Netal VAL D	tt, 12-0	
Strate 1 ( ) 1 HE USE Al vate 77 ( ) Ann Tes Tollevitte Late : 03/12/07 13:00 <u>Annatore</u> <u>Recult</u> <u>Det. Late : 100 Store</u> <u>Care 711.</u> <u>Primovale Sectory (0-100)</u> <u>Subsectory (0-100)</u> <u>Subsecto</u>					so smole		
Del votto Tyr. : Anno Tes.     Derivet & : Vote esy.       Vallestich Late : 03/12/07 13:00       Bonnetter     Anno Tes.       Petrick : 10:00       Bonnetter       Petrick : 03/12/07 13:00       Bonnetter       Petrick : 03/12/07 13:00       Bonnetter       Petrick : 03/12/07 13:00       Bonnetter     Out       Petrick : 03/12/07 13:00       Bonnetter       Petrick : 03/12/07 1       Bonnetter     Out       Petrick : 03/12/07 1       Bendete     Out       Petrick : 03/12/07 1       Bendeter     Out       Petrick : 03/12/07 1<	Served as 1 1 11 11 210		(1) we find the second second				
Jackson         Atoult         Det. Lame         Anto Net Original         Date         Det.           P-1 (0.110) rescare to 0         0.0         1.11         mg.1         AD15000         C3/0/07         1           P-1 (0.110) rescare to 0         0.0         1.11         mg.1         AD15000         C3/0/07         1           Sumptions Sectory (0-100)         0.0         1.11         mg.1         AD15000         C3/0/07         1           Lenter         0.0         1.1000         0.01         0.000         C3/10/07         1           Lenter         0.0         1.1000         0.01         0.000         C3/10/07         1           Lenter         0.0         1.1000         0.01         0.000         C3/10/07         1           Styperson         3.01         1.1000         0.01         0.000         C3/10/07         1           Styperson         3.01         1.1000         0.01         0.000         C3/10/07         1           Styperson         3.01         1.1000         mg/1         0.000         C3/10/07         1           Styperson         3.01         0.0000         mg/1         0.000         C3/10/07         1           H	Call untitle By I - Jama Tela				Surgers € :	the second	
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$						· ··	
Spinnopics Seriesry (010):         K/20         North Constant (020):           Spinnopics Seriesry (010):         K/20         North Constant (0.00):         0.00000000000000000000000000000000000		X11(			2221222	14.10 111.	
System         System <thsystem< th=""> <thsystem< th=""> <thsystem< td="" th<=""><td></td><td>1.4</td><td>• . • •</td><td>···, 1</td><td>A.01 F14-11</td><td>03/00/07 1</td></thsystem<></thsystem<></thsystem<>		1.4	• . • •	···, 1	A.01 F14-11	03/00/07 1	
Psicence         Sul         11000         0001		*2.3		5,555	3 03 550	(3/2)/(2) = 1	
Distribution         Cold	Lenzere	200		2Q/1	00000		
Distribution         Cold			11.000			03/14/07 1	
Tophetalene         RDI         0.0000         mpv1         004.05         00/24/07         1           Openation Descenty         notematic Descenty         45.7         41.0         3260.         03/24/07         1           Descenty         45.7         41.7         41.0         3260.         03/24/07         1           Learner difference         30.1         31.00         31.00         000000         00000         1           Contraction problematics         40.7         41.00         41.00         000000         1           Science describer         40.1         41.00         41.00         000000         1           Science describer         40.15         40.10         000100         1         0000000         1			0.000			(0)% (1) (2)	
Tophetalene         RDI         0.0000         mpv1         004.05         00/24/07         1           Openation Descenty         notematic Descenty         45.7         41.0         3260.         03/24/07         1           Descenty         45.7         41.7         41.0         3260.         03/24/07         1           Learner difference         30.1         31.00         31.00         000000         00000         1           Contraction problematics         40.7         41.00         41.00         000000         1           Science describer         40.1         41.00         41.00         000000         1           Science describer         40.15         40.10         000100         1         0000000         1			1.107.0				
Specificate Disestry         AP.7         Ar.         Ar.         AP.7         Ar.         Ar.         AP.7         Ar.         Ar. </td <td></td> <td></td> <td>1,1010</td> <td></td> <td></td> <td></td>			1,1010				
Information         ART         Art.         S260         C3/84007         1           Lingramitic point bound		105	0.00.0	- 19 C	810.05	(0/24/07) 1	
Libricilius pono Exame 301 & 2001 visto 60/10/000 1 2-Entres nonotempane 201 & 2020 visto 00/00/000 1 Suiradishle seiroleum dydroward 0005 0010 mg/1 dsm. 60/00/07 1 Suiradishle seiroleum dydroward 0005 0000 1		A			1120	63 '6 V67 V	
C-Bringst nonotonizand         Kell         Kell         Cl/Cr/C/         L           Staracteble setroleum dyddoward         0.15         0.10         mg/1         Dat         CD/CC/C7         L           Outrovale kedivery         0.15         0.10         mg/1         Dat         CD/CC/C7         L		•					
Intradichle setroleum Nydrovard (115 (111 mg/l dan 60/00/07 1 Outrovate Reditery						0.0000000000000000000000000000000000000	
Aniradobile optimetik (yalovali) - ynig - ynig - ngyl Esi - Gylovo, i Ourivyan wedrery							
During And Meriya Di Tung Tang King Ang Ting (Karang Ting King Ting King Ting King Ting King Ting Ting King Ting Ting Ting Ting T		24.17	::	$m_{\rm e}/1$	_لوت_	60/06/67 1	
		×1.4		s nati	7717	(3/20/07 - 1	

565 - D. W Denos or Fin Berlin, Harris M. Andreas in FileByd() Note: The reported analytical results in atomic the single spacificad. This report the minimum being remained, rescale of the single spacification. This report the minimum being remained, rescale of the single statistical systems. File 785.

Reported: 01/26/07 19:28 Juniters (3/20/17 00:01)

save 1 00 C

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 63 of 74

Envir Science								21. 61 (61.) 2 0-000-2 Fam (01	-Lesam 63, 147, 111 (7 24, 2858 (2-2019 20, 200-200 20, 200-200, 200-200 20, 200-2000 20, 200-20000000000000000000000000000000	ç.
			777	OTT OF ANAL	жато					
re, Alila Kirs ABC Cristi tarta 12. mnywiele Jure Sanwaarne, 73.000						5000000	857 25	, 10040a		
						- 84	S.0017	6 9 32	00000-01	
NUS RAIN STOLLE		ter inter st	10. v							
resoldy lon (	slave	τ_				čite.				
Consiler TO Consiler St	Th: 111	n=								
Collected Ly : : collection face :							ant fr	: 123	3:5	
hand o Lon			R <sup>1</sup> (11 <sup>°</sup> ).	NG	БG	14 B	2	a⊱1 €	14.15	100.7
0(0			3.8	34.0	6.0	ha71	20	2892100	12/07/06	2
A			S2.	6.3	( C .	w <b>y</b> / 1		41,1,4	16/11/06	1.
seconda secologi	N.		0,043	0.031	9,00	$n_{3/1}$	- 12	259.4	19/00/05	
Supported Solid	-		10 <b>3</b>	9.33	1.,3	wy/1		1626.8	157 7.00	
Coopled Lond				6.0133 6.0126	0.020 0.0050	ж.4/1 ж.4/1			12/00/06 12/00/06	

c and first moreousd: cfl = Minimum Extremion that the suff = SQL2 KEV REL = Reported Derection that = E02 = RQL = E01 = XQL(TERP) Xole: the reported sublytical menuits beliefs only to the sufficient enomistics. this report shall not be restricted, early in fail, without the unitten approval from Svc. this report. Shall not be restricted, early in fail, without the unitten approval from Svc.

September 12/14/06 13:355 Decideds 15/16/06 09:10

Coge 1. St 3.

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 64 of 74

The following qualifier codes are used when reporting data values that either meet the specified description outlined below or do not meet the quality control criteria of the laboratory:

(This table provided for example and is subject to revision without notice. For a list current qualifiers, contact the laboratory)

# Table5.10.2ESC Qualifiers and Descriptions (Updated 7/15/09)

QUAL	DESCRIPTION
А	ALC(EPA)-Aldol Condensation: Labels a suspected Aldol Condensation product for TICs.
В	(EPA) - The indicated compound was found in the associated method blank as well as the laboratory sample.
B1	(ESC) - The blank depletion was greater than the recommended maximum depletion of 0.2mg/L.
B2	(ESC) - The detection limit has been elevated due to blank contamination.
B3	(ESC) - The indicated compound was found in the associated method blank, but all reported samples were non- detect.
B4	(ESC) - The indicated compound was found in the associated instrument blank, but all reported samples were non- detect.
B5	(ESC) - The indicated compound was found in the associated instrument blank as well as the laboratory sample.
С	CBC(EPA)-Cannot be calculated: The analytical result cannot be calculated because the internal standard was not found.
D	Less than lower calibration limit. Actual value is known to be less than the lower calibration range due to dilution.
Е	GTL (EPA) - Greater than upper calibration limit: Actual value is known to be greater than the upper calibration range.
F	SRN (EPA) - Diluted: The original sample was diluted due to high amounts of one or more target analytes. All associated method analytes will be subject to an elevated detection limit relative to the dilution factor.
G	SRS(EPA)-Secondary Dilution: The indicated analysis results were generated from a secondary dilution of the same sample. The sample had to undergo serial dilution.
Н	RIN(EPA)-Re-Analyzed: The indicated analytical results were generated from a reinjection of the same sample extract or aliquot.
I1	(ESC) Not analyzed due to interference. (Sample reacted with method reagent or could not be analyzed due to interferences that could not be corrected)
J	(EPA) - Estimated value below the lowest calibration point. Confidence correlates with concentration.
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 limits cannot be evaluated; surrogates were diluted out
J8	The internal standard associated with this data responded abnormally low. The data is likely to show a high bias concerning the result.
J9	The internal standard associated with this data responded abnormally high. The data is likely to show a low bias concerning the result.
K	REX(EPA)- Re-prepared: The indicated analytical results were generated from a re-extraction or preparation of the sample.
L	(ESC)Sample Pretreatment: The sample reaction impaired the ability to analyze the sample using normal analytical determination. Treatment outside of method protocol was required to determine the analytical result.
L1	(ESC) The associated batch LCS exceeded the upper control limit, which indicates a high bias; The sample analyte was "not detected" and is therefore unaffected.

# Table

# **5.10.2** ESC Qualifiers and Descriptions (Updated 7/15/09)

QUAL	DESCRIPTION
L2	(ESC) The associated surrogate compound falls below 10%. The data should be used with caution. A re-extraction was not possible due to limited sample volume.
L3	(ESC) Sample reanalysis could not be performed due to lack of additional volume.
М	AVE(EPA)-Average Value: Used to report a range of values; e.g., relative response factors
Ν	PRE (EPA) - Presumptive evidence of material.
N8	PRE (EPA) - Presumptive evidence. The component has been tentatively identified based on mass spectral data.
N9	PRE (EPA) - Presumptive evidence. There is indication that the analyte is present, but QC requirements for confirmation were not met
0	(ESC) Sample diluted due to matrix interferences that impaired the ability to make an accurate analytical determination. The detection limit is elevated in order to reflect the necessary dilution.
01	(ESC) The analyte failed both the method required serial dilution test and subsequent post-spike criteria. These failures indicate matrix interference.
Р	NRP(EPA)-Non-Reproducible: Results of two or more injections are not comparable
P1	RPD value not applicable for sample concentrations less than 5 times the reporting limit.
Q	(ESC) Sample held beyond the accepted holding time.
R	REJ(EPA)-Rejected: Results have been rejected by the lab and should not be used
S	Subcontracted (ESC) - This analysis was performed by a subcontractor chosen to meet the project requirements.
Т	(ESC) - Additional method/sample information: Sample collected using improper field protocol
T1	(ESC) - Additional method/sample information: Sample(s) received at greater than 4 degrees C.
T2	(ESC) - Additional method/sample information: The laboratory analysis was from an unpreserved or improperly preserved sample.
T3	(ESC) - Additional method/sample information: TOX analysis. Greater than 10% Breakthrough
T4	(ESC) - Additional method/sample information: QNS - Quantity Not Sufficient
T5	(ESC) - Additional method/sample information: QNS - Quantity not sufficient for reanalysis or replication as required by method.
T6	(ESC) - Additional method/sample information: Method used is an alternative to current approved methodology
T7	(ESC) - Additional method/sample information: Method 1664 (Total Oil & Grease), performed without silica gel
T8	(ESC) - Additional method/sample information: Sample(s) received past/too close to holding time expiration.
Т9	(ESC) - Additional method/sample information: The sample result represents blank correction
U	BDL (EPA) - Below Detectable Limits: Indicates that the compound was analyzed but not detected.
V	(ESC) - Additional QC Info: The sample concentration is too high to evaluate accurate spike recoveries.
V1	(ESC) - Additional QC Info: Estimated concentration: due to inability to achieve ending QC standard as a result of sample matrix interference.
V2	(ESC) - Additional QC Info: The Total Cyanide value was below the reporting limit. Amenable Cyanide is assumed not to be present.
V3	(ESC) - Additional QC Info: The internal standard exhibited poor recovery due to sample matrix interference. The analytical results will be biased high. BDL results will be unaffected.
V4	(ESC) - Additional QC Info: Cont. Calibration Verification exhibited a response outside of the QC criteria, but within a 5% window. The associated analytical results are biased high. Non-detect results are unaffected.
V5	(ESC) - Additional QC Info: The Laboratory Control Sample exhibited a response outside of the QC criteria, but within a 5% window. The associated analytical results are biased high. Non-detect results are unaffected.
V6	(ESC) - Additional QC Info: The ICV responded above the recovery range for one of the following: Al, Ca, K, Fe, Na, Zn. The associated analytical results are biased high.
V7	(ESC) - Additional QC Info: This compound is not a 524.2 compound and was therefore evaluated using 8260B QC Criteria.
V8	(ESC) - Additional QC Info: The Interference Check Standard responded above the acceptable recovery range. The associated analytical result may be biased high for this element.

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 66 of 74

Table

### 5.10.2 ESC Qualifiers and Descriptions (Updated 7/15/09)

QUAL	DESCRIPTION
V9	(ESC) - Additional QC Info: Please refer to the Case Narrative provided with the report.
W	(ESC)-The laboratory analysis was from a sample collected in an improper container
W1	(ESC) - The laboratory analysis was from a sample collected in containers provided by the client.
W2	(ESC) - Insufficient sample amount to perform method as required. Sample amount approved per client instruction.
W3	(ESC) - BOD cannot be determined due to apparent toxicity exhibited by the sample.
Х	(ESC)-Holding time exceeded due to National Emergency
X1	(ESC)-National Emergency: Temperature requirement has been exceeded due to delayed transportation.
Y	This sample most closely matches the laboratory standard for Kerosene
Y0	Significant peaks were detected outside of the hydrocarbon range defined by the method.
Y1	This sample most closely matches the laboratory standard for Diesel
Y2	This sample most closely matches the laboratory standard for #6 Fuel Oil
Y3	This sample most closely matches the laboratory standard for Hydraulic Fluid
Y4	This sample most closely matches the laboratory standard for Motor Oil
Y5	This sample has responded in the Diesel range, however it does not appear to be a hydrocarbon product
Y6	This sample has responded in the Oil range, however it does not appear to be a hydrocarbon product
Y7	This sample most closely matches the laboratory standard for Gasoline
Y8	This sample has responded in the Gasoline range, however it does not appear to be a hydrocarbon product
¥9	Sample has one or more single components in the gasoline range but the chromatographic trace is not characteristic of gasoline.
Z	(ESC)-Too many colonies were present(TNTC), the numeric value represents the filtration volume.

### 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 ESC client. In addition to the EPA qualifiers adopted by ESC, the laboratory has implemented ESC qualifiers to provide more information pertaining to analytical results. Each qualifier is designated in the qualifier explanation as either EPA or ESC. Definitions used in this table can be found in Section 3.

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.

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 67 of 74

### 5.10.5 Opinions and Interpretations

Opinions and interpretations are allowed in final reports, in the form of qualifiers, provided that it is clear that the qualifiers are present to provide additional analytical information. In the event that a report must be issued with a revision, the original report remains unaltered and the revision is clearly identified. See SOP #030223, *Report Revision*.

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

Data packages are provided when requested by the client. They range from QC summaries to "CLP-like" packages with raw data. When a data package is requested at the beginning of a project, the level of package is identified, and it is then logged into the LIMS using the appropriate product code.

The analyst performing the analysis or a QC Specialist generates the QC documentation. The package is generated using the following process:

- Data and Supporting documentation is gathered by the QC Specialist (QCS)
- The package is formatted to the client request and submitted for review:
- Section Supervisor or Senior analyst
- Technical Specialist, Department Manager, Lab Director or designee.
- Once the reviews are complete, the package is logged, copied/scanned/burned to CD, and shipped. The ESC preferred means of delivery is via ESC's secure web site (PDF format) in recognition of the paperwork reduction act.
- See Table 10.8 for typical data package information.

### 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:

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 68 of 74

	Table 5.10.8 Data Package Contents
Level I Level II	Standard QC Data Package Provided Upon Request
	Final Analytical Report with qualifiers where necessary
	Sub-Contract Final Report if applicable
	Chain of Custody (COC) Form
	Method Blank
	Matrix Spike/Spike Duplicate Summary (MS/MSD) - with Control Limits
	Laboratory Control Sample Summary (LCS) - with Control Limits
	Reporting Limits listed on all reports
	Surrogate Recoveries for GC and GC/MS analyses (on final report)
	Case Narrative upon request
Level III	Data Package Provided Upon Request
	All QC Data Included in Levels I and II plus:
	MS/MSD analysis performed on specific sample upon request
	Initial and Continuing Calibration Information
	Instrument blank performance
Level III - Mod	Data Package Provided Upon Request
	All QC Data Included in Levels I, II and III plus:
	Chromatograms, including Batch QC, and Samples
Level III - Mod	Data Package Provided Upon Request
	Quantitation Reports
	Analysis Log
	Extraction Logs
Level IV	Data Package Provided Upon Request
	("CLP-Like" Validation Package)
	All QC Data Included in Levels I, II, III and III mod plus:
	Multiple Sample Dilutions Reported
	Before/After reports when manual integration is necessary (where requested)
	Initial and Continuing Calibration Chromatograms and Quantitation
	Surrogate, Tune, Internal Std & Method Blank summary forms
	Standard Preparation Logs

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 69 of 74

### 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 LABORATORY DATA REDUCTION (SOP 030201 Data Handling & Reporting)

The primary analyst completes the majority of data reduction using the following:

- Manual calculation, as represented on the bench sheet.
- Input of raw data for computer processing.
- Direct acquisition of raw data by computer.

### 5.11.1 Manual Calculations

If data requires manual calculation, the analyst has the responsibility of recording all steps involved directly on the bench sheet. Each bench sheet must be completed in a manner so that during review the person checking the raw data can easily reproduce the calculations. All pertinent information is included such as: response factors, dilution factors, and calibration constants. The analyst signs and dates each page of calculations in ink. A secondary review is required for all data. The second reviewer also initials and dates the worksheet. The worksheets are bound in chronological order in a laboratory workbook designated for each analysis.

### 5.11.2 Computer Processing

If data is input and processed using a computer, a hard copy of the input and output is reviewed to ensure that no discrepancies exist. The person 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, 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.

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 70 of 74

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 done 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

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 71 of 74

### 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 DATA VALIDATION PROCESS

### 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 filled out, 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.

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 72 of 74

### 5.12.3 Laboratory Analysis, QC, and Data Review

### Lab Analyst

- **§** After the COC has been reviewed and the sample has been logged in, the laboratory performs all required analyses. The Lab Supervisors are responsible for ensuring that all samples are run within holding time.
- **§** At the beginning of each analysis or sample preparation, the analyst is responsible for making sure that all laboratory ID numbers on the sample bottles match those listed on the benchsheet or logbook.
- **§** Sample transfer from bottle to container is periodically spot checked by a qualified senior analyst.
- **§** Upon completion of the analysis the analyst verifies that analytical information and results are correct and complete, the appropriate SOP has been followed, manual integrations (where applicable) have been correctly interpreted, QC samples are within established limits, and supporting documentation is complete.
- **§** The benchsheet is then given to a QC Specialist who reviews the same information and ensures all portions of the benchsheet are complete.
- **§** The review person then initials and dates the benchsheet.

### Extraction/Sample Prep

- The Department supervisor's are responsible for reviewing all extraction/ preparation logs. The review verifies completeness regarding method, sample amount, reagent amount, times, temperatures, etc.
- The extraction/prep logs are reviewed for sample prep method as well as sample extraction date versus holding time.

### Final Data Responsibility

- **§** The Department supervisor for each section of the laboratory is responsible for reviewing instrument run logs and benchsheets to ensure that the samples are being prepared and analyzed within holding times.
- **§** The QC Specialist performs a final review before the data is approved for input into the computer.
- **§** This review includes performance of the various blanks, precision QC and accuracy QC to determine if the set is within quality control criteria. Data reviews are conducted according to the SOP #030227, *Data Review*, that provides more detail regarding specific steps taken in the review process. In some cases, specific regulatory guidelines on the data review process include additional requirements (i.e. Ohio VAP's data review checklist use) that are also included in the SOP.

Section 5.0, Ver. 7.0 Date: April 15, 2009 Page: 73 of 74

- **§** If the data is not approved during the final review process, it is given a pending status and returned to the laboratory.
- **§** Pending data is reviewed for corrective action and may require only recalculation or may result in re-analysis.

# <u>Final Report Review</u>

- **§** For manual data, the reviewed data is entered in the LIMS; the input is reviewed against the raw data by a second person for accuracy.
- **§** Data transfer is reviewed and approved by a QC Specialist.
- **§** The client reports are then prepared for review by the assigned Technical Service Representative (TSR). The report is reviewed for correlation between related parameters as well as possible trends. The TSR reviews related supporting documentation such as chain of custody records, field documents, sample receipt information, compliance with client/project specific requirements, etc.
- **§** All field documents are reviewed and approved before the final review. Field data that does not pass established criteria is not processed through the final report review.
- **§** The Environmental Monitoring Manager is responsible for any corrective actions necessary concerning field results.
- Laboratory result values that appear anomalous are sent back to the laboratory for a second review of the raw data.
- If there is no apparent reason for the anomaly the sample is re-analyzed.
- If the sample holding time has expired, the sample is re-analyzed and flagged.
- If the client desires, a new sample can be collected and evaluated.
- The chain of custody is also reviewed for a final time to ensure that all project objectives have been met.
- The LIMS footnotes any parameters that may exceed established limits as provided by the client.
- When the LIMS notes that a limit has been exceeded, the Technical Service Representative is notified and the client is contacted.

Table 5.12.3       DATA REDUCTION AND VALIDATION FLOW							
Primary Activity	Supporting Activity	Responsibility					
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	boratory QC Review of Data Completion and QC Limit Verification Primary Analyst/Chem						
Approval of Laboratory QC	Review of Data Completion and QC Limit Verification	QC Specialist/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 QC Specialist					
Data Entry to LIMS	Data Transfer - Application of Qualifiers	Data Entry Specialist followed by QC Specialist Verification					
Data Entry to LIMS	Manual Entry of Data and Qualifiers	Data Entry Specialist followed by QC Specialist Verification					
Draft Final Report	Report printed and given to TSR	Data Entry Specialist or					
Generation	for Approval	Administrative Assistant					
Final Report Review and Approval	TSR Approval/Signature	Technical Service Representative (TSR)					

Section 6.0, Ver. 10.0 Date: April 15, 2012 Page: 1 of 7

# 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 6.1) 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 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.

# 6.2 MOLD/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) until 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 contain the following information: date prepared, analyst initials, expiration date, and reagent name. In house reagents are recorded with the same information in a reagent prep book assigned to that method.
- Purchased reagent solutions are labeled when received and 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 sample storage.
- Reagents and standards used for volatile organic analysis are stored in a separate refrigerator and are not stored with samples.
- See the following table for more information regarding reagent storage.

Item	Reagent Storage
Acids	Designated acid storage cabinets, in original container.
Organic Reagents -	Stored in flammables cabinet on separate air system from volatiles
Flammables	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 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

Section 6.0, Ver. 10.0 Date: April 15, 2012 Page: 3 of 7

### 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.
- ESH 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.
  - Ø 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.
  - Ø
  - Ø
  - Ø Table 6.1 lists waste disposal methods for various test byproducts.
- 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, food 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 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. Any detectable concentration must be  $\leq 1/2$  of the established regulatory limit for each metal being analyzed. If any detectable amount exceeds the established criteria, then the area must be cleaned and verified before analysis can resume.

6.4.2 VOCs

Section 6.0, Ver. 10.0 Date: April 15, 2012 Page: 4 of 7

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.

### 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. 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.

Section 6.0, Ver. 10.0 Date: April 15, 2012 Page: 5 of 7

# TABLE 6.1 - 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 CLASSIFICATIO N	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	None		
Cyanide, Total	acidic waste	corrosive	neutralize-sanitary sewer
Cyanide, Amenable	acidic waste	corrosive	neutralize-sanitary sewer
Flashpoint	Misc. Organic waste contiaining Chlorobenzene	Flammable	Dispose via haz waste regulations
Fluoride, Electrode	neutral waste solution	none	sanitary sewer
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
рН	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 (% solids)	None		
Solids, Total Dissolved	None		
Solids, Total Suspended	None		
Turbidity	None	none	none
Metals	acids, metal solutions		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	Food samples, enrichment broth,	none	Disinfect – sanitary sewer

# ESC Lab Sciences Quality Assurance Manual *Waste Minimization/Disposal And Reagent Storage*

Section 6.0, Ver. 10.0 Date: April 15, 2012 Page: 6 of 7

PARAMETER WASTE PRODUCTS		WASTE CLASSIFICATIO N	DISPOSAL METHOD	
Biological Non- biohazardous Waste	Gloves, plastic containers	none	Standard refuse	

### FIGURE 6.1 (reprint of excerpt – current as of 3/12/08)

#### 40 CFR PART 261-IDENTIFICATION AND LISTING OF HAZARDOUS WASTE

Subpart A-General Sec.

- 261.1 Purpose and definition.
- 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.

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 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.

ESC Lab Sciences Site Plan Appendix I to the ESC QAM App. I, Ver. 10.0 Date: April 15, 2012 Page 1 of 2

# **ESC Site Plan**

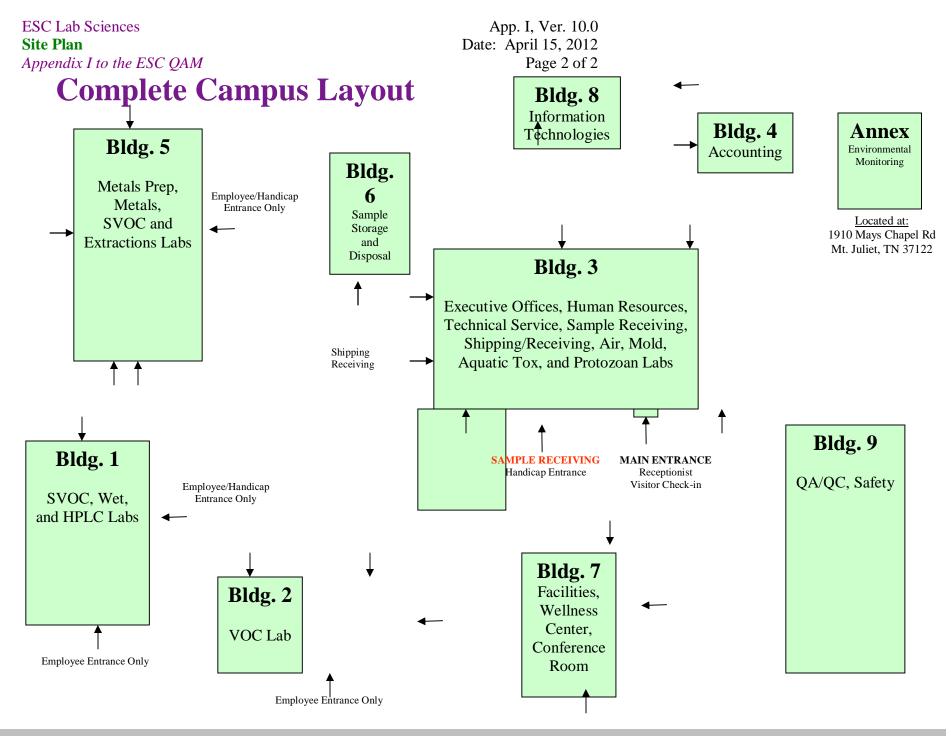
# APPENDIX I TO THE ESC QUALITY ASSURANCE MANUAL

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



12065 Lebanon Road Mt. Juliet, TN 37122

App. II, Ver. 10.0 Date: April 15, 2012

# **ESC Certifications**

# APPENDIX II 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

# **ENVIRONMENTAL SCIENCE CORPORATION**

State/Agency	Certificate Number	Expiration Date/Status	Cert. REV. Date	Date Posted	Certified Programs	Approved Programs	Cert.Type	Cert. Authority
<u>Alabama</u>	40660	6/30/2011		7/23/2010	DW	WW, RCRA, UST	Reciprocity	TN
<u>Alaska</u>	UST-080	1/11/2011		4/20/2010	UST	UST	AK	AK
<u>Arizona</u>	AZ0612	6/25/2011		7/8/2010	AIR, DW, WW, RCRA, UST		Audit	AZ
<u>Arkansas</u>	88-0469	1/21/2011		2/18/2010	WW, RCRA, UST, Bloassay		NELAP	NJ
<u>California</u>	01157CA	8/31/2011		9/28/2010	WW, RCRA, UST		NELAP	NJ
<u>Colorado</u>	None	3/31/2011		6/2/2009	DW	WW, RCRA, UST	Reciprocity	TN
Connecticut	PH-0197	3/31/2011		4/16/2009	DW AIR, DW, WW, RCRA,	WW, RCRA, UST	Reciprocity	TN, NJ
<u>Florida</u>	E87487	6/30/2011		7/8/2010	UST		NELAP	NJ
Georgia DW	923	Renewal	Renewal	8/17/2007	DW		Reciprocity	TN
Georgia	None	6/30/2011		7/8/2010	WW, RCRA, UST		NELAP	NJ
Idaho	TN00003	6/1/2011		8/5/2010	DW	WW, RCRA, UST	NELAP	NJ
<u>Illinois</u>	200008	11/30/2011		11/22/201 0	DW, WW, RCRA, UST		NELAP	NJ
<u>Indiana</u>	C-TN-01	6/16/2013		8/5/2010	DW	WW, RCRA, UST	Reciprocity	TN
<u>lowa</u>	364	5/1/2012		8/5/2010	WW, RCRA, UST		NELAP	IA
<u>Kansas</u>	E-10277	10/31/2011		11/2/2010	DW, WW, RCRA, UST		NELAP	NJ
Kentucky DW	90010	12/31/2010		4/13/2010	DW	WW, RCRA	Reciprocity	TN
Kentucky UST	16	10/16/2011		11/3/2010	UST		Audit	A2LA
Louisiana	Agency ID 30792	6/30/2011		11/22/201 0	WW, RCRA, UST, AIR		NELAP	NJ
<u>Maine</u>	TN0002	7/5/2011		8/4/2009	DW, WW	RCRA, UST	Reciprocity	TN, NJ
Maryland	324	12/31/2010		2/16/2010	DW		Reciprocity	TN
Massachusetts	M-TN003	6/30/2011	7/1/10	9/28/2010	DW,WW	RCRA, UST	Reciprocity	TN
<u>Michigan</u>	9958	6/16/2013		8/31/2010	DW	WW, RCRA, UST	Reciprocity	TN
<u>Minnesota</u>	047-999- 395	12/31/2011		11/3/2010	WW, RCRA, UST		Audit	MN
<u>Mississippi</u>	None	6/16/2013		9/28/2010	DW	WW, RCRA, UST	NELAP	NJ
Missouri	340	6/16/2013		9/28/2010	DW	WW, RCRA, UST	NELAP	NJ
<u>Montana</u>	CERT0086	Renewal	Renewal	7/16/2007	DW	WW, RCRA, UST	Reciprocity	TN
<u>Nebraska</u>		6/30/2011		8/31/2010	DW	WW, RCRA, UST	Reciprocity	TN
<u>Nevada</u>	TN-03- 2002-34	7/31/2011	Extended	8/19/2010	WW, DW, RCRA, UST		NELAP	NJ
New Hampshire	2975	5/20/2011	Jul-10	7/8/2010	DW, WW	RCRA, UST	NELAP	NJ
<u>New Jersey -</u> NELAP	TN002	6/30/2011		7/8/2010	DW, WW, RCRA, UST, AIR		NELAP	NJ
New Mexico	None	6/30/2011		7/9/2010	DW	WW, RCRA, UST	NELAP	NJ

	44740	4/4/0044	0/0/4.0	0/0/0040				
New York North Carolina	11742	4/1/2011	6/3/10	6/9/2010	WW, RCRA, UST, AIR		NELAP	NJ
DW	DW21704	7/31/2011		8/5/2010	DW		Audit	NC
North Carolina	Env375	12/31/2010		1/15/2010	WW. RCRA. UST		Audit	NC
North C. Aquatic				11/22/201	,			
<u>Tox</u>	41	11/1/2011		0	Aquatic Toxicity		Audit	NC
North Dakota	R-140	6/30/2011		7/23/2010	DW, WW, RCRA		Reciprocity	TN, WI
Ohio VAP	CL0069	4/14/2011	Jan-08	6/2/2009	WW, RCRA, UST, AIR		Audit	ОН
<u>Oklahoma</u>	9915	8/31/2011		11/3/2010	WW, RCRA, UST, BIOASSAY		NELAP	NJ
Oregon	TN200002	1/15/2011		2/16/2010	DW, WW, RCRA, UST		NELAP	NJ
Pennsylvania	68-02979	12/31/2010		1/15/2010	DW, WW, RCRA, UST		NELAP	NJ
Rhode Island	221	12/31/2010		2/16/2010	DW, Env. Lead	WW, RCRA, UST	Reciprocity	TN, AIHA
South Carolina	84004	6/30/2011		11/22/201 0	WW, RCRA, UST		NELAP	NJ
South Dakota	Pending	Pending						
T	0000	0/4.0/00.40		7/00/0040	DW	WW, RCRA,	A	-
Tennessee DW Tennessee DW	2006	6/16/2013		7/23/2010	DW	UST	Audit	TN
Micro	2006	10/12/2012		2/16/2010	DW Micro		Audit	TN
Texas Mold	LAB0152	3/10/2011		5/7/2007	MOLD		NA	ТΧ
<u>Texas - Env</u>	T 104704245- 07-TX	10/31/2011		11/3/2010	DW, WW, RCRA, AIR		Reciprocity	NJ
<u>Utah</u>	615758585 8	6/30/2011		8/5/2010	DW, WW, RCRA, UST		NELAP	NJ
Vermont	VT2006	1/5/2011	Jan-10	1/15/2010	DW	WW, RCRA, UST	Reciprocity	TN
<u>Virginia</u>	109	6/30/2011		7/8/2010	DW	WW, RCRA, UST	NELAP	NJ
Washington	C1915	8/19/2011	9/18/08	11/3/2010	DW, WW, RCRA, UST, AIR		Audit	A2LA
West Virginia	233	2/28/2011		4/9/2010	WW, RCRA, UST		Audit	WV
<u>Wisconsin</u>	998093910	8/31/2011		9/14/2009	WW, RCRA, UST		Audit	WI
Wyoming	A2LA	11/30/2011		7/23/2010	UST	WW, RCRA	Audit	A2LA
	Other Age	encies						
					DW, WW, RCRA, UST,			
<u>A2LA</u>	1461.01	11/30/2011	4/30/2010	7/23/2010	AIR, MICRO		Audit	A2LA
<u>AIHA*</u>	100789	6/1/2012		6/1/2010	IHLAP, ELLAP, EMLAP		Audit	AIHA
DOD	1461.01	11/30/2011		3/8/2010	RCRA, UST		Audit	A2LA
EPA	TN00003	None			Cryptospiridium		Audit	EPA
<u>USDA</u>	S-67674	8/7/2012		8/7/2009	Quarantine Permit		Audit	USDA

(1) A2LA = American Association for Laboratory Accred. (2) AIHA = American Industrial Hygiene Association (3) NELAP = National Environmental Laboratory Accred. 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

(9) Pending = The state is processing our application.
(10) EPA = Environmental Protection Agency

(4) IHLAP = Industrial Hygiene Laboratory Accred. Program
 (5) ELLAP = Environmental Lead Laboratory Accred. Program

ESC Lab Sciences Sampling Quality Assurance Manual Appendix III to the ESC QAM App. III, Ver. 10.0 Date: April 15, 2012 Page: 1 of 66

**1.0** SIGNATORY APPROVALS

# SAMPLING PROTOCOL QUALITY ASSURANCE MANUAL

# APPENDIX III 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. A signed cover page is available upon request

Judith R. Morgan, M.S., VP of Technical and Regulatory Affairs 615-773-9657

Eric Johnson, B.S., Laboratory Director 615-773-9654

Dixie Marlin, B.S, QC Manager, 615-773-9681

the

Tom Stinson, B.S., Environmental Monitoring Department Manager, 615-773-7551

App. III, Ver. 10.0 Date: April 15, 2012 Page: 2 of 66

# 2.0 APPENDIX TABLE OF CONTENTS

Section	Section Title			Rev. Date	Rev.#
1.0	Approval and Signature Page		1	4/15/11	2
2.0	Table of Contents		2	4/15/11	2
3.0	Scope and Application		3	4/15/11	2
4.0	List of Sampling Capabilities	Page	3	4/15/11	2
5.0	General Considerations	Page	4	4/15/11	2
6.0	Ancillary Equipment and Supplies	Page	10	4/15/11	2
7.0	Wastewater Sampling	Page	11	4/15/11	2
8.0	Surface Water and Sediment Sampling	Page	17	4/15/11	2
9.0	Groundwater and Drinking Water Sampling	Page	26	4/15/11	2
10.0	Soil Sampling	Page	36	4/15/11	2
11.0	Waste Sampling	Page	38	4/15/11	2
12.0	Standard Cleaning Procedures	Page	42	4/15/11	2
13.0	Sample History	Page	51	4/15/11	2
14.0	Sample Containers, Preservation, Methods, and Holding Times	Page	51	4/15/11	2
15.0	Sample Dispatch	Page	58	4/15/11	2
16.0	Investigation Waste	Page	60	4/15/11	2
17.0	Sampling Bibliography	Page	61	4/15/11	2
	TABLES				
4.0	List of Sampling Capabilites	Page	3	4/15/11	2
5.9.1	Quality Control Samples	Page	7	4/15/11	2
6.1	Ancillary Equipment and Supplies	Page	10	4/15/11	2
7.1	Wastewater Sampling Equipment	Page	11	4/15/11	2
8.1	Equipment List	Page	17	4/15/11	2
9.1	Groundwater and Drinking Water Sampling Equipment	Page	26	4/15/11	2
10.1	Soil Sampling Equipment		36	4/15/11	2
11.1	Waste Sampling Equipment	Page Page	38	4/15/11	2
14.6A	Solids Preservation, Holding Time and Containers	Page	55	4/15/11	2
14.6B	Wastewater Preservation, Holding Time and Containers	Page	55	4/15/11	2

App. III, Ver. 10.0 Date: April 15, 2012 Page: 3 of 66

# **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).

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
Microbiology	Surface water, groundwater, drinking water, wastewater
Macro Invertebrate Identification	Surface water, wastewater, sediments
Biotoxicity	Surface water and wastewater

### 4.0 LIST OF SAMPLING CAPABILITIES

App. III, Ver. 10.0 Date: April 15, 2012 Page: 4 of 66

## 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 5 of 66

### 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 shall be 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 shall be collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated samples shall 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 should use 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 shall <u>not</u> be used to collect samples for trace organic compounds analyses.
- When fuel powered units are utilized, they will be placed downwind and away from any sampling activities.
- Monitoring wells with free product shall not be sampled for trace contaminant analysis.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 6 of 66

# 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) should be 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 will be 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 occasionally inversion of the material.

Soil and sediment samples collected for volatile organic compounds shall <u>not</u> 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:

App. III, Ver. 10.0 Date: April 15, 2012 Page: 7 of 66

Number of samples	Precleaned 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.

### 5.9.1 Quality Control Samples

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 one crystal of 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. If unsuccessful, discard the vial and resample with a new preserved vial and septum. At a minimum, duplicate vials should always be collected from each sample location.

### ESC Lab Sciences Sampling Quality Assurance Manual Appendix III to the ESC OAM

App. III, Ver. 10.0 Date: April 15, 2012 Page: 8 of 66

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 or ascorbic acid or 3mg of sodium thiosulfate for each 5mg/L of residual chlorine present. Sample vials are then 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 should be sampled using traditional core sampling methods. Once the core sample is collected, additional samples should be taken using an Encore<sup>TM</sup> 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<sup>®</sup> 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 4°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 <u>never</u> be mixed or composited.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 9 of 66

# 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 shall be collected in polyethylene or glass containers and shall be 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  $4 \pm 2^{\circ}$ 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  $4 \pm 2^{\circ}$ 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 10 of 66

# 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 4°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 should be collected with minimum aeration during collection and the container should be 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 shall be identified as such on the chain of custody. Information explaining the potential hazard (i.e., corrosive, flammable, poison, etc.) shall also be listed.

### 5.15 COLLECTION OF AUXILIARY DATA

All auxiliary data shall be 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 shall be kept using local time in the military (24 hour) format and shall be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 11 of 66

# 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, Half- and Full-Face Respirators, 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 (Wildco)

App. III, Ver. 10.0 Date: April 15, 2012 Page: 12 of 66

# 7.0 WASTEWATER SAMPLING

### 7.1 SAMPLING EQUIPMENT

Туре	Use	Materials	Permissible Parameter Groups
Continuous Wastewater	Sampling	Tygon tubing; glass or plastic sample container	All parameter groups except oil & grease, extractable organics, and VOCs
Samplers-Peristaltic Pump	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 <u>NPDES Compliance Inspection</u> <u>Manual</u>. Additional guidance is given in the EPA <u>Handbook for Monitoring Industrial</u> <u>Wastewater</u>. 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 regulating 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 13 of 66

### 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 flowproportional 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 14 of 66

# 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 15 of 66

### 7.5.2 Equipment Installation

### 7.5.2.1 Conventional Sampling

Automatic samplers may be used to collect time-proportional composite or flowproportional 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 shall be used to maintain and calibrate ESC automatic samplers. Any variance from these procedures will be documented.

Proper sampler operation will be 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 shall be checked at least once. The sample volume will be calibrated using a graduated cylinder at least twice, and the flow pacer that activates the sampler shall be checked to be sure it operates properly.

Upon return from a field trip, the sampler shall be examined for damage. The operation will be checked and any required repairs will be performed and documented. The sampler will then be cleaned as outlined in Section 12.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 16 of 66

# 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 should be collected in the actual sample container that will be 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 mLs 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, sample shall be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 17 of 66

### 7.7.2 Bacterial Analysis

Samples for bacterial analysis will always be 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 prepreserved (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 side.

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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 18 of 66

# 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., will be entered in field logbooks or attached thereto.

# 8.0 SURFACE WATER AND SEDIMENT SAMPLING

# 8.1 EQUIPMENT

Equipment Type	Use	Material	Permissible Parameter Groups	
Surface Water Sampling				
Kemmerer Sampler	Depth	PVC	All parameter groups except extractable organics,	
	sampling		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	Sampling	Stainless	All parameter groups	
Container		steel		
Bailers	Sampling	Teflon <sup>®</sup>	All parameter groups	
	Sampling	PVC	All parameter groups except extractable organics,	
			VOCs, and oil & grease	
		Sediment	Sampling	
Hand Augers	Sampling	Carbon	Demand, nutrients, and extractable organics (for hard	
		Steel	packed soils only)	
Sediment Core Sampler	Sampling	Stainless	All parameter groups	
		Steel,		
		Teflon®		
Encore <sup>TM</sup>	Sampling	Teflon <sup>®</sup>	VOC Sediment/soil	
Scoops	Sampling	Teflon <sup>®</sup>	All parameter groups	
		coated		
Mixing Bowl	Compositing	Glass	All parameter groups except VOCs	
Spoons, spatula	Sampling,	Stainless	All parameter groups	
	compositing	Steel		

App. III, Ver. 10.0 Date: April 15, 2012 Page: 19 of 66

# 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 at otherwise acceptable locations and are 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 crosssectional 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 20 of 66

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 will 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 midchannel where flow is greater and less toward the banks where the flow proportionally lower.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 21 of 66

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 transaction 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 hypo-limnetic 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 22 of 66

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 will dictate 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 shall 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 23 of 66

# 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 will be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 24 of 66

### 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 raise 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 25 of 66

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  $\text{Encore}^{^{\text{TM}}}$  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  $\text{Encore}^{^{\text{TM}}}$  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 preweighed vial (with a Teflon<sup>®</sup> 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<sup>®</sup> 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 26 of 66

# 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 volatile compounds.

Only Teflon<sup>®</sup> or stainless steel should be used in sediment sampling for trace contaminant analyses. Teflon<sup>®</sup> 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 <sup>1</sup>/<sub>2</sub> 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 <sup>1</sup>/<sub>2</sub> 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 will be used to record data pertinent to sampling activities. This data shall describe all sampling locations and techniques, list 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 27 of 66

# 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 shall not be 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. 5. Label each sample container with a field number and complete a chain of custody.

# 9.0 GROUNDWATER AND DRINKING WATER SAMPLING

Equipment type	Purpose	Component(s)	Allowable Parameter Groups
Bailers (disposable	Purging	Teflon <sup>®</sup> & SS	All parameter groups
and non-disposable)	Sampling	Teflon <sup>®</sup>	All parameter groups
	Purging <sup>2</sup>	Tygon Tubing	All parameter groups except organics
Peristaltic Pump <sup>1</sup>	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

# 9.1 GROUNDWATER AND DRINKING WATER SAMPLING EQUIPMENT

<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> Dump will be alsoned after each use

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 <u>RCRA Groundwater Monitoring Technical Enforcement Guidance Document</u> (TEGD); ESC field personnel will at a minimum meet, and when possible exceed, the requirements of this document.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 29 of 66

# 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 will include depth to standing water surface and total depth of the well. This data will then be 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 will be 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 will be recorded on the Monitoring Well Data Sheet (Figure 2).

- 9.3.1 Procedure For Water Level Measurement
  - 1. Clear debris from area around well (lay plastic sheathing around well pad as an option).
  - 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 will be 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 shall be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 30 of 66

### 9.3.2 Calculating Water Volume

Total volume of standing water in a well is calculated by the following formula:

$$V = \pi r^2 h x 7.48 \text{ gallons/ft}^3$$

where;

V	=	volume of standing water in the well (gallons)
r	=	radius of well (ft)
h	=	depth of water column in the well (ft)
π	=	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.

# **ESC Lab Sciences Sampling Quality Assurance Manual** Appendix III to the ESC QAM

App. III, Ver. 10.0 Date: April 15, 2012 Page: 31 of 66

# 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 (⁰F)	рН (S.U.)	Conductivity (Tmho/cm)	Time/Date

Well casing material / diameter:

Sampled by / signature:

NOTES / CALCULATIONS:

App. III, Ver. 10.0 Date: April 15, 2012 Page: 32 of 66

Except for low recovery wells, all wells shall be 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 fuelpowered pumping units shall be placed downwind of any sampling site. If purging equipment is reused, it shall be cleaned following standard procedures. Disposable latex gloves shall be 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 on Monitoring Well Data Sheet. Discard the sample into the collection pail. Purge until the change between samples of each parameter is less than 5 percent.
  - 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 33 of 66

NOTE: For wells sampled in the State of Florida, three well volumes will be purged prior to pH, temperature, and conductivity screening. Following evacuation of three well volumes, purge water will be 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 will be maintained 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 34 of 66

### 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 shall be purged at the maximum rate for at least 15 minutes. Monitoring of field parameters will continue 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 35 of 66

ESC generally supplies sampling devices for wells sampled by ESC. However, some clients have wells equipped with dedicated sampling devices. All dedicated equipment will be cleaned between sampling events with the exception of dedicated pump systems or dedicated pipes that are never removed. ESC will evaluate the device and the project manager shall approve/disapprove of the dedicated device prior to sampling.

If sampling includes dissolved parameters, samples shall be 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 shall be preserved as required following filtration.
- 7. Unfiltered samples will be collected in conjunction with filtered samples.

**NOTE:** Filtered samples will be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 36 of 66

## 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 37 of 66

# 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 will be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 38 of 66

# 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. 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 39 of 66

# **10.0** SOIL SAMPLING

Soil samples are preserved as per Section 14. When compositing subsamples, the quantity of each subsample used shall be measured and recorded in the field logbook.

# **10.1 SAMPLING EQUIPMENT**

Туре	Use	Materials	Allowable Parameter Groups <sup>1</sup>	
Hand Auger	Sampling	PVC	All parameter groups except	
(Bucket type)			VOC's, extractables and organics	
Encore <sup>TM</sup> Sampler	VOC soil	Teflon <sup>®</sup>	VOC's only	
	subsampling			
Split Spoons	Sampling	Carbon Steel	All parameter groups	
Trowel, Spatula	Sampling and	Chrome-Plated	All parameter groups	
-	Compositing*	Steel		
Spoons	Sampling and	Stainless Steel	All parameter groups	
	Compositing*			
Shovel	Sampling	Carbon Steel	All parameter groups	
Mixing Pan	Compositing*	Pyrex & Aluminum	All parameter groups except metals	
-		-	in aluminum pan	
Carbon steel & Chrome-plated steel tools may be used for collecting soils where trace metal				

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 taken 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).

### ESC Lab Sciences Sampling Quality Assurance Manual Appendix III to the ESC OAM

App. III, Ver. 10.0 Date: April 15, 2012 Page: 40 of 66

- 6. Using a clean auger, extract the desired sample. Subsampling is performed for VOC sample collection using an Encore<sup>™</sup> sampling device. Once the core sample is collected, additional samples should be taken using an Encore<sup>™</sup> 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<sup>®</sup> 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 41 of 66

# **11.0 WASTE SAMPLING**

# 11.1 SAMPLING EQUIPMENT

Туре	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 shall be cleaned and repaired before being stored at the conclusion of a field study. Special decontamination procedures may be necessary in some instances and will be developed on a case-by-case basis. Any deviation from standard cleaning procedures and all field repairs shall be documented in field logbooks. Equipment that has not been properly cleaned must be tagged and labeled.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 42 of 66

### 11.2.2 Collection of Supplementary Information

The collection of supplementary data is important when collecting waste samples. Any field analyses shall be recorded in field logbooks. Sketches of sampling locations and layout shall be documented in the logbooks. Photographs shall be 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 shall be 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 will at least provide 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 43 of 66

### 11.3.3 Sampling Techniques

Containers containing unknown materials or known hazardous materials shall be 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 shall be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 44 of 66

11.4.2 Sampling Locations

Sampling locations should be selected that will 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 <u>Test Methods for Evaluating Solid Waste</u> (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 <u>Test Methods for Evaluating Solid Waste</u> (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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 45 of 66

# 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 will be 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 will be labeled as "dirty" or "contaminated equipment" in the field and transported separately from clean equipment.
- 4. All cleaning procedures shall be documented. Field decontamination shall be documented in the field records. These records will 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 shall be followed without modification.
- 6. All field sampling equipment shall be 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 shall be made from reagent-grade nitric acid and deionized water.

The standard cleaning solvent used will be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 46 of 66

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 shall be 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 will be 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 shall be decontaminated prior to leaving the site. This decontamination procedure shall consist 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 47 of 66

### 12.1.7 Safety Procedures For Cleaning Operations

All applicable safety procedures shall be followed during cleaning operations. The following precautions shall be taken during cleaning operations:

- Safety glasses or goggles, gloves, and protective clothing will be worn during all cleaning operations.
- Solvent rinsing operations will be conducted under a hood or in an open, well ventilated area.
- No eating, smoking, drinking, chewing, or hand to mouth contact shall be permitted during cleaning operations.
- 12.1.8 Storage of Field Equipment

All clean field equipment shall be 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 will be 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 shall determine the cause(s) of the problem(s) and take immediate corrective action.

### 12.2.2 Rinse Water

The quality of water used shall be monitored once per quarter by placing water in standard, precleaned sample containers and submitting them to the ESC laboratory for analysis. Organic-free water will also be 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 will be 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 nitrogencontaining 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 will be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 49 of 66

## 12.5 CLEANING PROCEDURES FOR AUTOMATIC SAMPLING EQUIPMENT

#### 12.5.1 General

All automatic wastewater samplers will be cleaned as follows:

- The exterior and accessible interior portions of automatic samplers will be washed with Liquinox and rinsed with tap water.
- The electronics casing will be cleaned with a clean damp cloth.
- All vinyl sample tubing will be discarded after each use.
- Teflon<sup>®</sup> tubing will be cleaned using procedures found in Section 12.6.2.
- Silastic pump tubing will be cleaned and re-used after each use, if possible. Tubing will be cleaned using cleaning procedures specified in Section 12.6.1 of this document. Tubing shall be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 50 of 66

#### 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, hand wash 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 51 of 66

#### **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 shall be 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 shall be discarded.

# 12.6.2 Teflon<sup>®</sup> Tubing

New Teflon<sup>®</sup> tubing shall be 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 shall be 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.

#### ESC Lab Sciences Sampling Quality Assurance Manual Appendix III to the ESC QAM

App. III, Ver. 10.0 Date: April 15, 2012 Page: 52 of 66

- 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 shall be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 53 of 66

- 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 will be 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 will be cleaned as described in Section 12.6.1.
  - 4. If Teflon<sup>®</sup> tubing is used, it will be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 54 of 66

#### 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 shall be decontaminated between wells in the following manner:

- 1. Interior of the pump and tubing shall be 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 shall be washed with laboratory detergent, rinsed with tap water, and air dried before storage or re-use. Extremely contaminated shipping containers shall be 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).

App. III, Ver. 10.0 Date: April 15, 2012 Page: 55 of 66

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 shall not be 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 shall 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" will be defined as within 15 minutes.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 56 of 66

Sample preservation is accomplished either by obtaining prepreserved containers from an acceptable source or by adding preservatives in the field.

It is the responsibility of the field team accepting prepreserved containers to make sure that the proper preservatives are used and desired results are achieved. The laboratory shall also supply 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 shall be at least reagent grade or higher. The acid for metals shall be suitable for trace metals analyses.
- 2. Fresh preservatives shall be 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 shall be added to 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 shall be added to the equipment or field blank.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 57 of 66

Samples requiring temperature preservation shall be 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 will be 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 shall be separated from sample containers and samples to avoid reaction and possible contamination.
- 6. Analyte free water shall be segregated from solvents to prevent contamination.

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	Stored in cabinet designated for standard and reagent storage. Stored in
standards, conductivity standards	temperature-controlled area of laboratory.
Sodium hydroxide	Stored in original container in designated cabinet in laboratory.
Sodium thiosulfate, zinc acetate,	Stored in original containers in designated area of laboratory. Reagent
ascorbic acid, lead acetate	solutions made fresh prior to use.

#### 14.4.1 Reagent and Standard Storage

App. III, Ver. 10.0 Date: April 15, 2012 Page: 58 of 66

# 14.5 SAMPLE TRANSPORT

In the majority of situations, samples will be delivered directly to the laboratory by the field sampling team or field courier following standard chain of custody protocols. Samples will be preserved immediately (i.e., within 15 minutes) and packed with ice prior to transport. The field team will relinquish 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.) shall be 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  $4 \pm 2^{\circ}$ 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 will 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
- rinse well with deionized water then rinse with dilution water

App. III, Ver. 10.0 Date: April 15, 2012 Page: 59 of 66

New glassware will be cleaned according to the same procedure as listed above except the first step will be preceded by soaking the glassware overnight in 10% HNO<sub>3</sub>. Sample collection containers used for automatic sampling devices are cleaned according to the same protocol listed above.

ESC does not reuse sample transport containers. All bottles used for sample transport are new.

# TABLE 14.6A: PRESERVATION, HOLDING TIME AND SAMPLE CONTAINERS(SOLID WASTE AND SOIL SAMPLES)

PARAMETER	PRESERVATIVE	HOLDING TIME	CONTAINER(S)
Metals	Cool, 4°C	* 6 Months	Plastic, glass
Volatile Organic Compounds in Water, Includes TPH GRO/BTEX	Cool, 4°C	14 Days	Glass, Teflon <sup>®</sup> -lined septum
Volatile Organic Compounds in Soil/Solid Includes TPH GRO/BTEX	Cool, 4°C (If using vials, then Sodium Bisulfate is used)	48 hours (using Encore <sup>™</sup> sampler) 14 Days (using pre-weighed, preserved, vials)	Encore <sup>™</sup> Sampler or Pre-weighed glass vials (Teflon <sup>®</sup> -lined septum) with magnetic stir bar
Semi-volatiles, non-volatile organics Includes TPH DRO	Cool, 4°C	14 Days until extraction, 40 days after extraction	Glass, Teflon <sup>®</sup> -lined cap
Solids	Cool, 4°C	7 Days	Plastic, glass
Cyanides	Cool, 4°C	14 Days	Glass
Oil and Grease	Cool, 4°C	28 Days	Glass, Teflon <sup>®</sup> -lined cap

\* Maximum holding time for mercury is 28 days.

# TABLE 14.6B: WASTEWATER PRESERVATION, HOLDING TIMEAND SAMPLE CONTAINERS (OTHER PARAMETERS)

Parameter	<b>Container</b> <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum Holding Time <sup>4</sup>	Required Sample Volume
		Biomonitoring		•
Biomonitoring Acute and Chronic	P, G	Cool, 4°C	36 hours to first use	Determined by analysis req. Min. 250 mL.
		Bacteriological		
Coliform, Fecal and Total Fecal Streptococci	P, G	Cool, 4°C, 0.008% $Na_2S_2O_3^5$	6 hours	150 mL
		Inorganics		
Acidity	P, G	Cool, 4°C	14 days	250 mL
Alkalinity	P, G	Cool, 4°C	14 days	250 mL
Ammonia	P, G	Cool, 4°C, $H_2SO_4$ to pH <2	28 days	500 mL Distilled-1000 mL
Biochemical Oxygen Demand	P, G	Cool, 4°C	48 hours	2000 mL

# **ESC Lab Sciences Sampling Quality Assurance Manual** Appendix III to the ESC QAM

App. III, Ver. 10.0 Date: April 15, 2012 Page: 60 of 66

Parameter	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum Holding Time <sup>4</sup>	Required Sample Volume
Bromide	P, G	None Required	28 days	200 mL
Biochemical Oxygen Demand, Carbonaceous	P, G	Cool, 4°C	48 hours	2000 mL
Chemical Oxygen Demand	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days	100 mL
Chloride	P, G	None Required	28 days	200 mL
Chlorine, Total Residual	P, G	None Required	Immediately	200 mL
Color	P, G	Cool, 4°C	48 hours	250 mL
Cyanide, Total and Amenable	P, G	Cool, 4°C, NaOH to pH >12, 0.6 g/l ascorbic acid <sup>5</sup>	14 days <sup>6</sup>	2000 mL
Fluoride	Р	None Required	28 days	100 mL
Hardness	P, G	HNO <sub>3</sub> to pH $<2$ , H <sub>2</sub> SO <sub>4</sub> to pH $<2$	6 months	100 mL
Hydrogen Ion (pH)	P, G	None Required	Immediately	100 mL
Kjeldahl and Organic Nitrogen	P, G	Cool, $4$ °C, $H_2$ SO <sub>4</sub> to pH <2	28 days	500 mL
Chromium VI	P, G	Cool, 4°C	24 hours	500 mL
Mercury <sup>7</sup>	P, G	HNO <sub>3</sub> to pH <2	28 days	500 mL
Metals <sup>7</sup> , except Chromium VI and Mercury	P, G	HNO <sub>3</sub> to pH <2	6 months	1000 mL
Nitrate	P, G	Cool, 4°C	48 hours	500 mL
Nitrate-Nitrite	P, G	Cool, $4^{\circ}$ C, $H_2$ SO <sub>4</sub> to pH <2	28 days	500 mL
Nitrite	P, G	Cool, 4°C	48 hours	200 mL
Oil and Grease	G	Cool, $4$ °C, HCl/H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days	1000 mL
Organic Carbon	P, G	Cool, $4$ °C, HCl/H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days	100 mL
Orthophosphate	P, G	Filter Immediately, Cool, 4°C	48 hours	200 mL
Oxygen, Dissolved Probe	G Bottle and Top	None Required	Immediately	Not Applicable
Phenols	G only	Cool, $4$ °C, $H_2$ SO <sub>4</sub> to pH <2	28 days	1000 mL
Phosphorus (elemental)	G	Cool, 4°C	48 hours	2000 mL
Phosphorus, Total	P, G	Cool, $4^{\circ}$ C, $H_2$ SO <sub>4</sub> to pH <2	28 days	500 mL
Residue, Total	P, G	Cool, 4°C	7 days	500 mL
Residue, Filterable	P, G	Cool, 4°C	7 days	500 mL
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days	500 mL
Residue, Settleable	P, G	Cool, 4°C	48 hours	1000 mL
Residue, Volatile	P, G	Cool, 4°C	7 days	500 mL
Specific Conductance	P, G	Cool, 4°C	28 days	500 mL
Sulfate	P, G	Cool, 4°C	28 days	500 mL
Sulfide	P, G	Cool, 4°C, add zinc acetate plus NaOH to pH >9	7 days	300 mL
Sulfite	P, G	None Required	Immediately	250 mL
Surfactants	P, G	Cool, 4°C	48 hours	500 mL
Temperature	P, G	None Required	Immediately	Not Applicable
Turbidity	P, G	Cool, 4°C	48 hours	200 mL

#### ESC Lab Sciences Sampling Quality Assurance Manual Appendix III to the ESC QAM

App. III, Ver. 10.0 Date: April 15, 2012 Page: 61 of 66

Parameter	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum Holding Time <sup>4</sup>	Required Sample Volume
		Organics <sup>8</sup>		•
Volatile Halocarbons	G, Teflon <sup>®</sup> - lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3^5$	14 days	2 x 40 mL
Volatile Aromatic Hydrocarbons	G, Teflon <sup>®</sup> - lined septum	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , HCl to pH 2 <sup>9</sup>	14 days	2 x 40 mL
Acrolein and Acrylonitrile	G, Teflon <sup>®</sup> - lined septum	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> . Adjust pH to $4-5^{10}$	14 days	2 x 40 mL
Phenols <sup>11</sup>	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3^5$	7 days to ext. then 40 days	3000 mL
Benzidines <sup>11</sup>	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3^5$	7 days to ext. <sup>13</sup>	3000 mL
Phthalate esters <sup>11</sup>	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C	7 days to ext. then 40 days	3000 mL
Nitrosamines <sup>11, 14</sup>	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3^5$ .	7 days to ext. then 40 days	3000 mL
PCBs <sup>11</sup> , Acrylonitrile	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C	7 days to ext. then 40 days	3000 mL
Nitroaromatics and Isophorone <sup>11</sup>	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3^5$ , store in dark	7 days to ext. then 40 days	3000 mL
Polynuclear Aromatic Hydrocarbons <sup>11</sup>	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3^5$ , store in dark	7 days to ext. then 40 days	3000 mL
Haloethers <sup>11</sup>	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3^5$	7 days to ext. then 40 days	3000 mL
Chlorinated Hydrocarbons <sup>11</sup>	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C	7 days to ext. then 40 days	3000 mL
TCDD <sup>11</sup>	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3^5$	7 days to ext. then 40 days	3000 mL
Pesticides <sup>11</sup>	G, Teflon <sup>®</sup> - lined cap	Cool, 4°C, pH 5-9 <sup>15</sup>	7 days to ext. then 40 days	2000 mL
Radiological Analyses: Alpha, beta and Radium	P, G	HNO <sub>3</sub> to pH <2	6 months	3000 mL

NOTES:

<sup>1</sup> Polyethylene (P) or Glass (G).

<sup>2</sup> Sample preservation should be performed immediately upon sample collection. If using an automatic sampler, preserve by maintaining at 4 deg. C until compositing and sample splitting is completed.

<sup>3</sup> Samples shipped by common carrier or sent through the United States Mail must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

<sup>4</sup> Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid for analytical and regulatory purposes. <sup>5</sup> Only to be used in the presence of precident eblering.

<sup>5</sup> Only to be used in the presence of residual chlorine.

#### ESC Lab Sciences Sampling Quality Assurance Manual Appendix III to the ESC OAM

App. III, Ver. 10.0 Date: April 15, 2012 Page: 62 of 66

<sup>6</sup> Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and pH adjusted to 12.

<sup>7</sup> Dissolved metals samples should be filtered immediately on-site before adding preservative.

<sup>8</sup> Applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

<sup>9</sup> Sample receiving no pH adjustment must be analyzed within seven days after collection.

<sup>10</sup> pH adjustment is not required if acrolein will not be measured.

<sup>11</sup> When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4° C, reducing residual chlorine, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction.

 $^{12}$  1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 plus or minus 0.2.

<sup>13</sup> Extracts may be stored up to seven days before analysis if stored in an oxidant-free atmosphere.

<sup>14</sup> For the analysis of diphenylnitrosamine, add 0.008%  $Na_2S_2O_3$  and adjust pH to 7-10 with NaOH within 24 hours of sampling.

<sup>15</sup> The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008%  $Na_2S_2O_3$ .

# 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."

App. III, Ver. 10.0 Date: April 15, 2012 Page: 63 of 66

# **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 will be collected, samples are segregated by analyses required. If multiple sites are sampled, or if specific and separate areas of interest are identified, samples will be further segregated for packaging prior to shipment.

Environmental samples shall be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 64 of 66

# **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 shall be contained on-site in temporary storage until it is characterized. Appropriate disposal and/or treatment methods will then be 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.

App. III, Ver. 10.0 Date: April 15, 2012 Page: 65 of 66

#### 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 <u>Engineering Support Branch Standard Operating Procedures and Quality Assurance</u> <u>Manual</u>, February 1, 1991, US EPA Region IV, Environmental Services Division.
- 17.2 <u>RCRA Ground-Water Monitoring Technical Enforcement Guidance Document</u> (GPO #5500000260-6), US EPA, September 1986.
- 17.3 <u>Test Methods for Evaluating Solid Waste</u>, SW-846, Third Edition, Office of Solid and Emergency Response, US EPA, November 1986.
- 17.4 <u>Methods for the Determination of Organic Compounds in Drinking Water</u>, EPA/600/4-88/039, December 1988.

#### **ESC Lab Sciences Sampling Quality Assurance Manual** Appendix III to the ESC OAM

App. III, Ver. 10.0 Date: April 15, 2012 Page: 66 of 66

- 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 <u>DER Manual for Preparing Quality Assurance Plans</u>, DER-QA-001/90, revised September 30, 1992.
- 17.7 <u>NPDES Compliance Inspection Manual</u>, United States Environmental Protection Agency, Enforcement Division, Office of Water Enforcement and Permits, EN-338, 1988.
- 17.8 <u>Handbook for Monitoring Industrial Wastewater</u>, United States Environmental Protection Agency, Technology Transfer, 1973.
- 17.9 EPA Primary Drinking Water Regulations, 40 CFR 141.
- 17.10 <u>Rapid Bioassessment Protocols For Use in Streams and Rivers</u>, United States Environmental Protection Agency, Office of Water, EPA/841/B-99-002.
- 17.11 <u>Environmental Sampling and Analysis: A Practical Guide</u>. Lawrence H. Keith, Ph.D., 1991. Lewis Publishers.
- 17.12 <u>Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to</u> <u>Freshwater and Marine Organisms</u>. Fifth Edition. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA/821/R-02/012
- 17.13 <u>Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving</u> <u>Waters to Freshwater Organisms.</u> Fourth Edition. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA/821/R-02/013.

App. IV, Ver. 10.0 Date: April 15, 2012 Page 1 of 24

**1.0** SIGNATORY APPROVALS

# WET LAB QUALITY ASSURANCE MANUAL

# APPENDIX IV 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. A signed cover page is available upon request

Judith R. Morgan, M.S. VP of Technical and Regulatory Affairs 615-773-9657

Eric Johnson, B.S., Laboratory Director 615-773-9654

()TUX

Dixie Marlin, B.S, QC Manager, 615-773-9681

Kenny Buckley, B.S., Organies/Wet Chemistry Department Manager, 615-773-9686

# 2.0 APPENDIX TABLE OF CONTENTS

Section	Section Title			Rev. Date	<i>Rev.</i> #
1.0	Approval and Signature Page	Page	1	4/15/11	2
2.0	Table of Contents	Page	2	4/15/11	2
3.0	Scope and Application	Page	3	4/15/11	2
4.0	Laboratory Organization and Responsibility	Page	3	4/15/11	2
5.0	Personnel and Training	Page	3	4/15/11	2
6.0	Facilities and Laboratory Safety	Page	3	4/15/11	2
7.0	Sampling Procedures	Page	4	4/15/11	2
8.0	Equipment	Page	4	4/15/11	2
9.0	Laboratory Practices	Page	13	4/15/11	2
10.0	Analytical Procedures	Page	14	4/15/11	2
11.0	Quality Control Checks	Page	15	4/15/11	2
12.0	Data Reduction, Validation and Reporting	Page	16	4/15/11	2
13.0	Corrective Actions	Page	19	4/15/11	2
14.0	Record Keeping	Page	22	4/15/11	2
15.0	Quality Audits	Page	22	4/15/11	2
	TABLES				
8.1	Equipment	Page	4	4/15/11	2
8.2	Equipment Preventative Maintenance,	Page	6	4/15/11	2
0.2	Equipment Calibration	1 age	0		
8.3A	Standards and Reagents	Page	7	4/15/11	2
8.3B	Working Standards	Page	8	4/15/11	2
8.3C	Standardization of Titration Solutions	Page	9	4/15/11	2
8.5	Instrument Calibration	Page	12	4/15/11	2
10.1	Wet Lab Department SOPs	Page	14	4/15/11	2
12.1	Data Reduction Formulas	Page	16	4/15/11	2
12.3	QC Targets and RLs	Page	17	4/15/11	2

# **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, 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 8.0*.

# 5.0 PERSONNEL AND TRAINING

# 5.1 **PERSONNEL**

Kenneth W. Buckley, with a B.S. degree in General Science, is the Department Manager of the Organics and Wet Chemistry laboratories. Mr. Buckley reviews and approves all data reduction associated with analyses in these areas and is responsible for the overall production of these laboratories; including the management of the staff and scheduling. Mr. Buckley has over 12 years of environmental laboratory experience. In his absence, Chad Pfalmer assumes responsibility for departmental decisions in the Wet Lab.

### 5.2 TRAINING

5.2.1 All new analysts to the laboratory are trained by a primary analyst or Manager according to ESC protocol. 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. 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 and Safety 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 Wet Lab environmental analyses include groundwater, wastewater, drinking water, soil, and sludge. The Wet Lab also performs analyses on sorbent media and air filters for Industrial Hygiene monitoring.
- 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 E	-	T: MAJOR ITEN	MS – Wet Lab				
This table is subject to revision without notice							
Item	Manufacturer	Model	Instrument Name	Serial #	Location		
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	6090000341	Wet Lab		
Autoanalyzer	Lachat	Quikchem 8500	Lachat 5	6090000342	Wet Lab		
Autoanalyzer	Lachat	Quikchem 8500	Lachat 6	70500000452	Wet Lab		
Autoanalyzer - digestor	Lachat	BD-46	DIG1	10070000-982	Wet Lab		
Autoanalyzer - digestor	Lachat	BD-46	DIG2	1000700000- 982	Wet Lab		
Autoanalyzer - digestor	Lachat	BD-46	DIG1	1800-871	Wet Lab		
Autoanalyzer - digestor	Lachat	BD-46	DIG2	1800-872	Wet Lab		
Automated distiller	Skalar	SAN++ system	Kelada 1	9719	Wet Lab		
Automated titrator	Metrohm	855 titrosampler	Titrando	3256	Wet Lab		
Centrifuge	Thermo	Megafuge 40	Centrifuge	41123868	Wet Lab		
Class "I" 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		

# **ESC Lab Sciences Wet Lab Quality Assurance Manual** Appendix IV to the ESC QAM

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	R07002510B	Wet Lab
Flash Point Tester	Koehler	Pensky- Martens K16201	Manual	R07002697B	Wet Lab
Hot Plate	Thermolyne Fisher	Туре 2200	Hot	16237	Wet Lab
Hot Plate	Thermolyne Fisher	Туре 2200	Hot	16240	Wet Lab
Ion Chromatograph	Dionex	ICS-2000	IC5	6050731	Wet Lab
Ion Chromatograph	Metrohm	850 Professional	IC2	185000003190	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
Muffle Furnace	Thermolyne	(1) 30400	FURNACE	23231	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

#### ESC Lab Sciences Wet Lab Quality Assurance Manual Appendix IV to the ESC QAM

		_	_		_
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
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 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
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

# 8.3 STANDARDS 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.

Table 8.3	Table 8.3A: Standard sources, description and calibration information.         This table is subject to revision without notice							
Instrument Group	Standard Source	How Received*	Source/ Storage	Preparation from Source	Lab Stock Storage	Preparation Frequency		
Alkalinity, Acidity	Lab preparation	Acidity-matrix standard grade KHP	Room temp.	0.0500N	$4^{\circ} \pm 2^{\circ} C$	6 months		
Ammonia-Nitrogen and Total Kjeldahl Nitrogen	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				Working Standards	Not stored	Prepared fresh as needed		
BOD	Lab preparation	As dry glucose and glutamic acid	Dessicator	150mg of each/L	$4^{\circ} \pm 2^{\circ} C$	Made fresh daily		
COD	Lab preparation	Acid grade KHP	Dessicator	Stock solution (10,000ppm)	$4^{\circ} \pm 2^{\circ}C$	When absorbance of curve changes or check samples are out of control		
Cyanide (Autoanalyzer)	Lab preparation	KCN	Reagent shelf	Stock solution (1,000ppm)	$4^{\circ} \pm 2^{\circ} C$	6 months. Working dilutions prepared daily as needed		
Fluoride	Inorganic Standard. NSI Lab preparation	ACS grade KF	Room temp.	100ppm stock solution	Room temp.	l year or as needed when reference standard fails		
Fluoride	<b>.</b>			Dilute standards	Not stored	Prepared fresh daily		
Hardness	Lab preparation	Chelometric Std. CaCO <sub>3</sub>	Room temp.	1mg/mL as CaCO3	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	$4^{\circ} \pm 2^{\circ} C$	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	$4^{\circ} \pm 2^{\circ} C$	Midpoint standard prepared weekly or sooner if necessary		
IC (Chloride, Nitrate, Nitrite, Bromide, Sulfate, Fluoride)	NSI (2nd source)	Varies	4°±2°C	Working Standards as needed per analyte	$4^{\circ} \pm 2^{\circ} C$	Prepared weekly or sooner if necessary		
MBAS	Lab preparation	LAS Reference Material	$4^{\circ} \pm 2^{\circ} C$	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		

Table 8.3A: Standard sources, description and calibration information.         This table is subject to revision without notice							
Instrument Group	Standard Source	How Received*	Source/ Storage	Preparation from Source	Lab Stock Storage	Preparation Frequency	
						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)	$4^{\circ} \pm 2^{\circ} C$	Every month. Working solutions prepared daily as needed.	
Phosphate	(H2O) - Prepared in Lab Total Phos. (soils) RICCA, ERA	KH2PO4	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 Na2SO4	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	

# **ESC Lab Sciences Wet Lab Quality Assurance Manual** Appendix IV to the ESC QAM

TABLE 8.3B: WORKING STANDARD CALIBRATION			
Analysis	Calibration Standard		
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		
BOD	D.OBarometric pressure/temp., Glucose and Glutamic acid reference		
	standard.		
Bromate IC	Low Range - 5.0, 10, 20, 30, 50, 100 ug/L		
Bromide IC	Range -1.0, 5.0, 10, 50, 100, mg/L		
Chlorate IC	Low Range - 5.0, 10, 20, 30, 50, 100 ug/L		
	High Range – 10, 20, 50, 100, 200, 400, 600 ug/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		
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	Buffers1.0, 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 (Colormetric)	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		

TABLE 8.3C: STANDARDIZATION OF TITRATION SOLUTIONS				
Solution	Primary Standard	Frequency		
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	6 months or with each new batch		
	(from KHP standard)			
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 INSTRUMENT CALIBRATION

#### Total Organic Carbon Analyzer (TOC) – 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  $\pm 15\%$  of the expected concentration.

#### 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

Quadratic Fit is the primary method of quantitation; however Linear Regression is required for sample analyzed in conjunction with the Ohio VAP program. When using quadratic fit a minimum of six standards are used. If linear regression is used for quantitation, 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 15% of the expected concentration for soil samples.

#### 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, NO2NO3 where  $\pm 10\%$  applies.

### Gravimetric Analyses – Various SOPs

Gravimetric analyses are performed using several different published methods, including TDS, TSS, TVDS, TS, TVS, VSS, Settleable Solids, Total Particulates, 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.

### 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.

App. IV, Ver. 10.0 Date: April 15, 2012 Page 13 of 24

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.

# 8.5 ACCEPTANCE/REJECTION OF CALIBRATION

The initial calibration curve is compared with previous curves for the same analyte. The curve is checked for linearity and the response must be within 10% of the previous curve. 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.

#### **ESC Lab Sciences** Wet Lab Quality Assurance Manual Appendix IV to the ESC QAM

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	Calculation of cell constant between 0.95 - 1.05	Daily as used
	Continuing	1	point	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)	Linear	Must be within 5% of true value	Every 10th sample; Field**
	Initial	At least 5 standards calibration standards		Calibration Curve must have a correlation of 0.995 or better	Daily as used
UV/VIS Spec.		2 laboratory control standard	Linear	Must be within <u>+</u> 15% of the calibration curve.	Daily as used
	Continuing	1 mid-level reference std.		Must be within 90 – 110%	Every 10th sample
Total	Initial	3 calibration standards		Calibration Curve must have a correlation of 0.995 or better	Daily as used
Organic Halogen Analyzer		1 laboratory control standard	Linear	Laboratory control standard must agree within $\pm$ 15% of calibration curve	Daily as used
	Continuing	1 mid-level reference std.		Must be within 90 – 110%	Every 10th sample
Total Organic Carbon Analyzer	Initial	5 calibration standards		Calibration Curve must have a correlation of 0.995 or better	Every 6 months or as needed
		2 laboratory control standard	Linear	Laboratory control standard must agree within <u>+</u> 15% of calibration curve	Daily as used
	Continuing	1 mid-level reference std.		Must be within 90 – 110%	Every 10th sample

#### **TABLE 8.5: INSTRUMENT CALIBRATION**

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 **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 GLASSWARE WASHING AND STERILIZATION PROCEDURES

#### <u>General</u>

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, BOD, 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.

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.

# **10.0** ANALYTICAL PROCEDURES

10.1 A list of laboratory SOPs associated with the Wet Lab can be found in the following table:

	This table is subject to revision without notice		
SOP #	Title		
340300	Acidity		
340301	Alkalinity (Titrimetric)		
340302	Alkalinity - Lachat		
340303	Biochemical Oxygen Demand		
340305	Chlorine, Total Residual		
340306	Corrosivity		
340307	Cyanide- All Forms (Colorimetric Automated UV) - Lachat		
340309	Chemical Oxygen Demand		
340310	Color by Visual Comparison		
340313	Density (Specific Gravity)		
340317	Total Hardness by Lachat		
340317	Total Hardness (mg/l as CaCO3) - (Titrimetric)		
340318	Hexavalent Chromium (Colorimetric) Water/Soil		
340319	Ion Chromatography - Anions		
340325	MBAS (Methylene Blue Active Substances)		
340327	Ammonia, Phenolate (Lachat)		
340328	Organic Nitrogen		
340331	Threshold Odor Test		
340333	Nitrate/Nitrite (Lachat Autoanalyzer)		
340334	Paint Filter Test		
340335	рН		
340336	Phenol - 4AAP (Lachat Autoanalyzer)		
340338	Orthophosphate Colorimetric		
340338	Total Phos. Colorimetric		
340339	Reactivity		
340340	Reactive Cyanide/Sulfide Distillation		
340342	Specific Conductance		

**TABLE 10.1: WET LAB DEPARTMENT SOPs** 

#### ESC Lab Sciences Wet Lab Quality Assurance Manual Appendix IV to the ESC QAM

SOP #	Title		
340344	Sulfide (Colorimetric Methylene Blue)		
340344	Sulfide Acid-soluble, and acid-insoluble		
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		
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)		
340357	Ignitability		
340357	Ignitability		
340359	UV254		
340360	TOX (total organic halides)		
340361	Ferrous Iron		
340362	Heat of Combustion		
340365	Particles Not Otherwise Regulated, Total (PNOR)		
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		
340372	Hexavalent Chromium in Water by IC		
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		
340377	Manual Nitrocellulose Analysis		
340378	Volatile Suspended Solids		
340379	Guanidine Nitrate by IC		

# **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 Environmental Resource Associates (ERA). 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*. The Quality Control Department performs the secondary review of the data package using the ESC SOP #030227, *Data Review*. The QC Reviewer verifies that the analysis has 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.)

PARAMETER	FORMULA
Acidity, Alkalinity	<u>mL titrant x normality titrant x 50,000</u>
	mL sample
BOD, 5-day	<u>Initial D.O Final D.O CF</u>
	% Dilution Sample
	Calculations are performed by computer software
Boron, COD, Sulfate	Concentration from curve x dilution factor
Nitrogen-Nitrate, Nitrite, Nitrogen-	Calculated by computer software as provided by Lachat Corp.
Nitrite, Ortho and Total Phosphate,	
Phenols, Chloride	
Fluoride**, Nitrogen-Ammonia**,	Calculated by computer software as provided by Lachat Corp.
Nitrogen-Total Kjeldahl**	
Anions	Calculated by computer software as provided by Dionex
Conductivity*, pH, Turbidity,	Directly read from instrument
Cyanide, Total and Amenable	µg from standard curve x mL total volume absorbing solution
	mL volume sample x mL volume of absorbing solution colored
	Calculated by software as provided by Lachat Corp.
Solids, Total and Total Dissolved	((mg wt of dried residue + dish) - mg wt of dish) x 1000
	mL sample
Solids, Total Suspended	((mg wt of dried residue + filter) - mg wt of filter) x 1000
	mL sample

#### TABLE 12.1: Data Reduction Formulas

### **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, controls and current reporting limits.

### **12.3 Reporting**

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.

App. IV, Ver. 10.0 Date: April 15, 2012 Page 20 of 24

Table 12.3: QC Targets for Wet Lab Accuracy (LCS), Precision and RLs           This table is subject to revision without notice						
Analyte	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)	
Acidity	SM 2310B	W	85 - 115	<20	1000	
Alkalinity	SM 2320	W	85 - 115	<20	10000	
Ammonia	350.1, SM 4500- NH3-H	W	-90-110	<20	100	
Ammonia	350.1 (mod.)	S	Certified Values	<20	500	
Bromide	300.0/9056/9056A	W	90 - 110	<20	1000	
Bromide	SM 4110B	W	90 - 110	<20	1000	
Bromide	300.0	S	Certified Values	<20	10000	
Chloride	300.0/9056/9056A	W	90 - 110	<20	1000	
Chloride	SM 4110B	W	90 - 110	<20	1000	
Chloride	300.0	S	Certified Values	<20	10000	
Color	SM 2120-E	W	n/a	<20	1 CU	
Conductivity	120.1/9050A, 2510	W	85 - 115	<20	1000	
Cyanide	335.3, 335.4, 335.2 (CLP-M), 9012A	W	90 - 110	<20	5	
Cyanide	SM 4500-CN-E	W	90 - 110	<20	5	
Cyanide	EPA 9012A	S	Certified Values	<20	250	
Ferrous Iron	3500FE B	W	85 - 115	<20	50	
Fluoride	300.0/9056/9056A	W	90 - 110	<20	100	
Fluoride	SM 4110B	W	90 - 110	<20	100	
Fluoride	9056A	S	Certified Values	<20	1000	
Hardness	130.1	W	85 - 115	<20	30000	
Hardness	SM 2340	W	85 - 115	<20	1000	
Hexavalent Chromium	SM3500 CrD/7196A	W	85 - 115	<20	10	
Hexavalent Chromium	7196A	S	Certified Values	<20	2000	
Ignitability	1010	WS	<u>+</u> 3 degrees C	<20	n/a	
Methylene Blue Active Substances	5540C SM20 <sup>th</sup>	w	85 - 115	<20	100	
Nitrate-Nitrite	300	W	90 - 110	<20	100	
Nitrate-Nitrite	SM 4110B	W	85 - 115	<20	100	
Nitrate-Nitrite	9056/9056A	W	90-110	<20	100	
Nitrate-Nitrite	9056/9056A	S	Certified Values	<20	1000	
Nitrite	300.0/9056/9056A	W	90 - 110	<20	100	
Nitrite	SM 4110B	W	90 - 110	<20	100	
Nitrite	300.0/9056/9056A	S	Certified Values	<20	1000	
Nitrate	300.0/9056/9056A	W	90 - 110	<20	1000	
Nitrate	SM 4110B	w	90 - 110	<20	100	
Nitrate	300.0/9056/9056A	s	Certified Values	<20	1000	
Moisture	Karl Fisher	WS	n/a	<20		
pH	SM 4500-H, 9040B	W	n/a	<1	n/a	
pH	9045C	s	n/a n/a	<1	n/a	

App. IV, Ver. 10.0 Date: April 15, 2012 Page 21 of 24

Table 12.3: QC Targets for Wet Lab Accuracy (LCS), Precision and RLs           This table is subject to revision without notice						
Analyte	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)	
Phosphate (ortho)	SM 4500-P	W	85 - 115	<20	25	
Phosphorous/Total	365.4, SM 4500-P	W	-90-110	<20	25	
Phosphorous/Total	365.4	S	Certified Values	<20	1000	
Phosphorous/Total	9056/9056A	S	Certified Values	<20	1000	
Residual Chlorine	SM 4500Cl G 20th	W	90 - 110	<20	100	
Residue, Total (TS)	SM 2540-B, SM2540-G	W	85 - 115	<20	1000	
Residue, Filterable (TDS)	SM 2540-C	W	95 - 105	<20	1000	
Residue Non-Filterable (TSS)	SM 2540-D	W	95 - 105	<20	1000	
Residue, Total Volatile (TVS)	160.4, SM 2540-E, SM2540-G	w,s	80 - 120	<20	1000	
Sulfate	300.0/9056/9056A	W	90 - 110	<20	5000	
Sulfate	SM 4110-B	W	90 -110	<20	5000	
Sulfate	300.0/9056/9056A	S	Certified Values	<20	50000	
Sulfide	SM 4500S2 D 20th	w	85 - 115	<20	100	
Sulfite	SM 4500-SO3	W	85 - 115	<20	500	
Total Kjeldahl Nitrogen	351.2	W	-90-110	<20	500	
Total Kjeldahl Nitrogen	351.2	S	Certified Values	<20	50000	
Total Organic Carbon	415.1, SM 5310B&C, 9060	w	85 - 115	<20	1000	
Total Organic Carbon	LOI	S	Certified Values	<20	10000	
Dissolved Organic Carbon	415.1, SM 5310B&C, 9060	W	85 - 115	<20	1000	
Total Inorganic Carbon	415.1, SM 5310B&C, 9060	W	85 - 115	<20	1000	
Total Organic Halogens	9020A, SM 5320B	W	85 - 115	<20	10	
EOX	9023	S	85 - 115	<20	20000	
Total Phenol	420.2	W	85 - 115	<20	50	
Total Phenol	9066	s, ws	Certified Values	<20	50	
Turbidity	180.1, SM 2130	W	n/a	<20	1 NTU	

### **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 reason for the nonconformance 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 QA 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 take precedence.

13.2.2 Calibration Verification Criteria Are Not Met: Inorganic Analysis

Rejection Criteria - See Table 8.5.

<u>Corrective Action</u> - 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 will be made fresh, intermediate dilutions will be re-checked and the instrument will be re-calibrated. If a problem persists, the group supervisor or QA 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 group supervisor or QA Department is notified for further action.

13.2.3 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

<u>Rejection Criteria</u> - Blank reading is more than twice the background absorbance or more than 1/2 RL.

<u>Corrective Action</u> - 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 group leader or QA Department will be 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)

<u>Rejection Criteria</u> - 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  $\pm$  3 times the standard deviation of those points. (Listed in Section 12).

<u>Corrective Action</u> - 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 group leader, lab supervisor, or QA Department will be consulted for further action.

13.2.5 Out Of Control Matrix Spike Samples

<u>Rejection Criteria</u> - If either the MS or MSD sample is outside the established control limits from accuracy charts on matrix spike samples of a similar matrix (i.e., water, solid, etc). Limits are calculated as the mean  $\pm$  three times the standard deviations.

<u>Corrective Action</u> - Spiking technique is assessed to ascertain if the sample has been spiked correctly. The spiked sample should be 1 - 5 times the concentration of the client sample; otherwise, the percent recovery (%R) or relative percent difference (RPD) of the MS/MSD should be flagged as not meaningful or usable The sample is re-spiked and re-analyzed, along with several other similar samples in subset. If an "out of control" situation persists, sample matrix interference is indicated. Samples to be analyzed by standard additions are prepared (where appropriate), and the group leader, lab supervisor, or QA Department is notified.

13.2.6 Out Of Control Duplicate Samples

<u>Rejection Criteria</u> - Lab-generated maximum RPD limit (as listed under precision in Section 12)

<u>Corrective Action</u> - Instrument and samples 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, lab supervisor or QA Department is notified to review the analytical techniques.

13.2.7 Out Of Control Matrix Spike Duplicates

These QC samples can be out of control for accuracy, precision, or both. The appropriate corrective actions listed for either matrix spikes, duplicate samples, or both are followed.

Analysis-specific corrective action lists are available for each type of analysis performed by ESC.

13.2.8 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

<u>Corrective Action</u> - 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 group leader, lab supervisor, or QA Department will be consulted for further action.

# 14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103 Document Control and Distribution, SOP #030203 Reagent Logs and Records and SOP #030201 Data Handling and Reporting

All calibration data and graphs generated for wet chemistry are kept in a calibration notebook 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 8.0.

App. V, Ver. 10.0 Date: April 15, 2012 Page 1 of 24

**1.0** SIGNATORY APPROVALS

# Metals Department QUALITY ASSURANCE MANUAL

# APPENDIX V 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. A signed cover page is available upon request

organ.

S. VP of Technical and Regulatory Affairs 615-773-9657

Eric Johnson, B.S., Laboratory Director 615-773-9654

KITO.A

Dixie Marlin, B.S, QC Manager, 615-773-9681

James Burns, B.S., Metals Department Manager 615-773-9685

# 2.0 APPENDIX TABLE OF CONTENTS

Section	Section Title			Rev. Date	Rev.#
1.0	Approval and Signature Page	Page	1	4/15/11	2
2.0	Table of Contents	Page	2	4/15/11	2
3.0	Scope and Application	Page	3	4/15/11	2
4.0	Laboratory Organization and Responsibilities	Page	3	4/15/11	2
5.0	Personnel and Training	Page	3	4/15/11	2
6.0	Facilities and Laboratory Safety	Page	4	4/15/11	2
7.0	Sampling Procedures	Page	4	4/15/11	2
8.0	Equipment	Page	5	4/15/11	2
9.0	Laboratory Practices	Page	12	4/15/11	2
10.0	Analytical Procedures	Page	13	4/15/11	2
11.0	Quality Control Checks	Page	13	4/15/11	2
12.0	Data Reduction, Validation, and Reporting	Page	14	4/15/11	2
13.0	Corrective Actions	Page	21	4/15/11	2
14.0	Record Keeping	Page	24	4/15/11	2
15.0	Quality Audits	Page	24	4/15/11	2
	TABLES				
8.1	Equipment	Page	5	4/15/11	2
8.2	Equipment Preventative Maintenance,	Page	6	4/15/11	2
0.2	Equipment Calibration	rage	0		
8.3A	Stock Standard Sources, & Receipt	Page	7	4/15/11	2
8.3B	Working Standard Sources & Prep	Page	8	4/15/11	2
8.4	General Calibration Standard Conc.	Page	10	4/15/11	2
8.5	Instrument Calibration	Page	11	4/15/11	2
10.1	Metals Department SOPs	Page	13	4/15/11	2
12.3A	QC Targets Environmental Metals and RLs	Page	15	4/15/11	2
12.3B	QC Targets for IH Metals and RLs	Page	21	4/15/11	2

### 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 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 8.0*.

### 5.0 PERSONNEL AND TRAINING

### 5.1 **PERSONNEL**

James Burns, with a B.S. degree in Medical Technology, is the Department Manager of the Metals Laboratory. Mr. Burns reviews and approves all data reduction associated with metals analysis. Scheduling for analyses and personnel are his responsibility and is paramount to achieving success and quality analyses of samples. His responsibilities also include the coordination with clients' analytical needs regarding regulatory compliance. Mr. Burns has previous experience with numerous regulatory agencies including: USACE, DOD, DOE, NAVY, AFCEE and CLP. Additionally, he has also been involved in waste management/disposal and has held the position of Radiation Safety Officer. In his absence, LaKeia Layne assume responsibility for departmental decisions.

### 5.2 TRAINING

The primary analyst or Manager trains all new analysts to the laboratory according to ESC protocol. 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 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 approximately1200 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 and Safety 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 and plastic 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 0.45µm 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 are stored at  $4 \pm 2^{\circ}$ C and do not require acid preservation.
  - Hold times for all metals, except Mercury, are 180 days. Mercury has a hold time of 28 days.

# 8.0 EQUIPMENT

#### <u>Instrument Software</u>

- PE ELAN ICPMS PE ICP Winlab Used for calibration, calculation, QC review, diagnostics, data storage
- Perkin Elmer ICP Optima DV PE ICP 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.

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Metals Analysis and Preparation This table is subject to revision without notice							
Item	Manufacturer	Model	Name	#	Serial number	Location	
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	Mercury Lab	
Hot Block	СРІ	Mod Block	А	1	NA	Metals Prep Lab	
Hot Block	Env. Express	SC154	С	1	3994CEC1880	Metals Prep Lab	
ICPMS with autosampler	Perkin Elmer	ELAN DRC-e ASX-520	ICPMS4	1	AH13650804	Metals Lab	
ICPMS with autosampler	Perkin Elmer	ELAN DRC-e ASX-510	ICPMS3	1	AH00110504H	Metals Lab	
ICPMS with autosampler	Perkin Elmer	ELAN 9000 ASX-510	ICPMS5	1	AJ12270805	Metals Lab	
ICPMS with autosampler	Perkin Elmer	ELAN DRC II ASX-510	ICPMS6	1	AI13820805H	Metals Lab	
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 4300DV ASX-520	ICP3	1	077NO110301	Metals Lab	
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 5300DV ASX-510	ICP5	1	077N5041802	Metals Lab	
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 5300DV ASX-510	ICP6	1	077N5091002	Metals Lab	
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 5300DV ASX-510	ICP7	1	077C6110602	Metals Lab	
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 7300	ICP8	1	077C0111203	Metals Lab	
Hot Block	CPI	Mod Block	NA	1	004412	Mercury Lab	
Hot Block	CPI	Mod Block	NA	1	604443	Mercury Lab	
Hot Block	CPI	Mod Block	MPA	1	4430	Mercury Lab	
Hot Block	CPI	Mod Block	MPB	1	4434	Mercury Lab	

# 8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Metals Analysis and Preparation This table is subject to revision without notice						
Item	Manufacturer	Model	Name	#	Serial number	Location
Mercury Auto Analyzer	Perkin Elmer	(2) FIMS 400	Ι	1	4545	Mercury Lab
Mercury Auto Analyzer	Perkin Elmer	(1) FIMS 100	III	1	110156051101	Mercury Lab
Mercury Auto Sampler	Perkin Elmer	(1) AS-91, (1) AS-93, (1) S10	NA	1	NA	Mercury Lab
Mercury Auto Sampler	Perkin Elmer	FIMS 100	NA	1	101S11061403	Mercury Lab
Microwave	CEM	MARS 5	NA	1	DS-8025	Metals Prep 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
Prep Station	Env. Express	Automated prep station	Autobloc k 1	1	1243	Metals Prep Lab
Prep Station	Env. Express	Automated prep station	Autobloc k 2	2	1783	Metals Prep Lab
TCLP Extraction Unit	Env. Express	6 Position	NA	1	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	5	4809-12-542	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	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	22	NA	TCLP Lab
Turbidimeter	НАСН	2100N		1	05090C020685	Metals Prep Lab
Water Purification - Nanopure	Barnstead	D11951		1	1372051120948	Metals Prep Lab
PH Meter	Orion	410A	NA	1	015683	TCLP Lab
Balance	Mettler Toledo			1	1128150150	TCLP Lab
Auto pipetters 1000µl to 20 µl	Oxford	Varies	NA		NA	Metals Lab
Auto pipetters	Eppendorf, Oxford	Varies	NA		NA	Metals Prep Lab
Drying Oven	VWR Scientific	1305U	NA	a	1000594	Metals Prep Lab
MAX/MIN Thermometer	VWR	MAX/MIN	TCLP #1		NA	TCLP Lab
MAX/MIN Thermometer		MAX/MIN	TCLP #2		NA	TCLP Lab

# 8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
INSTRUMENT	P. WI. DESCRIPTION	FREQUENCI

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	<ul> <li>Calibrated according to manufacturers instructions.</li> <li>The slope is documented and acceptable range 95-105%</li> </ul>	Daily
Analytical Balance	<ul> <li>Analytical balances are checked and calibrated by a certified technician semi-annually.</li> <li>Calibration is checked daily with class S weights. Must be within 0.1% S class weights calibrated annually</li> </ul>	Semi-annually Daily
TCLP Tumblers	•Visually timed and confirmed to be 30±2 rpm.	Monthly
Microwaves	•Checked and calibrated by a certified technician	Sami annually calibrated weekly
Microwaves	Check cap membranes for leaks	As needed

# 8.3 STANDARDS 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)					
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 (Sulfur is analyzed individually) *ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn					
Instrument Group/Standard	Standard Source*	How Received*	Source/ Storage	Lab Stock Storage	Receipt Frequency
ICP (single element standards)	Env. Express or High Purity	1000ppm	Room temp.		Annual/Expiration Date
ICP/ICV		500ppm – Al. Ca, Fe, Mg, Na, K 5ppm – Ag 50ppm – All others	Room temp.	5% HNO3 w/ Tr HF	As needed

Tabl	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 (Sulfur is analyzed individually) *ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn						
Instrument Group/Standard	Standard Source*	How Received*	Source/ Storage	Lab Stock Storage	Receipt Frequency		
ICP/Calibration Standard and CCV	Env. Express	1000ppm – Al, Ca, Fe, K, Mg, Na 10ppm – Ag 100ppm – All others	Room temp.	5% HNO3 w/ Tr HF	As needed		
ICP/LCS water	Ultra Scientific	1000ppm – Ca, Mg, K, Na 100ppm – all others except Li (spiked separately)	Room temp.	5% HNO3	As needed		
ICP/LCS soil	ERA	Varies with Lot #	Room temp.	none	As needed		
ICP/ICSA	Env. Express	5000ppm – Al, Ca, Mg, Na 2000ppm – Fe 100ppm – K	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		
ICPMS/ICV	High Purity	5 ppm	Room temp.	5% HNO3 w/ Tr HF	As needed		
ICPMS/ Calibration Standard and CCV	Env. Express	100 ppm	Room temp.	5% HNO3 w/ Tr HF	As needed		
ICPMS/LCS water	Ultra Scientific	1000ppm – Ca, Mg, K, Na 100ppm – all others except Li (spiked separately)	Room temp.	5% HNO3	As needed		
ICPMS/LCS soil	ERA	Varies with Lot #	Room temp.	none	As needed		
ICPMS/ICSA	Env. Express	10000ppm – Cl 2000ppm – C 1000ppm – Al, Ca, Fe, Mg, P, K, Na, S 20ppm – Mo, Ti	Room temp.	10% HNO3	As needed		
ICP/ICSB	Env. Express	2ppm – Sb, As, Be, Ca, Cr, Co, Cu, Pb, Ni, Se, Ag, Tl, Sn, Zn	Room temp.	4% 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 (Sulfur is analyzed individually) \*ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn

Instrument Group/Standard	How Prepared	Final Concentration	Source/ Storage	Expiration
ICP/ICV	10mL Custom Stock ICV A and B, 0.1 mL stock Sc adjusted to 100mL with 5% HNO3	50ppm – Al, Ca, Fe, K, Mg, Na 0.5ppm – Ag 2ppm - Sr 5ppm – All others	Room temp.	1 month
ICP/Calibration Standard	Std 6 – 10mL Stock Cal. Std. Std 5 – 1mL Stock Cal. Std. Std 4 – 1mL Std. 6 Std 3 – 1mL Std. 5 Std 2 – 0.5mL Std. 5 Std 1 – 2mL Std. 4 All adjusted to 100 mL with 5%HNO3	Std 6 $- 1/10/1000$ ppm Std 5 $- 0.1/1/10$ ppm Std 4 $- 0.01/0.1/1$ ppm Std 3 $- 0.01/0.1$ ppm Std 2 $- 0.005$ ppm Std 1 $- 0.002$ ppm	Room temp.	1 month
ICP/CCV	50mL Custom Stock CCV adjusted to 1000mL with 5% HNO3	50ppm – Al, Ca, Fe, K, Mg, Na 0.5ppm – Ag 5ppm – All others	Room temp.	1 month
ICP/ICSA	100mL Custom Stock ICSA adjusted to 1000mL with 5% HNO3	500ppm – Al, Ca, Mg, Na 200ppm – Fe 10ppm – K	Room temp.	1 month
ICP/ICSAB	100mL Custom Stock ICSA, 10mL Stock ICSAB adjusted to 1000mL with 5% HNO3	500ppm – Al, Ca, Mg, Na 200ppm – Fe 10ppm – K 1ppm – B, Cd, Pb, Ag, Ni, Si, Zn, 0.5ppm – all others except Sr, Li	Room temp.	1 month
ICP/Yttrium	5mL Stock Yttrium adjusted to 10L with 5% HNO3	5 ppm	Room temp.	1 month
ICPMS/ICV	1.0mL Stock ICV adjusted to 100mL with 5% HNO3	0.05 ppm	Room temp.	1 month
ICPMS/ Calibration Standard	0.1 Stock Cal Std adjusted to 100mL with 5%HNO3. Serial Dilutions are done each calibration from 0.1ppm Std.	Cal 5 – 0.1ppm Cal 4 – 0.05ppm Cal 3 – 0.01ppm Cal 2 – 0.001ppm Cal 1 – 0.0005ppm	Room temp.	1 month
ICPMS/CCV	0.05mL Stock CCV adjusted to 100mL with 5% HNO3.	0.050 ppm	Room temp.	1 month
ICPMS/ICSA	10mL Stock ICSA adjusted to 100mL with 5% HNO3	1000ppm – Cl 200ppm – C 100ppm – Al, Ca, Fe, Mg, P, K, Na, S 2ppm – Mo, Ti	Room temp.	1 month
ICPMS/ICSAB	10mL Stock ICSA, 1mL Stock ICSAB adjusted to 100mL with 5% HNO3	1000ppm – Cl 200ppm – C 100ppm – Al, Ca, Fe, Mg, P, K, Na, S 2ppm – Mo, Ti 0.02ppm – Sb, As, Be, Ca, Cr, Co, Cu, Pb, Ni, Se, Ag, Tl, Sn, Zn	Room temp.	1 Month
Hg/ICV	30µL of 3ppm Intermediate	0.003ppm – Hg	Room temp.	1 Month
Hg/Calibration Standard	<ul> <li>Std 6 - 100μL of 3ppm Intermediate</li> <li>Std 5 - 50μL of 3ppm Intermediate</li> <li>Std 4 - 200μL of 300ppb Intermediate</li> <li>Std 3 - 100μL of 300ppb Intermediate</li> <li>Std 2 - 40μL of 300ppb Intermediate</li> </ul>	Std 6 – 0.01ppm Std 5 – 0.005ppm Std 4 – 0.002ppm Std 3 – 0.001ppm Std 2 – 0.0004ppm	Room temp.	4 days

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 (Sulfur is analyzed individually)         *ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn					
	Std 1 - 20µL of 300ppb Intermediate	Std 1 – 0.0002ppm			
Hg/CCV	2.5ppb CCV - 25µL of 3ppm Intermediate	0.0025ppm	Room temp.	1 Month	
Hg/LCS	30µL of 3ppm Intermediate	0.003ppm – Hg	Room temp.	1 Month	

### 8.4 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  $\geq 0.998$ . 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. Duplicate and 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 - SOP Numbers 340386 & 340390

The PE ICP Optima 4300DV, 5300DV and PE ELAN 6100 and DRC-e ICPMS 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 0.998 or better for all methods, 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.

TABLI	TABLE 8.4: CALIBRATION STANDARD CONCENTRATIONS           This table is subject to revision without notice							
HIGH LEVEL	ICP (mg/L)	ICP/MS (mg/L)						
Aluminum	0.10 - 100							
Antimony	0.01 - 10	0.0005 - 0.05						
Arsenic	0.01 - 10	0.0005 - 0.10						
Barium	0.005 - 10	0.0005 - 0.10						
Beryllium	0.002 - 10	0.0005 - 0.01						
Boron	0.10 - 10							
Cadmium	0.005 - 10	0.0005 - 0.10						
Calcium	0.10 - 100							
Chromium	0.01 - 10	0.0005 - 0.10						
Cobalt	0.01 - 10	0.0005 - 0.10						
Copper	0.01 - 10	0.0005 - 0.10						
Iron	0.10-100							
Lead	0.005 - 10	0.0005 - 0.10						
Lithium	0.005 - 10							
Magnesium	0.10 - 100							
Manganese	0.010 - 10	0.0005 - 0.10						
Molybdenum	0.002 - 10	0.0005 - 0.10						
Nickel	0.01 - 10	0.0005 - 0.10						
Potassium	0.50 - 100							
Selenium	0.01 - 10	0.0005 - 0.10						
Silicon	0.10 - 10							
Silver	0.01 - 1.0	0.0005 - 0.05						
Sodium	0.50 - 100							
Strontium	0.002 - 10							
Sulfur	10 - 100							
Thallium	0.01 - 10	0.0005 - 0.05						
Tin	0.01 - 10	0.0005 - 0.10						
Titanium	0.01 - 10							
Vanadium	0.01 - 10	0.0005 - 0.10						
Zinc	0.010 - 10	0.001 - 0.10						
MERCURY								
Mercury	Blank, 0.2 - 0.010 µg/L							

### 8.5 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 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 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	6010C, 6020A, 6010B, 6020, 200.7 200.8: 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	< 1/2 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 10th sample							
ICP & ICPMS	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, No criteria for ICPMS	After initial calibration, at end and every 8 hours of run time.							
ICP, ICPMS, Mercury	Continuing	1 Method Blank	< 1/2 RL, concentrations of common laboratory contaminants must not exceed the RL	1 per batch							
ICP, ICPMS, Mercury	Continuing	1 Laboratory Control Standard	Liquid Samples (all methods) - LCS must be within <u>+</u> 15%. Solid Samples (all methods) - LCS must be within the certified standard value determined by the provider.	1 per batch							
ICP, ICPMS, Mercury	Continuing	1 Sample Duplicate	Sample and Duplicate must have an RPD $\leq 20\%$	1 per batch							

	TABLE 8.5 INSTRUMENT CALIBRATION & QC											
Instrument (Analysis)	Calibration Type	Number of Standards	Acceptance/ Rejection Criteria	Frequency								
ICP & ICPMS	Continuing	1 Matrix Spike (MS), 1 Matrix Spike Duplicate (MSD)	Spike must be within ±25%, MS and MSD must have an RPD ≤20%	1 of each per batch								
Mercury	Linear/ Initial	3 - 5	Must have a correlation coefficient of at least 0.998	Daily								
Mercury	Initial	Secondary source (ICV)	7470A, 7471: ICV must be within <u>+</u> 10% 245.1: ICV must be within <u>+</u> 5%	After initial calibration								
Mercury	Continuing	1 Continuing Calibration Blank	< RL	Every 10 <sup>th</sup> sample								
Mercury	Continuing	1 Matrix Spike (MS), 1 Matrix Spike Duplicate (MSD)	Spike must be within ±30%, MS and MSD must have an RPD <u>&lt;</u> 20%	1 of each per batch								

# 9.0 LABORATORY PRACTICES

### 9.1 REAGENT GRADE WATER

ASTM Type I grade water.

# 9.2 GLASSWARE WASHING AND STERILIZATION PROCEDURES

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.

SOP #	Title
	TCLP SOPs
340358	TCLP
340362	SPLP
340363	EP TOX
340364	MEP
340705	California Waste Extraction Test
	Mercury SOPs

# TABLE 10.1: METALS DEPARTMENT SOPS

SOP #	Title
340384A	Mercury in Liquid Waste (Cold-Vapor Technique) 7470A/245.1
340384B	Mercury in Solid Waste (Cold-Vapor Technique) 7471A
	Metals Prep SOPs
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
340701	Metals Digestion of personal cassettes Method 7300, 3051
340702	Metals Digestion for Sediments, Soils, and Sludge NIOSH 7300, Method 3051 for
340702	ELLAP Paint chips and ELLAP soils
340703	Metals Digestion of Hi-Vol filters and Environmental Lead
540705	Wipes 3050B and 3051
340391	Silver (Photographic Waste) Method 7760 and 272.1
340392	Sodium Adsorption Ratio
	Metals Analysis SOPs
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 Environmental Resource Associates (ERA). 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, including lead in air, by NIOSH 7300 is completed every quarter. Soil, wipes and paint PTs are also completed in conjunction with the AIHA Environmental Lead Laboratory Accreditation Program (ELLAP). AIHA 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 Sample Duplicates, 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%.

App. V, Ver. 10.0 Date: April 15, 2012 Page 15 of 25

- 11.4 A laboratory control sample (LCS) is analyzed one per batch of samples. The acceptance criteria for all water samples is  $\pm 15\%$ . See certificate of analysis for soil true values. For Industrial Hygiene samples, the LCS is analyzed in duplicate per batch.
- 11.5 A method preparation blank is performed per batch of samples processed. If one-half 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 is 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*. The Quality Control Department performs the secondary review of the data package using the ESC SOP #030227, *Data Review*. The QC Reviewer verifies that the analysis has 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 1	Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs										
	(subject to revision without notice)										
Class	Analyte	Prep	Analysis	Matrix	Accuracy Range	Precision	RL				
		Method	Method		(%)	(RPD)	(ppb)				

Table 1	Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs         (subject to revision without notice)										
Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)				
(ICP-AES)	Aluminum	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000				
(ICP-AES)	Aluminum	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000				
(ICP-AES)	Aluminum	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	100				
(ICP-AES)	Aluminum	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	100				
(ICP-MS)	Antimony	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50				
(ICP-AES)	Antimony	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000				
(ICP-AES)	Antimony	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000				
(ICP-MS)	Antimony	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1				
(ICP-MS)	Antimony	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1				
(ICP-AES)	Antimony	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20				
(ICP-AES)	Antimony	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20				
(ICP-MS)	Arsenic	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50				
(ICP-AES)	Arsenic	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000				
(ICP-AES)	Arsenic	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000				
(ICP-AES)	Arsenic	1311, 1312	6010B/C	Leachate	85 - 115	<20	50				
(ICP-MS)	Arsenic	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1				
(ICP-MS)	Arsenic	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1				
(ICP-AES)	Arsenic	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20				
(ICP-AES)	Arsenic	NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20				
(ICP-MS)	Barium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	100				
(ICP-AES)	Barium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	250				
(ICP-AES)	Barium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	250				
(ICP-AES)	Barium	1311-12	6010B/C	Leachate	85 - 115	<20	150				
(ICP-AES)	Barium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	5				
(ICP-MS)	Barium	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	2				
(ICP-MS)	Barium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	2				
(ICP-AES)	Barium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	5				
(ICP-MS)	Beryllium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50				
(ICP-MS)	Beryllium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1				
(ICP-MS)	Beryllium	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1				
(ICP-AES)	Beryllium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	100				

Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs         (subject to revision without notice)										
Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)			
(ICP-AES)	Beryllium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	100			
(ICP-AES)	Beryllium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	2			
(ICP-AES)	Beryllium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	2			
(ICP-AES)	Boron	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	10000			
(ICP-AES)	Boron	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	10000			
(ICP-AES)	Boron	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	200			
(ICP-AES	Boron	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	200			
(ICP-MS)	Cadmium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	25			
(ICP-AES)	Cadmium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	250			
(ICP-AES)	Cadmium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	250			
(ICP-AES)	Cadmium	1311-1312	6010B/C	Leachate	85 - 115	<20	50			
(ICP-MS)	Cadmium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.5			
(ICP-MS)	Cadmium	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	0.5			
(ICP-AES)	Cadmium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	5			
(ICP-AES)	Cadmium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	5			
(ICP-AES)	Calcium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000			
(ICP-AES)	Calcium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000			
(ICP-AES)	Calcium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	500			
(ICP-AES)	Calcium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	500			
(ICP-MS)	Chromium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50			
(ICP-AES)	Chromium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500			
(ICP-AES)	Chromium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500			
(ICP-AES)	Chromium	1311-12	6010B/C	Leachate	85 - 115	<20	50			
(ICP-MS)	Chromium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1			
(ICP-MS)	Chromium	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1			
(ICP-AES)	Chromium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10			
(ICP-AES)	Chromium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10			
(ICP-MS)	Cobalt	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50			
(ICP-AES)	Cobalt	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500			
(ICP-AES)	Cobalt	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500			
(ICP-MS)	Cobalt	200.2 (mod.),	200.8	Liquid/Aqueous	85 - 115	<20	1			

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
		NPDES					
(ICP-MS)	Cobalt	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Cobalt	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Cobalt	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-MS)	Copper	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Copper	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Copper	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Copper	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Copper	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Copper	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Copper	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Copper	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Iron	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000
(ICP-AES)	Iron	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000
(ICP-AES)	Iron	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	100
(ICP-AES)	Iron	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	100
(ICP-MS)	Lead	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Lead	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	250
(ICP-AES)	Lead	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	250
(ICP-AES)	Lead	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Lead	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Lead	200.2 (mod.)	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Lead	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	5
(ICP-AES)	Lead	NPDES	200.7	Liquid/Aqueous	85 - 115	<20	5
(ICP-AES)	Lithium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	750
(ICP-AES)	Lithium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	750
(ICP-AES)	Lithium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	15
(ICP-AES)	Lithium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	15
(ICP-AES)	Magnesium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000
(ICP-AES)	Magnesium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000
(ICP-AES)	Magnesium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	100

Table	Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs         (subject to revision without notice)										
Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)				
(ICP-AES)	Magnesium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	100				
(ICP-MS)	Manganese	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	100				
(ICP-AES)	Manganese	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500				
(ICP-AES)	Manganese	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500				
(ICP-MS)	Manganese	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	2				
(ICP-MS)	Manganese	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	2				
(ICP-AES)	Manganese	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10				
(ICP-AES)	Manganese	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10				
(CVAA)	Mercury	7471 (mod.)	7471	Solid	Certified Standard Values	<20	20				
(CVAA)	Mercury	1311-12	7470A	Leachate	85 - 115	<20	1				
(CVAA)	Mercury	245.1 (mod.)/7470A	245.1/7470A	Liquid/Aqueous	85 - 115	<20	0.2				
(ICP-MS)	Molybdenum	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	100				
(ICP-AES)	Molybdenum	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	250				
(ICP-AES)	Molybdenum	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	250				
(ICP-MS)	Molybdenum	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	2				
(ICP-MS)	Molybdenum	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	2				
(ICP-AES)	Molybdenum	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	5				
(ICP-AES)	Molybdenum	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	5				
(ICP-MS)	Nickel	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50				
(ICP-AES)	Nickel	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000				
(ICP-AES)	Nickel	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000				
(ICP-AES)	Nickel	1311-12	6010B/C	Leachate	85 - 115	<20	50				
(ICP-AES)	Nickel	3015/3010 (mod)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1				
(ICP-MS)	Nickel	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1				
(ICP-AES)	Nickel	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20				
(ICP-AES)	Nickel	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20				
(ICP-AES)	Potassium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000				
(ICP-AES)	Potassium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000				
(ICP-AES)	Potassium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	500				
(ICP-AES)	Potassium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	500				
(ICP-MS)	Selenium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard	<20	50				

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
					Values		
(ICP-AES)	Selenium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Selenium	3051 (mod.),	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Selenium	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Selenium	3015/3010 (mod.)	6020/A	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Selenium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Selenium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Selenium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Silicon	3050B (mod.)	6010B/C	Solid	85-115	<20	10000
(ICP-AES)	Silicon	3051 (mod.)	6010B/C	Solid	85-115	<20	10000
(ICP-AES)	Silicon	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	200
(ICP-AES)	Silicon	200.2 (mod.) NPDES	200.7	Liquid/Aqueous	85 - 115	<20	200
(ICP-MS)	Silver	3050B (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	25
(ICP-AES)	Silver	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Silver	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Silver	3015/3010 (mod.)	6020/A	Liquid/Aqueous	85 - 115	<20	0.5
(ICP-MS)	Silver	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.5
(ICP-AES)	Silver	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Silver	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Sodium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000
(ICP-AES)	Sodium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000
(ICP-AES)	Sodium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	500
(ICP-AES)	Sodium	200.2 (mod.) NPDES	200.7	Liquid/Aqueous	85 - 115	<20	500
(ICP-AES)	Strontium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Strontium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Strontium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Strontium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Sulfur	3050B (mod.)	6010B/C	Solid	85-115	<20	50000
(ICP-AES)	Sulfur	3051 (mod.)	6010B/C	Solid	85-115	<20	50000
(ICP-AES)	Sulfur	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	1000
(ICP-AES)	Sulfur	200.2 (mod.) NPDES	200.7	Liquid/Aqueous	85 - 115	<20	1000
(ICP-MS)	Thallium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard	<20	50

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
					Values		(TT <sup>13</sup> )
(ICP-AES)	Thallium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Thallium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Thallium	3015/3010 (mod.)	6020/A	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Thallium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Thallium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Thallium	NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20
(ICP-MS)	Tin	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Tin	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Tin	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-MS)	Tin	3015/3010 (mod.)	6020/A	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Tin	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Tin	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Tin	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Titanium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Titanium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Titanium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Titanium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-MS)	Vanadium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	100
(ICP-AES)	Vanadium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Vanadium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-MS)	Vanadium	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	2
(ICP-MS)	Vanadium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	2
(ICP-AES)	Vanadium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Vanadium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-MS)	Zinc	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	500
(ICP-AES)	Zinc	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1500
(ICP-AES)	Zinc	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1500
(ICP-AES)	Zinc	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Zinc	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	10
(ICP-MS)	Zinc	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	10

Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RLs         (subject to revision without notice)								
Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)	
(ICP-AES)	Zinc	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	30	
(ICP-AES)	Zinc	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	30	

Table 12.3B: QC Targets for IH Metals Accuracy (LCS), Precision and RLs								
	(subject to revision without notice)							
Class	Analyte	Prep	Analysis	Matrix	Accuracy Range	Precision	RL	
		Method	Method		(%)	(% RPD)		
(ICP-AES)	Lead	3050B (mod.)	6010B/C	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	

# **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 reason for the nonconformance 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 QA 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.

<u>Corrective Action</u> - 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 Manager or QA 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 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 Manager or QA department is notified for further action.

13.2.3 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

<u>Rejection Criteria</u> - Blank reading is more than <sup>1</sup>/<sub>2</sub> the RL for Method Blanks and/or Instrument Blanks.

<u>Corrective Action</u> - 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 group leader or QA department is consulted to determine if data needs to be rejected or if samples need to be reprepped.

13.2.4 Out Of Control Laboratory Control Standards (LCS)

<u>Rejection Criteria</u> - If the performance is outside of lab-generated control (Listed in Table 12.3).

<u>Corrective Action</u> - 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 group leader, Department Manager, or QA department is consulted for further action.

#### 13.2.5 Out Of Control Matrix Spike Samples

<u>Rejection Criteria</u> - If spike recovery is outside of lab-generated control limits determined from accuracy charts on matrix spike samples from a similar matrix (i.e., water, solid, etc).

<u>Corrective Action</u> - Spiking technique is assessed to ascertain if the sample has been spiked correctly. The spiked sample should be 1-5 times the client sample concentration; otherwise, the percent recovery (%R) or relative percent difference (%RPD) of the MS/MSD is flagged as not meaningful or usable per the EPA method. The sample is reanalyzed. If an out of control situation persists, sample matrix interference is suspected and flagged.

13.2.6 Out Of Control Duplicate Samples

<u>Rejection Criteria</u> - Lab-generated maximum RPD limit (as listed under precision in Table 12.3)

<u>Corrective Action</u> - Instrument and samples checked to see if precision variance is likely (i.e., high suspended solids content, high viscosity, etc.). The duplicates are re-analyzed along with the parent sample. If problem persists, matrix interference is suspected and flagged

13.2.7 Out Of Control Matrix Spike Duplicates

These QC samples can be out of control for either accuracy, precision, or both. The appropriate corrective actions listed for either matrix spikes, duplicate samples, or both are followed.

**NOTE**: Some samples cannot be duplicated. This is the case for wipe samples, filters, and some water samples. When possible, sampling personnel should collect duplicate samples.

Analysis-specific corrective action lists are available for each type of analysis performed by ESC.

13.2.8 Out Of Control Calibration Standards: ICV, CCV, SSCV

<u>Rejection Criteria</u> - If the performance is outside of method requirements.

<u>Corrective Action</u> - 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 group leader, Department Manager, or QA department is consulted for further action.

- 13.3 Responsibility It is the Department Manager's responsibility to evaluate the validity of the corrective action response and submit it to QA department for processing. In addition, the manager 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 Manager
  - Technical Service Representative

### 14.0 RECORD KEEPING

App. VI, Ver. 10.0 Date: April 15, 2012 Page 1 of 29

**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. A signed cover page is available upon request

Judith R. Morgan, M.S., VP of Technical and Regulatory Affairs 615-773-9657

Eric Johnson, B.S., Laboratory Director 615-773-9654

Dixie Marlin, B.S, QC Manager, 615-773-9681

Kenny Buckley, B.S., Organics/Wet Chemistry Department Manager, 615-773-9686

App. VI, Ver. 10.0 Date: April 15, 2012 Page 2 of 29

# 2.0 APPENDIX TABLE OF CONTENTS

Section	Section Title			Rev. Date	<i>Rev.</i> #
1.0	Approval and Signature Page	Page	1	4/15/11	2
2.0	Table of Contents	Page	2	4/15/11	2
3.0	Scope and Application	Page	3	4/15/11	2
4.0	Laboratory Organization and Responsibility	Page	3	4/15/11	2
5.0	Personnel and Training	Page	3	4/15/11	2
6.0	Facilities and Laboratory Safety	Page	3	4/15/11	2
7.0	Sampling Procedures	Page	4	4/15/11	2
8.0	Equipment	Page	5	4/15/11	2
9.0	Laboratory Practices	Page	14	4/15/11	2
10.0	Analytical Procedures	Page	14	4/15/11	2
11.0	Quality Control Checks	Page	15	4/15/11	2
12.0	Data Reduction, Validation and Reporting	Page	15	4/15/11	2
13.0	Corrective Actions	Page	25	4/15/11	2
14.0	Record Keeping	Page	27	4/15/11	2
15.0	Quality Audits	Page	28	4/15/11	2
	TABLES				
8.1	Equipment	Page	5	4/15/11	2
8.2	Equipment Preventative Maintenance, Equipment Calibration	Page	6	4/15/11	2
8.3A	Standards and Reagents	Page	7	4/15/11	2
8.3B	Working Standards	Page	7	4/15/11	2
8.5	Instrument Calibration	Page	13	4/15/11	2
10.1	Semi-Volatile Department SOPs	Page	14	4/15/11	2
12.1	Data Reduction Formulas	Page	16	4/15/11	2
12.3	QC Targets and RLs	Page	17	4/15/11	2

### 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 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 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 8.0*.

### 5.0 PERSONNEL AND TRAINING

### 5.1 **PERSONNEL**

Kenneth W. Buckley, with a B.S. degree in General Science, is the Department Manager of Organics and Wet Chemistry laboratories. Mr. Buckley reviews and approves all data reduction associated with analyses in these areas and is responsible for the overall production of these laboratories; including the management of the staff and scheduling. Mr. Buckley has 12 years of environmental laboratory experience. In his absence, J. D. Gentry, with a B.S. degree in Chemistry and 11 years of environmental laboratory experience, assumes responsibility for Volatiles Department decisions.

### 5.2 TRAINING

5.2.1 All new analysts to the laboratory are trained by a primary analyst or Manager according to ESC protocol. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in VOC 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.

App. VI, Ver. 10.0 Date: April 15, 2012 Page 4 of 29

### 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. The laboratory reagent water is created by reverse osmosis/DI filtration and evaluated to 0.055uS/cm to ensure purity. 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 and Safety 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 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

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Volatiles Analysis This table is subject to revision without notice							
Item	Manufacturer	Model	Instrument Name	#	Serial #	Location	
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	8	3033A31856	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/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	1	GC336A50093 MS3329A00703	Volatiles	
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5975 MSD	VOCMS	2	GCCN10641044 MSUS63234371	Volatiles	
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	3	GC3310A48625 MS3435A01982	Volatiles	
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	5	GC3310A48625 MS3341A01200	Volatiles	
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	6890 GC/ 5973 MSD	VOCMS	6	CN10343037 US44647141	Volatiles	
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	9	GC3308A46997 MS3609A03629	Volatiles	
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	10	GC2921A22675 MS3329A00524	Volatiles	
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	12	GC3336A51994 MS3549A03312	Volatiles	
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5971 MSD	VOCMS	11	GC3336A61599 MS3306A04478	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			VOCMS	8	GCUS00040221 MS03940725	Volatiles	
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	13	GCCN103390006 MSUS91911078	Volatiles	

### **ESC Lab Sciences Volatiles Laboratory Quality Assurance Manual** Appendix VI to the ESC QAM

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Volatiles Analysis This table is subject to revision without notice							
Item	Manufacturer	Model	Instrument Name	#	Serial #	Location	
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	17	GCUS10232130 MSUS03940744	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 MS71236616	Volatiles	
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	
Centurion Autosampler	(8) PTS/EST	Centurion				Volatiles	
Autosampler	(27) Varian	Archon				Volatiles	
Autosampler	Tekmar	Solatek 72				Volatiles	
Purge and Trap	(16) OI Analytical	Eclipse				Volatiles	
Purge and Trap	(14) PTS/EST	Encon				Volatiles	

8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION						
INSTRUMENT	P. M. DESCRIPTION	FREQUENCY				
Analytical Balances	•Check with Class "I" weights	Daily; tolerance <u>+</u> 0.1%				
Analytical Balances	•Service/Calibration (semiannual contract	Semiannually				

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY	
	maintenance and calibration check)		
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	
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 **STANDARDS AND REAGENTS**

Table 8.3A: Standard stock sources, description and calibration information.           This table is subject to revision without notice					
Method	Vendor*	Description	Calibration	Storage Req.	Expiration
	NSI Gases Mix		Primary	-10°C to -20°C	1 week
	NSI	Custom VOC Mix1	Primary	-10°C to -20°C	6 months
	NSI	Mix 2	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
	Absolute Stds	n-Hexane	Primary	-10°C to -20°C	6 months
	Restek	TX TPH Mix (GRO)	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
	Ultra	CUS-5661	Primary	-10°C to -20°C	6 months
8260	NSI	Custom Std	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
8200	Absolute Std	Acrolein	Primary	$4^{\circ} \pm 2^{\circ}C$	3 months
	NSI	2-CEVE	Secondary	$4^{\circ} \pm 2^{\circ}C$	6 months
	Restek	Vinyl Acetate	Secondary	-10°C to -20°C	6 months
	Restek	Custom LCS Additions	Secondary	-10°C to -20°C	6 months
	Restek	Custome Voa LCS Mix 1	Secondary	-10°C to -20°C	6 months
	Absolute Stds	n-Hexane	Secondary	-10°C to -20°C	6 months
	Restek	Acrolein	Secondary	$4^{\circ} \pm 2^{\circ}C$	3 months
8015	Restek	Certified BTEX in Unleaded Gas Composite Standard	Primary	$4^{\circ} \pm 2^{\circ} C$	6 months
(GRO)	NSI	Gas Composite	Secondary	$4^{\circ} \pm 2^{\circ}C$	6 months
8021	Restek	estek WISC PVOC/GRO Mix		-10°C to -20°C	6 months
8021 NSI		PVOC/GRO Mix	Secondary	$4^{\circ} \pm 2^{\circ}C$	6 months
VDU	NSI	VPH ICV MIX	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
VPH	NSI	VPH LSC MIX	Secondary	$4^{\circ} \pm 2^{\circ}C$	6 months

\*Equivalent Providers may be utilized.

TABLE 8.3B: Working Standard Concentrations         This table is subject to revision without notice				
ORGANIC COMPOUNDS	Method #	GC/MS	GC	
VOCs by GC/MS	524.2, 624, SM6200B 20 <sup>th</sup> , 8260B	GW/WW 0.5, 1, 2, 5, 10, 25, 40, 50, 100 μg/L DW 0.5, 1, 2, 5, 10, 25, 50, 100, 150 μg/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 8021 GRO 8015 or state specific		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, 11 mg/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, 2.0 mg/L Aliphatic C5-C8: 0.006, 0.012, 0.06, 0.12, 0.3, 0.6, 1.2, 2.4, 6.0, 12.0 mg/L Aliphatic C9-C12: 0.007, 0.014, 0.07, 0.14, 0.36, 0.7, 1.4, 2.8, 7.0, 14.0 mg/L	
BTEX/OA1	BTEX OA1		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, 11 mg/L	

### 8.4 INSTRUMENT CALIBRATION

### <u>602 - BTEX - SOP Number 330351</u>

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 DOD 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.

During the analytical sequence, the stability of the initial calibration is verified, following every  $10^{\text{th}}$  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 DOD 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  $\pm 15\%$ , 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.

### 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 determinative SOP. 8015GRO analysis, 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 +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 15% 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.

### <u>8260B/C, 624, SM6200B, 524.2 - Gas Chromatography/Mass Spectrometry (GC/MS):</u> Volatile Organics - SOP Numbers 330363 & 330364

Detector mass calibration is performed daily 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 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	0% to 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 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:

<15% RSD for methods 8260B/C and SM6200B, ≤20% RSD for method 524.2, and ≤35% RSD for method 624.

The calibration checks compounds (CCCs) for method 8260 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. Per the analytical method, specific target analytes are defined as calibration check compounds (CCCs) or system performance check compounds (SPCCs).

Linear regression can be used for any target compound exceeding the 15% RSD criteria but less than 40% (poor performers <50%), if the correlation coefficient is 0.990 or better. For USACE projects the correlation coefficient must meet 0.995 or better. The same is true for the CCCs as long as the RSD does not exceed 30%. A second source calibration verification standard is analyzed after each calibration and should meet the criteria of  $\pm$  20%. For 524.2 the second source calibration verification standard must be within  $\pm$  30%.

SPCCs:		
Analyte	Minimum Average Response Factor	
Chloromethane	0.10	
1,1-Dichloroethane	0.10	
Bromoform	0.10	
Chlorobenzene	0.30	
1,1,2,2-Tetrachloroethane	0.30	

CC	Cs:
1,1-Dicholoethene	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 20% 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 limits. 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, 524.2 & SM6200B and 24 hours for 624). Following the expiration of the tuning clock, the instrument must be retuned and either recalibrated or existing calibration may be re-verified.

For 8260B, 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, 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 the minimum response factors listed above. The CCC 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 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.

Dichlorofluoromethane	Vinyl acetate
Bromomethane	trans-1,4-Dichloro-2-butene
Chloroethane.	Alcohols (Ethanol, TBA, TAA, ETBA, TBF,
Chloroethane.	Butanol)
2,2-Dichloropropane.	Iodomethane.
1,2-Dibromo-3-chloropropane	Naphthalene
2-Chloroethylvinylether (2-CEVE)	2- Methylnaphthalene
Acrolein	1- Methylnaphthalene
Acetone	4-Methyl-2-pentanone
2-Butanone	2-Hexanone

Poor performing compounds for 8260B/524.2/SM6200B/624:

### 8.5 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**

Instrume nt (Analysis)	Calibration Type	Minimum Number of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
	Initial	3 –600 series 5 –All others	Avg. RF	Must be ≤10% RSD for 601/602, ≤20%RSD for 8021B, and ≤20% difference for 8015B	As needed
	Second Source	1 Second Source		+/- 20% of true value	With each calibration
GC (VOC)	Daily / Cont.	1/10	External	Must be within 15% of the initial calibration curve	Beginning, every 10 and ending
		1	Internal	Must be within 15% of the initial calibration curve	Every 12 hours
	Initial	5 –8000 series	Avg. RF	8260B - Must be $\leq 15$ %RSD for all target analytes and $\leq 30\%$ for CCCs	As needed
GC/MS VOC 8260	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
8200	Daily / Cont.	Tune & CCV every 12 hours		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.	Every 12 hours
	Initial	3 –600 series	Avg. RF	624 - Must be $\leq$ 35 %RSD for all target analytes and $\leq$ 30% for CCCs	As needed
GC/MS VOC 624	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
624	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**

ASTM Type I grade water.

### 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. Volatiles spargers are kept on the autosampler at all times. Between runs, spargers are cleaned with a distilled water rinse. When a highly contaminated sample is purged, a blank is analyzed in the sparger before another sample can be purged in it. If the sparger is contaminated, it is removed from the autosampler and cleaned with soap and water then a methanol rinse followed by heating to drive off any remaining volatile contaminants. The sparger is then returned to its position and a blank analysis is performed. If the blank proves to be contaminant free, the system is then ready for further field sample analysis.

### **10.0** ANALYTICAL PROCEDURES

10.1 A list of laboratory SOPs associated with the volatiles laboratory can be found in the following table:

This table is subject to revision without notice		
SOP #	Title	
330351	BTEX and Gasoline Range Organics by Gas Chromatography (8015B)	
330351A	TNGRO	
330351B	BTEXM (8021B)	
330354	NC - Volatile Petroleum Hydrocarbons	
330357	Volatile Organic Compounds (GRO by GCMS)	
330362	8021B (601/602) Volatile Organic Compounds by Gas Chromatography	
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	
330751	5035 Closed System Purge and Trap and Extraction for VOCs in Soil and Waste	
330752	5030B Purge and Trap for Aqueous Samples	

**TABLE 10.1: VOLATILE DEPARTMENT SOPS** 

### 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 Environmental Resource Associates (ERA). 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 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 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*. The Quality Control Department performs the secondary review of the data package using the ESC SOP #030227, *Data Review*. The QC Reviewer verifies that the analysis has 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.)

PARAMETER	FORMULA
GC	$\frac{\text{response of sample analyte } \{area\} \text{ x final extract volume } \{mL\} \text{ x dilution} \\ \text{response factor } \{area/(mg/L)\} \text{ x initial extract volume-mass } \{mL \text{ or } g\} \\ Calculations \text{ performed by HP Enviroquant Software} \end{cases}$
GC/MS	$\frac{\text{response of analyte } \{area\} \text{ x extract volume } \{mL\} \text{ x dilution x int. std amt. } \{area\} \\ \text{response factor } \{area/(mg/mL)\} \text{ x initial volume-mass } \{mL \text{ or } g\} \text{ x int. std cal. } \{area\} \\ \text{Calculations performed by HP Enviroquant Software} \end{cases}$

### **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.

<u>Marginal Excedence</u> – 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.

Г

App. VI, Ver. 10.0 Date: April 15, 2012 Page 18 of 29

٦

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	

<u>Organic Control Limits</u> - The organic QC targets are statutory in nature; warning and control limits for organic analyses are initially set 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	Propene	8260B/C, 624, 6200B	GW, WW			0.0025	mg/L				
Volatiles	1,3-Butadiene	8260B/C, 624, 6200B	GW, WW			0.001	mg/L				
Volatiles	4-Ethyltoluene	8260B/C, 624, 6200B	GW, WW			0.001	mg/L				
Volatiles	Dicyclopentadiene	8260B/C, 624, 6200B	GW, WW			0.001	mg/L				
Volatiles	Dichlorodifluoromethane	8260B/C, 624, 6200B	GW, WW	39-189	24	0.001	mg/L				
Volatiles	Chloromethane	8260B/C, 624, 6200B	GW, WW	45-152	20	0.001	mg/L				
Volatiles	Vinyl Chloride	8260B/C, 624, 6200B	GW, WW	55-153	20	0.001	mg/L				
Volatiles	Bromomethane	8260B/C, 624, 6200B	GW, WW	45-175	20	0.001	mg/L				
Volatiles	Chloroethane	8260B/C, 624, 6200B	GW, WW	49-155	20	0.001	mg/L				
Volatiles	Trichlorofluoromethane	8260B/C, 624, 6200B	GW, WW	54-156	20	0.001	mg/L				
Volatiles	Ethyl Ether	8260B/C, 624, 6200B	GW, WW	60-142	20	0.001	mg/L				
Volatiles	Acrolein	8260B/C, 624, 6200B	GW, WW	6-182	39	0.050	mg/L				
Volatiles	1,1-Dichloroethene	8260B/C, 624, 6200B	GW, WW	60-130	20	0.001	mg/L				
Volatiles	1,1,2-Trichloro-1,2,2- trifluoroethane	8260B/C, 624, 6200B	GW, WW	51-149	20	0.001	mg/L				
Volatiles	Acetone	8260B/C, 624, 6200B	GW, WW	48-134	20	0.050	mg/L				
Volatiles	Iodomethane	8260B/C, 624, 6200B	GW, WW	61-148	20	0.050	mg/L				
Volatiles	Carbon Disulfide	8260B/C, 624, 6200B	GW, WW	41-148	20	0.001	mg/L				
Volatiles	Methylene Chloride	8260B/C, 624, 6200B	GW, WW	64-125	20	0.005	mg/L				

App. VI, Ver. 10.0 Date: April 15, 2012 Page 19 of 29

		rgets for Volatiles Accu table is subject to revisi			and RLs		
Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	Acrylonitrile	8260B/C, 624, 6200B	-	60-140	20	0.050	mg/L
Volatiles	trans-1,2-Dichloroethene	8260B/C, 624, 6200B		67-129	20	0.001	mg/L
Volatiles	Methyl Tert Butyl Ether	8260B/C, 624, 6200B		51-142	20	0.001	mg/L
Volatiles	1,1-Dichloroethane	8260B/C, 624, 6200B	GW, WW	67-133	20	0.001	mg/L
Volatiles	Vinyl Acetate	8260B/C, 624, 6200B	GW, WW	34-178	26	0.050	mg/L
Volatiles	Di Isopropyl Ether	8260B/C, 624, 6200B		63-139	20	0.001	mg/L
Volatiles	2,2-Dichloropropane	8260B/C, 624, 6200B	GW, WW	46-151	20	0.001	mg/L
Volatiles	cis-1,2-Dichloroethene	8260B/C, 624, 6200B	GW, WW	72-128	20	0.001	mg/L
Volatiles	2-Butanone (MEK)	8260B/C, 624, 6200B	GW, WW	53-132	20	0.050	mg/L
Volatiles	Bromochloromethane	8260B/C, 624, 6200B	GW, WW	75-128	20	0.001	mg/L
Volatiles	Tetrahydrofuran	8260B/C, 624, 6200B	GW, WW	50-140	20	0.001	mg/L
Volatiles	Chloroform	8260B/C, 624, 6200B	GW, WW	66-126	20	0.005	mg/L
Volatiles	1,1,1-Trichloroethane	8260B/C, 624, 6200B	GW, WW	67-137	20	0.001	mg/kg
Volatiles	Carbon Tetrachloride	8260B/C, 624, 6200B	GW, WW	64-141	20	0.001	mg/kg
Volatiles	1,1-Dichloropropene	8260B/C, 624, 6200B	GW, WW	68-132	20	0.001	mg/kg
Volatiles	Benzene	8260B/C, 624, 6200B	GW, WW	67-126	20	0.001	mg/kg
Volatiles	1,2-Dichloroethane	8260B/C, 624, 6200B	GW, WW	67-133	20	0.001	mg/kg
Volatiles	Trichloroethene	8260B/C, 624, 6200B	GW, WW	74-126	20	0.001	mg/kg
Volatiles	1,2-Dichloropropane	8260B/C, 624, 6200B	GW, WW	74-122	20	0.001	mg/kg
Volatiles	Dibromomethane	8260B/C, 624, 6200B	GW, WW	73-125	20	0.001	mg/kg
Volatiles	Bromodichloromethane	8260B/C, 624, 6200B	GW, WW	68-133	20	0.001	mg/kg
Volatiles	2-Chloroethylvinyl Ether	8260B/C, 624, 6200B	GW, WW	0-171	27	0.050	mg/kg
Volatiles	cis-1,3-Dichloropropene	8260B/C, 624, 6200B	GW, WW	73-131	20	0.001	mg/kg
Volatiles	4-Methyl-2-Pentanone (MIBK)	8260B/C, 624, 6200B	-	60-142	20	0.050	mg/kg
Volatiles	Toluene	8260B/C, 624, 6200B	GW, WW	72-122	20	0.005	mg/kg
Volatiles	trans-1,3-Dichloropropene	8260B/C, 624, 6200B	GW, WW	66-137	20	0.001	mg/kg
Volatiles	1,1,2-Trichloroethane	8260B/C, 624, 6200B	GW, WW	79-123	20	0.001	mg/kg
Volatiles	Tetrachloroethene	8260B/C, 624, 6200B	GW, WW	67-135	20	0.001	mg/kg
Volatiles	1,3-Dichloropropane	8260B/C, 624, 6200B	GW, WW	77-119	20	0.001	mg/kg
Volatiles	2-Hexanone	8260B/C, 624, 6200B	GW, WW	56-147	20	0.050	mg/kg
Volatiles	Chlorodibromomethane	8260B/C, 624, 6200B	GW, WW	73-138	20	0.001	mg/kg
Volatiles	1,2-Dibromoethane	8260B/C, 624, 6200B	GW, WW	75-126	20	0.001	mg/kg
Volatiles	Chlorobenzene	8260B/C, 624, 6200B	GW, WW	77-125	20	0.001	mg/kg
Volatiles	1,1,1,2-Tetrachloroethane	8260B/C, 624, 6200B	GW, WW	75-134	20	0.001	mg/kg
Volatiles	Ethylbenzene	8260B/C, 624, 6200B	GW, WW	76-129	20	0.001	mg/kg
Volatiles	Total-Xylene	8260B/C, 624, 6200B	GW, WW	75-128	20	0.003	mg/kg

App. VI, Ver. 10.0 Date: April 15, 2012 Page 20 of 29

		gets for Volatiles Accu able is subject to revisi			and RLs		
Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	Styrene	8260B/C, 624, 6200B		78-130	20	0.001	mg/kg
Volatiles	Bromoform	8260B/C, 624, 6200B		60-139	20	0.001	mg/L
Volatiles	Isopropylbenzene	8260B/C, 624, 6200B		73-132	20	0.001	mg/L
Volatiles	Bromobenzene	8260B/C, 624, 6200B		76-123	20	0.001	mg/L
Volatiles	1,1,2,2-Tetrachloroethane	8260B/C, 624, 6200B	GW, WW	72-128	20	0.001	mg/L
Volatiles	1,2,3-Trichloropropane	8260B/C, 624, 6200B	GW, WW	68-130	20	0.001	mg/L
Volatiles	trans-1,4-Dichloro-2-Butene	8260B/C, 624, 6200B		48-139	20	0.001	mg/L
Volatiles	n-Propylbenzene	8260B/C, 624, 6200B	GW, WW	71-132	20	0.001	mg/L
Volatiles	2-Chlorotoluene	8260B/C, 624, 6200B	GW, WW	74-128	20	0.001	mg/L
Volatiles	4-Chlorotoluene	8260B/C, 624, 6200B	GW, WW	74-130	20	0.001	mg/L
Volatiles	1,3,5-Trimethylbenzene	8260B/C, 624, 6200B	GW, WW	73-134	20	0.001	mg/L
Volatiles	tert-Butylbenzene	8260B/C, 624, 6200B		72-134	20	0.001	mg/L
Volatiles	1,2,4-Trimethylbenzene	8260B/C, 624, 6200B	GW, WW	72-135	20	0.001	mg/L
Volatiles	sec-Butylbenzene	8260B/C, 624, 6200B	GW, WW	70-135	20	0.001	mg/L
Volatiles	1,3-Dichlorobenzene	8260B/C, 624, 6200B	GW, WW	70-121	20	0.001	mg/L
Volatiles	p-Isopropyltoluene	8260B/C, 624, 6200B	GW, WW	68-138	20	0.001	mg/L
Volatiles	1,4-Dichlorobenzene	8260B/C, 624, 6200B	GW, WW	70-121	20	0.001	mg/L
Volatiles	1,2,3-Trimethylbenzene	8260B/C, 624, 6200B	GW, WW	70-127	20	0.001	mg/L
Volatiles	1,2-Dichlorobenzene	8260B/C, 624, 6200B	GW, WW	75-122	20	0.001	mg/L
Volatiles	n-Butylbenzene	8260B/C, 624, 6200B	GW, WW	63-142	20	0.001	mg/L
Volatiles	1,2-Dibromo-3-Chloropropane	8260B/C, 624, 6200B	GW, WW	55-134	20	0.001	mg/L
Volatiles	1,2,4-Trichlorobenzene	8260B/C, 624, 6200B	GW, WW	65-137	20	0.001	mg/L
Volatiles	Hexachlorobutadiene	8260B/C, 624, 6200B	GW, WW	67-135	20	0.001	mg/L
Volatiles	Naphthalene	8260B/C, 624, 6200B		56-145	20	0.005	mg/L
Volatiles	1,2,3-Trichlorobenzene	8260B/C, 624, 6200B	GW, WW	63-138	20	0.001	mg/L
Volatiles	Hexane	8260B/C, 624, 6200B	GW, WW	33-167	20	0.010	mg/L
Volatiles	Acetonitrile	8260B/C, 624, 6200B	GW, WW	61.3-1347	25	0.050	mg/L
Volatiles	Allyl Chloride	8260B/C, 624, 6200B	GW, WW	77.9-1277	25	0.005	mg/L
Volatiles	Chloroprene	8260B/C, 624, 6200B	GW, WW	49.4-142.3	25	0.050	mg/L
Volatiles	Isobutanol	8260B/C, 624, 6200B	GW, WW	59.3-137.6	25	0.100	mg/L
Volatiles	1,4-Dioxane	8260B/C, 624, 6200B	GW, WW	76.2-132.3	25	0.100	mg/L
Volatiles	Methacrylonitrile	8260B/C, 624, 6200B	GW, WW	74.7-126.1	25	0.050	mg/L
Volatiles	Methyl Methacrylate	8260B/C, 624, 6200B	GW, WW	62-142.2	25	0.005	mg/L
Volatiles	Ethyl methacrylate	8260B/C, 624, 6200B	GW, WW	55.4-126.3	25	0.005	mg/L
Volatiles	Propionitrile	8260B/C, 624, 6200B	GW, WW	53.7-143.7	25	0.050	mg/L

App. VI, Ver. 10.0 Date: April 15, 2012 Page 21 of 29

	Table 12.3: QC TargThis ta	ets for Volatiles Accu ble is subject to revisi			and RLs		
Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	Pentachloroethane	8260B/C, 624, 6200B	GW, WW	10-200	25	0.005	mg/L
Volatiles	Cyclohexanone	8260B/C, 624, 6200B	GW, WW	36.5-138.1	25	0.010	mg/L
Volatiles	Bromoethane	8260B/C, 624, 6200B	GW, WW	74.3-136.2	25	0.001	mg/L
Volatiles	2Butanol	8260B/C, 624, 6200B	GW, WW	64.8-140.6	25	0.050	mg/L
Volatiles	Ethanol	8260B/C, 624, 6200B	GW, WW	51.8-153.6	25	0.050	mg/L
Volatiles	Di-isopropyl ether	8260B/C, 624, 6200B	GW, WW	63-139	20	0.001	mg/L
Volatiles	Ethyl tert-butyl ether	8260B/C, 624, 6200B	GW, WW	63.5-131.4	25	0.001	mg/L
Volatiles	Methyl-tert-butyl ether	8260B/C, 624, 6200B	GW, WW	51-142	20	0.001	mg/L
Volatiles	Tert-Butyl alcohol	8260B/C, 624, 6200B	GW, WW	44.2-173.9	25	0.050	mg/L
Volatiles	Tert-Amyl Methyl Ether	8260B/C, 624, 6200B	GW, WW	69.3-125.1	25	0.001	mg/L
Volatiles	Propene	8260B/C, 624, 6200B	solid			0.0025	Mg/l
Volatiles	1,3-Butadiene	8260B/C, 624, 6200B	solid			0.001	Mg/L
Volatiles	4-Ethyltoluene	8260B/C, 624, 6200B	solid			0.001	Mg/L
Volatiles	Dicyclopentadiene	8260B/C, 624, 6200B	solid			0.001	Mg/L
Volatiles	Dichlorodifluoromethane	8260B/C	Solid	26-186	22	0.001	mg/kg
Volatiles	Chloromethane	8260B/C	Solid	42-149	20	0.001	mg/kg
Volatiles	Vinyl Chloride	8260B/C	Solid	50-151	20	0.001	mg/kg
Volatiles	Bromomethane	8260B/C	Solid	41-175	20	0.001	mg/kg
Volatiles	Chloroethane	8260B/C	Solid	44-159	20	0.001	mg/kg
Volatiles	Trichlorofluoromethane	8260B/C	Solid	52-147	20	0.001	mg/kg
Volatiles	Ethyl Ether	8260B/C	Solid	56-147	20	0.001	mg/kg
Volatiles	Acrolein	8260B/C	Solid	3-181	31	0.050	mg/kg
Volatiles	1,1-Dichloroethene	8260B/C	Solid	53-136	20	0.001	mg/kg
Volatiles	1,1,2-Trichloro-1,2,2- trifluoroethane	8260B/C	Solid	49-155	20	0.001	mg/kg
Volatiles	Acetone	8260B/C	Solid	44-140	25	0.050	mg/kg
Volatiles	Iodomethane	8260B/C	Solid	55-156	20	0.050	mg/kg
Volatiles	Carbon Disulfide	8260B/C	Solid	36-161	20	0.001	mg/kg
Volatiles	Methylene Chloride	8260B/C	Solid	57-129	20	0.005	mg/kg
Volatiles	Acrylonitrile	8260B/C	Solid	55-143	20	0.050	mg/kg
Volatiles	trans-1,2-Dichloroethene	8260B/C	Solid	61-133	20	0.001	mg/kg
Volatiles	Methyl Tert Butyl Ether	8260B/C	Solid	44-148	20	0.001	mg/kg
Volatiles	1,1-Dichloroethane	8260B/C	Solid	61-134	20	0.001	mg/kg
Volatiles	Vinyl Acetate	8260B/C	Solid	45-163	20	0.050	mg/kg
Volatiles	Di Isopropyl Ether	8260B/C	Solid	59-143	20	0.001	mg/kg
Volatiles	2,2-Dichloropropane	8260B/C	Solid	50-147	20	0.001	mg/kg
Volatiles	cis-1,2-Dichloroethene	8260B/C	Solid	71-129	20	0.001	mg/kg

App. VI, Ver. 10.0 Date: April 15, 2012 Page 22 of 29

	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	2-Butanone (MEK)	8260B/C	Solid	51-131	25	0.050	mg/kg			
Volatiles	Bromochloromethane	8260B/C	Solid	73-130	20	0.001	mg/kg			
Volatiles	Tetrahydrofuran	8260B/C	Solid	44-144	25	0.001	mg/kg			
Volatiles	Chloroform	8260B/C	Solid	63-123	20	0.005	mg/kg			
Volatiles	1,1,1-Trichloroethane	8260B/C	Solid	62-135	20	0.001	mg/kg			
Volatiles	Carbon Tetrachloride	8260B/C	Solid	60-140	20	0.001	mg/kg			
Volatiles	1,1-Dichloropropene	8260B/C	Solid	63-132	20	0.001	mg/kg			
Volatiles	Benzene	8260B/C	Solid	65-128	20	0.001	mg/kg			
Volatiles	1,2-Dichloroethane	8260B/C	Solid	58-141	20	0.001	mg/kg			
Volatiles	Trichloroethene	8260B/C	Solid	71-126	20	0.001	mg/kg			
Volatiles	1,2-Dichloropropane	8260B/C	Solid	71-128	20	0.001	mg/kg			
Volatiles	Dibromomethane	8260B/C	Solid	70-130	20	0.001	mg/kg			
Volatiles	Bromodichloromethane	8260B/C	Solid	66-126	20	0.001	mg/kg			
Volatiles	2-Chloroethylvinyl Ether	8260B/C	Solid	0-188	39	0.050	mg/kg			
Volatiles	cis-1,3-Dichloropropene	8260B/C	Solid	73-132	20	0.001	mg/kg			
Volatiles	4-Methyl-2-Pentanone (MIBK)	8260B/C	Solid	61-143	23	0.050	mg/kg			
Volatiles	Toluene	8260B/C	Solid	70-120	20	0.005	mg/kg			
Volatiles	trans-1,3-Dichloropropene	8260B/C	Solid	70-135	20	0.001	mg/kg			
Volatiles	1,1,2-Trichloroethane	8260B/C	Solid	77-124	20	0.001	mg/kg			
Volatiles	Tetrachloroethene	8260B/C	Solid	65-135	20	0.001	mg/kg			
Volatiles	1,3-Dichloropropane	8260B/C	Solid	76-120	20	0.001	mg/kg			
Volatiles	2-Hexanone	8260B/C	Solid	62-145	23	0.050	mg/kg			
Volatiles	Chlorodibromomethane	8260B/C	Solid	72-137	20	0.001	mg/kg			
Volatiles	1,2-Dibromoethane	8260B/C	Solid	76-127	20	0.001	mg/kg			
Volatiles	Chlorobenzene	8260B/C	Solid	75-125	20	0.001	mg/kg			
Volatiles	1,1,1,2-Tetrachloroethane	8260B/C	Solid	73-134	20	0.001	mg/kg			
Volatiles	Ethylbenzene	8260B/C	Solid	74-128	20	0.001	mg/kg			
Volatiles	Total-Xylene	8260B/C	Solid	74-127	20	0.003	mg/kg			
Volatiles	Styrene	8260B/C	Solid	76-133	20	0.001	mg/kg			
Volatiles	Bromoform	8260B/C	Solid	64-139	20	0.001	mg/kg			
Volatiles	Isopropylbenzene	8260B/C	Solid	73-130	20	0.001	mg/kg			
Volatiles	Bromobenzene	8260B/C	Solid	75-123	20	0.001	mg/kg			
Volatiles	1,1,2,2-Tetrachloroethane	8260B/C	Solid	74-129	20	0.001	mg/kg			
Volatiles	1,2,3-Trichloropropane	8260B/C	Solid	70-133	20	0.001	mg/kg			
Volatiles	trans-1,4-Dichloro-2-Butene	8260B/C	Solid	52-143	20	0.001	mg/kg			
Volatiles	n-Propylbenzene	8260B/C	Solid	71-132	20	0.001	mg/kg			

App. VI, Ver. 10.0 Date: April 15, 2012 Page 23 of 29

	Table 12.3: QC TargThis ta	ets for Volatiles Acc ble is subject to revis			and RLs		
Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	2-Chlorotoluene	8260B/C	Solid	73-128	20	0.001	mg/kg
Volatiles	4-Chlorotoluene	8260B/C	Solid	72-129	20	0.001	mg/kg
Volatiles	1,3,5-Trimethylbenzene	8260B/C	Solid	71-133	20	0.001	mg/kg
Volatiles	tert-Butylbenzene	8260B/C	Solid	72-132	20	0.001	mg/kg
Volatiles	1,2,4-Trimethylbenzene	8260B/C	Solid	68-135	20	0.001	mg/kg
Volatiles	sec-Butylbenzene	8260B/C	Solid	71-134	20	0.001	mg/kg
Volatiles	1,3-Dichlorobenzene	8260B/C	Solid	71-132	20	0.001	mg/kg
Volatiles	p-Isopropyltoluene	8260B/C	Solid	67-138	20	0.001	mg/kg
Volatiles	1,4-Dichlorobenzene	8260B/C	Solid	72-123	20	0.001	mg/kg
Volatiles	1,2,3-Trimethylbenzene	8260B/C	Solid	73-126	20	0.001	mg/kg
Volatiles	1,2-Dichlorobenzene	8260B/C	Solid	77-123	20	0.001	mg/kg
Volatiles	n-Butylbenzene	8260B/C	Solid	60-145	20	0.001	mg/kg
Volatiles	1,2-Dibromo-3-Chloropropane	8260B/C	Solid	61-134	21	0.001	mg/kg
Volatiles	1,2,4-Trichlorobenzene	8260B/C	Solid	61-148	20	0.001	mg/kg
Volatiles	Hexachlorobutadiene	8260B/C	Solid	65-137	20	0.001	mg/kg
Volatiles	Naphthalene	8260B/C	Solid	61-142	20	0.005	mg/kg
Volatiles	1,2,3-Trichlorobenzene	8260B/C	Solid	62-146	20	0.001	mg/kg
Volatiles	Hexane	8260B/C	Solid	28-169	20	0.010	mg/kg
Volatiles	Acetonitrile	8260B/C	Solid	59.6-170.4	25	0.050	mg/kg
Volatiles	Allyl Chloride	8260B/C	Solid	66.7-106.4	25	0.005	mg/kg
Volatiles	Chloroprene	8260B/C	Solid	61-114.3	25	0.050	mg/kg
Volatiles	Isobutanol	8260B/C	Solid	80.4-130.2	25	0.100	mg/kg
Volatiles	1,4-Dioxane	8260B/C	Solid	78.4-148.5	25	0.100	mg/kg
Volatiles	Methacrylonitrile	8260B/C	Solid	87.1-108.6	25	0.050	mg/kg
Volatiles	Methyl Methacrylate	8260B/C	Solid	90.4-141.9	25	0.005	mg/kg
Volatiles	Ethyl methacrylate	8260B/C	Solid	41.6-159	25	0.005	mg/kg
Volatiles	Propionitrile	8260B/C	Solid	77.8-136	25	0.050	mg/kg
Volatiles	Pentachloroethane	8260B/C	Solid	63.5-179.2	25	0.005	mg/kg
Volatiles	Cyclohexanone	8260B/C	Solid	21.3-170	25	0.010	mg/kg
Volatiles	Bromoethane	8260B/C	Solid	61.7-123.8	25	0.001	mg/kg
Volatiles	2Butanol	8260B/C	Solid	82.5-138.5	25	0.050	mg/kg
Volatiles	Ethanol	8260B/C	Solid	65.6-136.3	25	0.050	mg/kg
Volatiles	Di-isopropyl ether	8260B/C	Solid	59-143	20	0.001	mg/kg
Volatiles	Ethyl tert-butyl ether	8260B/C	Solid	81.4-110.9	25	0.001	mg/kg
Volatiles	Methyl-tert-butyl ether	8260B/C	Solid	44-148	20	0.001	mg/kg

App. VI, Ver. 10.0 Date: April 15, 2012 Page 24 of 29

	Table 12.3: QC TargThis ta	ets for Volatiles Acc ble is subject to revisi			and RLs		
Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	Tert-Butyl alcohol	8260B/C	Solid	59.5-170.4	25	0.050	mg/kg
Volatiles	Tert-Amyl Methyl Ether	8260B/C	Solid	82-115.5	25	0.001	mg/kg
Volatiles	GRO	8015B/C/D	GW, WW	70-124	20	0.100	mg/L
Volatiles	Benzene	8021B, 602, 6200C	GW, WW	79 - 131	20	0.0005	mg/L
Volatiles	Toluene	8021B, 602, 6200C	GW, WW	68 - 114	20	0.005	mg/L
Volatiles	Ethylbenzene	8021B, 602, 6200C	GW, WW	68 - 125	20	0.0005	mg/L
Volatiles	m&p-Xylene	8021B, 602, 6200C	GW, WW	67 - 113	20	0.001	mg/L
Volatiles	o-Xylene	8021B, 602, 6200C	GW, WW	72 - 114	20	0.0005	mg/L
Volatiles	MTBE	8021B, 602, 6200C	GW, WW	60 - 133	20	0.001	mg/L
Volatiles	Benzene	502.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Toluene	502.2	DW	70 - 130	25	0.005	mg/L
Volatiles	Ethylbenzene	502.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	m&p-Xylene	502.2	DW	70 - 130	25	0.001	mg/L
Volatiles	o-Xylene	502.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	MTBE	502.2	DW	70 - 130	25	0.001	mg/L
Volatiles	GRO	8015B	Solid	67 - 135	20	0.500	mg/kg
Volatiles	Benzene	8021B	Solid	78 - 141	20	0.0025	mg/kg
Volatiles	Toluene	8021B	Solid	65 - 117	20	0.025	mg/kg
Volatiles	Ethylbenzene	8021B	Solid	69 - 133	20	0.0025	mg/kg
Volatiles	m&p-Xylene	8021B	Solid	61 - 121	20	0.005	mg/kg
Volatiles	o-Xylene	8021B	Solid	71 - 121	20	0.0025	mg/kg
Volatiles	MTBE	8021B	Solid	54 - 129	20	0.005	mg/kg
Volatiles	1,1,1,2-Tetrachloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1,1-Trichloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1,2,2-Tetrachloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1,2-Trichloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1-Dichloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1-Dichloroethene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1-Dichloropropanone	524.2	DW	70 - 130	25		mg/L
Volatiles	1,1-Dichloropropene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2,3-Trichlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2,3-Trichloropropane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2,4-Trichlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2,4-Trimethylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2-Dibromo-3-chloropropane	524.2	DW	70 - 130	25	0.0010	mg/L

App. VI, Ver. 10.0 Date: April 15, 2012 Page 25 of 29

	Table 12.3: QC TargThis ta	gets for Volatiles Ac uble is subject to revi			and RLs		
Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	1,2-Dibromoethane	524.2	DW	70 - 130	25	0.0010	mg/L
Volatiles	1,2-Dichlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2-Dichloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2-Dichloropropane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,3,5-Trimethylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,3-Dichlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,3-Dichloropropane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,4-Dichlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1-Chlorobutane	524.2	DW	70 - 130	25		mg/L
Volatiles	2,2-Dichloropropane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	2-Butanone	524.2	DW	70 - 130	25		mg/L
Volatiles	2-Chlorotoluene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	2-Hexanone	524.2	DW	70 - 130	25		mg/L
Volatiles	2-Nitropropane	524.2	DW	70 - 130	25		mg/L
Volatiles	4-Chlorotoluene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	4-Isopropyltoluene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	4-Methyl-2-pentanone	524.2	DW	70 - 130	25		mg/L
Volatiles	Acetone	524.2	DW	70 - 130	25	0.01	mg/L
Volatiles	Acrylonitrile	524.2	DW	70 - 130	25		mg/L
Volatiles	Allyl Chloride	524.2	DW	70 - 130	25		mg/L
Volatiles	Benzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Bromobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Bromochloromethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Bromodichloromethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Bromoform	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Bromomethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Carbon Disulfide	524.2	DW	70 - 130	25		mg/L
Volatiles	Carbon Tetrachloride	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Chloroacetonitrile	524.2	DW	70 - 130	25		mg/L
Volatiles	THMs	524.2	DW	70 - 130	25		mg/L
Volatiles	Chlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Chloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Chloroform	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Chloromethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Cis-1,2-dichloroethene	524.2	DW	70 - 130	25	0.0005	mg/L

App. VI, Ver. 10.0 Date: April 15, 2012 Page 26 of 29

	Table 12.3: QC TargThis ta	ets for Volatiles Ac ble is subject to revi	•		and RLs		
Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	Cis-1,3-dichloropropene	524.2	DW	70 - 130	25	0.0010	mg/L
Volatiles	Dibromochloromethane	524.2	DW	70 - 130	25		mg/L
Volatiles	Dibromomethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Dichlorodifluoromethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Diethyl ether	524.2	DW	70 - 130	25		mg/L
Volatiles	Ethyl Methacrylate	524.2	DW	70 - 130	25		mg/L
Volatiles	Ethylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Hexachlorobutadiene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Hexachloroethane	524.2	DW	70 - 130	25		mg/L
Volatiles	Isopropylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Meta-xylene	524.2	DW	70 - 130	25		mg/L
Volatiles	Methacrylonitrile	524.2	DW	70 - 130	25		mg/L
Volatiles	Methyl Iodide	524.2	DW	70 - 130	25		mg/L
Volatiles	Methylacrylate	524.2	DW	70 - 130	25		mg/L
Volatiles	Methylene Chloride	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Methylmethacrylate	524.2	DW	70 - 130	25		mg/L
Volatiles	Methyl-t-butyl ether	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Naphthalene	524.2	DW	70 - 130	25	0.0050	mg/L
Volatiles	N-butylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Nitrobenzene	524.2	DW	70 - 130	25		mg/L
Volatiles	N-propylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Ortho-xylene	524.2	DW	70 - 130	25		mg/L
Volatiles	Para-xylene	524.2	DW	70 - 130	25		mg/L
Volatiles	Pentachloroethane	524.2	DW	70 - 130	25		mg/L
Volatiles	Propionitrile	524.2	DW	70 - 130	25		mg/L
Volatiles	Sec-butylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Styrene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Tert-butylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Tetrachloroethene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Tetrahydrofuran	524.2	DW	70 - 130	25		mg/L
Volatiles	Toluene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Trans-1,2-dichloroethene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Trans-1,3-dichloropropene	524.2	DW	70 - 130	25	0.0010	mg/L
Volatiles	Trans-1,4-dichloro-2-butene	524.2	DW	70 - 130	25		mg/L
Volatiles	Trichloroethene	524.2	DW	70 - 130	25	0.0005	mg/L

	Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RLs         This table is subject to revision without notice								
Class	ClassAnalyteMethodMatrixAccuracy (%)**Prec.** (RPD)RLUnit								
Volatiles	Trichlorofluoromethane	524.2	DW	70 - 130	25	0.0005	mg/L		
Volatiles	Vinyl Chloride	524.2	DW	70 - 130	25	0.0005	mg/L		
Volatiles	Xylenes – total	524.2	DW	70 - 130	25		mg/L		

\*\* Specific organizations may require limits that supersede 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 CAR are kept on file by the QA 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 take precedence.

13.2.2 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

<u>Rejection Criteria</u> - Blank reading is more than twice the background absorbance or more than 1/2 RL.

<u>Corrective Action</u> - Blanks are reanalyzed 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 Manager or QA Department 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)

<u>Rejection Criteria</u> - 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 excedence allowance is surpassed (see section 12.2).

<u>Corrective Action</u> - 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 rerun. The group leader, Department Manager, or QA Department is consulted for further action.

13.2.4 Out Of Control Matrix Spike Samples

<u>Rejection Criteria</u> - If sample is outside of lab-generated control limits from accuracy charts on matrix spike samples from a similar matrix (i.e., water, solid, etc). Limits are calculated as the mean of at least 20 data points +/- 3 times the standard deviation of those points.

<u>Corrective Action</u> - Spiking technique is assessed to ascertain if the sample has been spiked correctly. The spiked sample should be 1-5 times the client sample concentration; otherwise, the percent recovery (%R) or relative percent difference (%RPD) of the MS/MSD is flagged as not meaningful or usable. The sample is re-spiked and re-analyzed, along with several other similar samples in subset. If an out of control situation persists, sample matrix interference is indicated. Samples to be analyzed by standard additions are prepared (where appropriate), and the group leader, Department Manager, or QA Department is notified.

### 13.2.5 Out Of Control Duplicate Samples

<u>Rejection Criteria</u> - Lab-generated maximum RPD limit (as listed under precision in Section 12)

<u>Corrective Action</u> - 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 before and just after the duplicated sample are re-checked. If problem still exists, Department Manager, or QA Department is notified to review the analytical techniques.

13.2.6 Out Of Control Matrix Spike Duplicates

Rejection Criteria - These QC samples can be out of control for accuracy, precision, or both.

<u>Corrective Action</u> - The appropriate corrective actions listed for either matrix spikes, duplicate samples, or both are followed.

**NOTE**: Some samples cannot be duplicated. This is the case for wipe samples, filters, and some water samples. When possible, sampling personnel should collect duplicate samples.

13.2.7 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

<u>Corrective Action</u> - 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 rerun. The group leader, Department Manager, or QA Department is consulted for further action.

### 14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* 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, 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 8.0.

App. VII, Ver. 10.0 Date: April 15, 2012 Page 1 of 44

**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

ESC LAB SCIENCES 12065 LEBANON ROAD MT. JULIET, TENNESSEE 37122 (615) 758-5858

**NOTE:** The QAM has been approved by the following people. A signed cover page is available upon request

Technical and Regulatory Affairs 615-773-9657 gan.

Er

inson, B.S., Laboratory Director 615-773-9654

Dixie Marlin, B.S, QC Manager, 615-773-9681

Kenny Buckley, B.S., Organics/Wet Chemistry Department Manager, 615-773-9686

## 2.0 APPENDIX TABLE OF CONTENTS

Section	Section Title			Rev. Date	<i>Rev.</i> #
1.0	Approval and Signature Page	Page	1	4/15/11	2
2.0	Table of Contents	Page	2	4/15/11	2
3.0	Scope and Application	Page	3	4/15/11	2
4.0	Laboratory Organization and Responsibility	Page	3	4/15/11	2
5.0	Personnel and Training	Page	3	4/15/11	2
6.0	Facilities and Laboratory Safety	Page	3	4/15/11	2
7.0	Sampling Procedures	Page	4	4/15/11	2
8.0	Equipment	Page	5	4/15/11	2
9.0	Laboratory Practices	Page	20	4/15/11	2
10.0	Analytical Procedures	Page	21	4/15/11	2
11.0	Quality Control Checks	Page	22	4/15/11	2
12.0	Data Reduction, Validation and Reporting	Page	23	4/15/11	2
13.0	Corrective Actions	Page	40	4/15/11	2
14.0	Record Keeping	Page	42	4/15/11	2
15.0	Quality Audits	Page	42	4/15/11	2
	TABLES				
8.1	Equipment	Page	5	4/15/11	2
8.2	Equipment Preventative Maintenance, Equipment Calibration	Page	7	4/15/11	2
8.3A	Standards and Reagents	Page	8	4/15/11	2
8.3B	Working Standards	Page	9	4/15/11	2
8.5	Instrument Calibration	Page	18	4/15/11	2
10.1	Semi-Volatile Department SOPs	Page	20	4/15/11	2
12.1	Data Reduction Formulas	Page	23	4/15/11	2
12.3	QC Targets and RLs	Page	24	4/15/11	2

### **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 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 8.0*.

### 5.0 PERSONNEL AND TRAINING

### 5.1 **PERSONNEL**

Kenneth W. Buckley, with a B.S. degree in General Science, is the Department Manager of Organics and Wet Chemistry laboratories. Mr. Buckley reviews and approves all data reduction associated with analyses in these areas and is responsible for the overall production of these laboratories; including the management of the staff and scheduling. Mr. Buckley has over 12 years of environmental laboratory experience. In his absence, Chris Johnson assumes responsibility for departmental decisions. Mr. Johnson has a B.S. degree in Biology and over 11 years of environmental laboratory experience.

### 5.2 TRAINING

5.2.1 All new analysts to the laboratory are trained by the primary analyst or Manager according to ESC protocol. 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 #1 has nearly 4500 square feet with approximately 220 square feet of bench area and an additional storage area of 210 square feet. The air handling system in this area is a 100-ton Trane split unit with natural gas for heating. The 4000 square feet of area in the extraction laboratory, contained in Building 5, 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 and Safety 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. Matrices for Industrial Hygiene analyses include: sorbent tubes, filters, or Organic Vapor Monitor (OVM) Badges.
- 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

LABOR	ATORY EQUIPM This	ENT LIST: MA table is subject to revi		emi-V	olatiles Analysis	
Item	Manufacturer	Model	Instrument Name	#	Serial #	Location
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 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

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis This table is subject to revision without notice						
Item	Manufacturer	Model	Instrument Name	#	Serial #	Location
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	Svcompat	29	F) U20277 B) U20299	SVOC
Gas Chromatograph Detectors 30	Detectors	ECD/ECD	svcompat	30	F) U20425 B) U20424	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 3	Agilent	6890 GC/5973MSD	svcompz	3	GC US00039611 MS US03940681	SVOC
Gas Chromatograph/Mass Spectrometer 4	Agilent	6890 GC/5973MSD	svcomph	4	GC CN10403067 MS US35120308	SVOC
Gas Chromatograph/Mass Spectrometer 5	Agilent	6890 GC/5973MSD	svcompi	5	GC US00024766 MS US91911297	SVOC
Gas Chromatograph/Mass Spectrometer 6	Agilent	6890 GC/5973MSD	svcompl	6	GC US00039647 MS US05040021	SVOC
Gas Chromatograph/Mass Spectrometer 7	Agilent	6890 GC/5973MSD	svcompm	7	GC MS US03940745	SVOC
Gas Chromatograph/Mass Spectrometer 9	Agilent	6890 GC/5973MSD	svcompx	9	GC CN10344042 MS US33220158	SVOC

App. VII, Ver. 10.0 Date: April 15, 2012 Page 7 of 44

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis This table is subject to revision without notice						
Item	Manufacturer	Model	Instrument Name	#	Serial #	Location
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
High Performance Liquid Chromatography	Agilent	1100 Series DAD/FLD	hplc1	1	DAD de01608402 FLD de23094489	SVOC
High Performance Liquid Chromatography	Agilent	1100 Series DAD/FLD	hplc2	2	DAD de30518420 FLD de11103457	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 FLD de92001880	SVOC
Analytical Balance	Mettler Toledo	PB1502-S		1	1126193668	Ext. Lab
Analytical Balance	Mettler Toledo	MS1602S/03		2	2213	Ext. Lab
Analytical Balance	Ohaus	ARA520		3	1202120618	Ext. Lab
Analytical Balance	Ohaus	ARA520		4	1202120814	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	1	2302	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	2	2304	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	3	2303	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	4	040000940	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	5	406583020005	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	6	1469	Ext. Lab

App. VII, Ver. 10.0 Date: April 15, 2012 Page 8 of 44

#### LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis This table is subject to revision without notice Instrument # Serial # Item Manufacturer Model Location Name 7 Automatic Concentrators Buchi Syncore Buchi 1461 Ext. Lab 8 Automatic Concentrators Buchi Buchi 417004020002 Ext. Lab Syncore Buchi 9 416870050003 Automatic Concentrators Buchi Syncore Ext. Lab Automatic Concentrators Buchi Syncore Buchi 10 1466 Ext. Lab 11 Automatic Concentrators Buchi Syncore Buchi 1463 Ext. Lab 12 1462 Automatic Concentrators Buchi Syncore Buchi Ext. Lab 13 Buchi Syncore Buchi 1468 Ext. Lab Automatic Concentrators MARS X Ext. Lab Capping station Horizon snxc2225 MARS X Capping station Horizon snxc2215 Ext. Lab Centrifuge Labnet Z-400 1 Ext. Lab 2158 ST-40 2 2224 Centrifuge Sorvall Ext. Lab 3 Centrifuge Sorvall ST-40 2225 Ext. Lab Centrifuge 4 Sorvall ST-40 2226 Ext. Lab Centrifuge 5 Sorvall ST-40 2227 Ext. Lab **Concentration Chiller** Lauda WKL 3200 2031 Ext. Lab Concentration Chiller Lauda WKL 3200 2039 Ext. Lab Furnace Thermo Scientific 1882 Ext. Lab Fisher Oven 6556 166 Ext. Lab LVI Shaker Eberbach 2159 Ext. Lab HAA Shaker Eberbach 6010-04 1834 Ext. Lab HAA water Bath Thermo Scientific 280 series 2033602-102 Ext. Lab High Intensity Ultrasonic Misonix 1 2193 Ext. Lab Processor High Intensity Ultrasonic Misonix 2 1382 Ext. Lab Processor High Intensity Ultrasonic 3 Misonix 1888 Ext. Lab Processor High Intensity Ultrasonic 4 Misonix 1381 Ext. Lab Processor High Intensity Ultrasonic 5 1640 Ext. Lab Misonix Processor Microwave CEM MARS X 1 1507 Ext. Lab 2 MARS X Ext. Lab Microwave CEM 1518 Microwave CEM MARS X 3 2269 Ext. Lab 1 SpeedVap III 1534 Ext. Lab OG concentrator Horizon 2 OG concentrator Horizon SpeedVap III SN04-2020 Ext. Lab SpeedVap III 3 Horizon Ext. Lab OG concentrator 2186 OG SPE extractor Horizon **SPE-DEX 3000** 1 1481 Ext. Lab 2 OG SPE extractor Horizon **SPE-DEX 3000** 1482 Ext. Lab OG SPE extractor Horizon **SPE-DEX 3000** 3 1483 Ext. Lab 4 OG SPE extractor Horizon SPE-DEX 3000 1484 Ext. Lab OG SPE Controllers Horizon 1000/3000XL 1 2125 Ext. Lab

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis This table is subject to revision without notice							
Item	Manufacturer	Model	Instrument Name	#	Serial #	Location	
OG SPE Controllers	Horizon	1000/3000XL		2	2126	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				1510	Ext. Lab	
Separatory funnel rotators	ATR				1511	Ext. Lab	
Separatory funnel rotators	ATR				1512	Ext. Lab	
Separatory funnel rotators	ATR				1513	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	
SPE Water Extractor	UCT				1944	Ext. Lab	
SPE Water Extractor	UCT				1945	Ext. Lab	
Water Bath Sonicator	Branson	8510			RPA040384175E	Ext. Lab	
Vacuum Pump	Gast			1	0908605640	Ext. Lab	
Vacuum Pump	Gast			2	0611012209	Ext. Lab	
Vacuum Pump	Gast			3	0311000841	Ext. Lab	

# 8.2 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 (semiannual contract maintenance and calibration check)	Semiannually
Refrigerators & Incubators	•Maintenance service	As needed - determined by daily temperature performance checks
Gas Chromatograph Detectors: ECD	<ul><li>Bake off or Replace</li><li>Perform wipe leakage test</li></ul>	As needed - when deterioration is noticeable Annually
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/Mass Spectrometer & Gas Chromatographs	•Replace septa and liner	As needed to maintain injection port inert
Gas Chromatographs/Mass	•Replace column	When separation begins to degrade

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
Spectrometer & Gas Chromatographs		
High Intensity Ultrasonic Processor - Misonix	•Check tuning criteria	Daily with use
Infrared Spectrophotometer - Foxboro Miran 1A	locintics allonment or replacement	As needed when response begins to deteriorate

### 8.3 STANDARDS AND REAGENTS

Method	Vendor*	This table is subject to <b>Description</b>	Calibration	Storage Req.	Expiration
8310	Ultra	Aromatic Hydrocarbon	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
	NSI	8310/610 Spike	Second Source	$4^{\circ} \pm 2^{\circ}C$	6 months
DRO	NSI	DRO #2 Cal Mix	Primary	-10°C to -20°C	6 months
DRO	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	$4^{\circ} \pm 2^{\circ}C$	6 months
rCD	NSI	1260 Spike	Second Source	$4^{\circ} \pm 2^{\circ}C$	6 months
Chlordane	Restek	Chlordane Mix	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
Toxaphene	Restek	Toxaphene	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
Pesticides	Ultra	Pest Mix	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
Pesticides	NSI	Pest Spike Mix	Second Source	$4^{\circ} \pm 2^{\circ}C$	6 months
Herbicides	NSI	Custom Herbicide Mis	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
	NSI	Herb Spike Mix	Second Source	$4^{\circ} \pm 2^{\circ}C$	6 months
	Ultra/NSI	OP Cal Mix A, B	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
	NSI	OP Spike Mix A, B	Second Source	$4^{\circ} \pm 2^{\circ}C$	6 months
507 NP Pest	Ultra/NSI	507 Cal Mix	Primary	$4^{\circ} \pm 2^{\circ}C$	2 months
507 NP Pest	NSI	NP Pest Spike	Second Source	$4^{\circ} \pm 2^{\circ}C$	2 months
THAA	Ultra/Accustd	HAA Cal Mix	Primary	-10°C to -20°C	6 months
ТПАА	Accustd/NSI	HAA Spike	Second Source	-10°C to -20°C	6 months
8270	Ultra	Custom Std Mega Mix	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
	Restek	Spike Mix	Second Source	$4^{\circ} \pm 2^{\circ}C$	6 months
8330 -	Restek	Mix1, Mix2, PETN	Primary	$4^{\circ} \pm 2^{\circ}C$	6 months
	Ultra, Chemservice	Mix1, Mix2, PETN	Second Source	$4^{\circ} \pm 2^{\circ}C$	6 months
8011, 504.1	Accustd	504.1 Cal Mix	Primary	$4^{\circ} \pm 2^{\circ}C$	1 month
	NSI	Spike Mix	Second Source	$4^{\circ} \pm 2^{\circ}C$	1 month
ndustrial Hygiene	Chemservice	Neat	Primary & Secondary	$4^{\circ} \pm 2^{\circ}C$	6 months

\*Equivalent Providers may be utilized.

App. VII, Ver. 10.0 Date: April 15, 2012 Page 11 of 44

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)	$4^{\circ} \pm 2^{\circ}C$	6 months		
Semi-Volatiles: RV/LVI	625, SM6410B 20 <sup>th</sup> , 8270C/D	10,20,50,100,200,500,1000,2000 ug/L	$4^{\circ} \pm 2^{\circ}C$	6 months		
PCBs 1016/1260	608, SM6431B 20 <sup>th</sup> , 8082	0.05, 0.1, 0.25, 0.5, 0.75, 1.0 μg/mL	$4^{\circ} \pm 2^{\circ}C$	6 months		
PCBs: RV	608, SM6431B 20 <sup>th</sup> , 8082	2.0,4.0,5.0,10,20,50 μg/L	$4^{\circ} \pm 2^{\circ}C$	6 months		
Pesticides	608, SM 6630C, 8081A, 508	0.05, 0.10, 0.20, 0.40, 0.60, 0.80 µg/mL	$4^{\circ} \pm 2^{\circ}C$	6 months		
Pesticides: RV	608, SM 6630C, 8081A,	0.5,1.0,2.0,5.0,10,15,20 μg/L	$4^{\circ} \pm 2^{\circ}C$	6 months		
Chlordane and/or Toxaphene	608, SM 6630C, 8081A, 508	0.1, 0.5, 1.0, 2.5, 5.0, 10.0 μg/mL	$4^{\circ} \pm 2^{\circ}C$	6 months		
Chlordane and/or Toxaphene	608, SM 6630C, 8081A,	10,20,50,100,150,200 µg/L	$4^{\circ} \pm 2^{\circ}C$	6 months		
PCB Arochlors 1221, 1232, 1242, 1248, 1254	8082	5.0 μg/mL	$4^{\circ} \pm 2^{\circ}C$	6 months		
Herbicides	515.2, 8151A, SM6640C 20th	0.02, 0.05, 0.1, 0.2, 0.5, 1.0 mg/L	$4^{\circ} \pm 2^{\circ}C$	6 months		
OP and NP Pesticides	507 by dual-NPD, 1657A, 8141A by dual-FPD	1.0, 2.0, 5.0, 10.0, 15.0, 20.0 ug/L	$4^{\circ} \pm 2^{\circ}C$	6 months		
PAHs	8310, 610, SM6440B 20 <sup>th</sup> 8270C/D SIM	0.04, 0.20,1.0,5.0,8.0,20.0,30.0,40.0 ug/L 0.025, 0.05, 0.10, 0.50, 2.0, 4.0, 10.0, 20.0 ug/L	$4^{\circ} \pm 2^{\circ}C$	6 months		
PAHs: RV/LVI	8270C/D SIM	4.0,20,40,100,160,400,600,800 ug/L 1.0,5.0,10,20,40,80,200	$4^{\circ} \pm 2^{\circ}C$	6 months		
Nitroaromatics & Nitramines	8330	ug/L .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*		

TABLE 8.3B: Working Standard Concentrations         This table is subject to revision without notice						
Organic Compounds	Method #	Standard Concentrations	Storage Requirements	Expiration		
DROMO: LVI PAHMO: LVI	MO DRO/PAH by 8270	5.0,10,20,40,80,120,160,200 mg/L 4.0,20,40,100,160,400,600,800 ug/L	$4^{\circ} \pm 2^{\circ}C$	6 months		
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 ug/L	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,	NA*	NA*		
ТХ ТРН	TX1005	<b>2500</b> mar/l 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*		
IH - Aromatics	NIOSH/OSHA.	10-10000 ug/sample	NA*	NA*		
DROMO PAHMO	MO DRO/PAH by 8270	300, 500, 1000, 2000, 4000, 6000, 8000, 10000 mg/L 1.0, 5.0, 10, 20, 40, 60, 80, 100 ug/L	$4^{\circ} \pm 2^{\circ}C$	6 months		

\* indicates solutions are prepared fresh daily as needed.

### 8.4 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, by the analysis of a continuing calibration verification (CCV) standard. The CCV must recover 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 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 if 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 for each compound and response factors are calculated. If the response factors of the initial calibration and exponse factors are 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 20% 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 by more than  $\pm 20\%$ 

App. VII, Ver. 10.0 Date: April 15, 2012 Page 14 of 44

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 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 minimum quarterly to verify calibration standards.

#### 515.1, 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 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. 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.

### <u>625, 8270C or D, SM6410B - Base/Neutrals/Acids by GC/MS: Semivolatile Organics –</u> <u>SOP Number 330345</u>

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 the 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.

App. VII, Ver. 10.0 Date: April 15, 2012 Page 17 of 44

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 +/- 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 midpoint 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.

#### <u>8330A/B/C – Nitroaromatics/Nitrosamines - SOP Number 330323</u>

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 <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. USACE projects must meet a correlation coefficient of 0.995 or better.

App. VII, Ver. 10.0 Date: April 15, 2012 Page 19 of 44

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. 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.

### NIOSH 1501 modified – Aromatic Hydrocarbons in Air - SOP Number 330303

The gas chromatograph is calibrated using the external standard procedure. A standard curve is prepared using a minimum of six concentration levels for each analyte of interest. The calibration range must represent the typical sample concentration. The linear range of the instrument must 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 <15% 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. When sample responses exceed the range of the standard curve, the sample is diluted and re-analyzed. A mid-level independently prepared calibration verification standard (ICV) is analyzed following each initial calibration and should meet criteria of +15% of the expected concentration for each analyte. Following each 10 samples and at the end of the analytical sequence, a continuing calibration verification standard is analyzed to demonstrate the continued stability of the analytical sequence. This standard should meet criteria of +15% of the expected concentration for each analyte.

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.

Desorption Efficiency for each lot of sorbent media is determined for each analyte of interest. Desorption Efficiency for analytes on badges has been determined and is available from the manufacturer. The reporting limit from media must be verified with each batch of samples analyzed. Additionally, a Laboratory Control Sample pair (LCS & LCSD) is prepared on media for each batch of samples analyzed.

### 8.5 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. If a check standard does not perform within established criteria then the instrument will undergo 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 will be re-analyzed.

	TA	BLE 8.5: INS	TRUME	NT CALIBRATION	
Instrument (Analysis)	Calibration Type	Minimum Number of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
Gas Chromatography	Initial	3 (600 series methods) - 5 (other) cal.stds	Avg. RF or Linear	8081A, 8151A, 6640C, 8141A, 657A: Must be ≤20% RSD 608 - ≤10% RSD	As needed
(Pest/PCB,	Second Source	1 Second Source		+/- 20% of true value	With each calibration
Herbicides, Organophos/ Organonitrogen Pesticides)	Daily / Continuing	OPPEST/HE RB1/10 P/PCB 1/20		Must be within 15% of the initial calibration curve, 20% for 6640C.	Beginning, every 10 and ending for external cal. Every 20 samples for internal cal
HPLC	Initial	3 (600 series methods) 5 (other) cal.stds	Avg. RF or Linear	8310, 8330: Must be ≤20% RSD 610 - ≤10% RSD	As needed
(PAH and Explosive)	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	Initial	At least 5 cal. stds	Avg. RF or Linear	<ul> <li>8270C - Must be ≤15% RSD, CCCs must be ≤ 30% RSD, Linear regression: 0.990 per method or 0.995 for USACE</li> <li>8270D - Must be ≤20% RSD for target analytes, Linear regression: 0.990 per method or 0.995 for USACE</li> </ul>	As needed
Semi-volatiles 8270C/D	Second Source Daily /	1 Second Source 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	With each calibration
	Continuing			Must pass established method criteria. See SOP.	Every 12 hours per method
	Initial	3 cal.stds	Avg. RF or	625 - ≤35% RSD all compounds	As needed
GC/MS Semi-volatiles	Second Source	1 Second Source	Linear	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
625	Daily / Continuing	Tune & CCV every 24 hours		Must pass established method tuning criteria; 625: CCV must be ≤20% difference for all compounds,	Every 24 hours
	Initial	5 cal.stds	Avg. RF or	≤30% RSD all compounds	As needed
HAA 552.2	Second Source(QCS)	1 Second Source	Linear	$\pm 30\%$ of true value	Quarterly
	Daily / Continuing	1/10		CCV must be $\leq 30\%$ 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				
	Initial	5 cal.stds	Avg. RF or	≤20% RSD all compounds	As needed				
Pesticides 507	Second Source(QCS)	1 Second Source	Linear	$\pm 20\%$ of true value	Quarterly				
	Daily / Continuing	1/10		CCV must be ≤20% difference for all compounds,	Beginning, every 10 and ending				
	Initial	5 cal.stds	Avg. 8015B/C/D - ≤20% RSD all compounds RF or		As needed				
DRO –8015, State Programs*	Second Source	1 Second Source	Linear	$\pm 20\%$ of true value	With each calibration				
* Or per state requirement	Daily / Continuing	1/10		CCV must be $\leq 15\%$ difference for all compounds,	Beginning, every 10 and ending				
	Initial	6 cal.stds	Avg. RF or	≤15% RSD all compounds	Daily				
NIOSH 1501 mod.	ICV	1 Independent Prep.	Linear	$\pm 15\%$ of true value	With each calibration Beginning,				
	Continuing	1/10		$\pm 15\%$ of true value	every 10 and ending				

# 9.0 LABORATORY PRACTICES

# 9.1 REAGENT GRADE WATER

ASTM Type I grade water.

# 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: methanol, 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:

	IABLE 10.1: SEIVII- VOLATILE DEPARTIVIENT SOPS           This table is subject to revision without notice
SOP #	Title
	Preparatory SOPs
330702	Separatory Funnel Liquid-Liquid Extraction 3510C
330702A	Separatory Funnel Liquid-Liquid Extraction 3510C for Minnesota Samples
330705	Ultrasonic Extraction 3550B
330707	Microwave Extraction 3546
330708	Buchi Syncore Concentration System
330743	Solid Phase Extraction
330754	Waste Dilution for SVOCs 3580A
330755	PCB in Oil Waste Dilution
	Extract Cleanup SOPs
330739	Silica Gel Cleanup 3630C
330740	Acid Cleanup 3665A
330741	Sulfur Cleanup 3660C
330742	Florisil Cleanup 3620B
	Semi-Volatiles Analysis SOPs
330303	Organics on Charcoal Tubes (includes badges)
330318	Organophosphorus Pesticides 8141A/ 1657A/ 614/ 622
330319	THAAs 552.2
330320	Chlorinated Herbicides by Gas Chromatography 8151A/ SM6640B
330322	PAH's by HPLC 8310/ 610/ SM6440B
330323	Explosives by HPLC 8330
330324	Carbamates by HPLC 531.1/ SM6610B
330343	PCBs 8082 & A
330344	Pesticides and PCBS by Gas Chromatography 8081A&B/ 608/ SM6630C
330345	Semi-volatile Organics by GC/MS using Capillary Column 8270C & D/ 625/ SM6410B
330346	EDB in Drinking Water by GC ECD 8011/ 504.1
330348	NP Pesticides in Drinking Water by GC NPD 507
330349	Chlorinated Pesticides in Drinking Water by GC ECD 508
330352	Method for Determination of Extractable Petroleum Hydrocarbons by GC/FID – DRO-KY, TN EPH, TPH-AZ, DRO CA and OH by Modified Method 8015. Includes Wyoming LAUST Requirements
330353	NC - Extractable Petroleum Hydrocarbons
330355	Florida PRO, WI DRO and CT ETPH
330356	TX TPH 1005/1006
330358	OA2 & NWTPH-Dx
330359	AK 102/103
330360	DRO Wisconsin/Minnesota
J 2510DX	

### **TABLE 10.1: SEMI-VOLATILE DEPARTMENT SOPS**

Add 3510RV SOP

# **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. For industrial hygiene accreditation, PTs are administered by AIHA. 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 are analyzed one per batch of samples.
- 11.5 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 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.

11.6 For Industrial Hygiene analyses (sorbent tubes and badges), a media blank will be prepared with each batch of samples. In addition, a media reporting limit verification will be prepared with each batch of samples. For accuracy and precision determinations, a LCS/LCSD pair will be spiked on media then desorbed and analyzed concurrently with every batch of field samples.

### 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 Quality Control Department performs the secondary review of the data package using the ESC SOP #030227, *Data Review*. The QC Reviewer verifies that the analysis has 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.)

PARAMETER	FORMULA
GC and HPLC	response of sample analyte {area} x final extract volume { $mL$ } x dilutionresponse factor { $area/(mg/mL)$ } x initial extract volume-mass { $mL \text{ or } g$ }Calculations performed by HP Enviroquant Software
GC/MS	$\frac{\text{response of analyte } \{area\} \text{ x extract volume } \{mL\} \text{ x dilution x int. std amt. } \{area\} \\ \text{response factor } \{area/(mg/mL)\} \text{ x initial volume-mass } \{mL \text{ or } g\} \text{ x int. std cal. } \{area\} \\ \text{Calculations performed by HP Enviroquant Software} \end{cases}$
GC - IH	Sample conc. (front tube + back tube) $(ug)$ - blank conc. (front tube + back tube) $(ug)$ Volume of air sampled $(L)$

# **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.

<u>Marginal Excedence</u> – 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.

App. VII, Ver. 10.0 Date: April 15, 2012 Page 26 of 44

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					

<u>Organic Control Limits -</u> The organic QC targets are statutory in nature; warning and control limits for organic analyses are initially set 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 Semi-Volatiles Accuracy (LCS), Precision and RLs         This table is subject to revision without notice								
Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit	
Pesticides	Azinphos-Methyl	8141A, 1657A	GW	49-126	27	0.001	mg/L	
Pesticides	Bolstar (Sulprofos)	8141A, 1657A	GW	49-122	25	0.001	mg/L	
Pesticides	Chlorpyrifos	8141A, 1657A	GW	46-124	25	0.001	mg/L	
Pesticides	Coumaphos	8141A, 1657A	GW	49-126	26	0.001	mg/L	
Pesticides	Demeton,-O And -S	8141A, 1657A	GW	10-105	23	0.002	mg/L	
Pesticides	Diazinon	8141A, 1657A	GW	43-143	23	0.001	mg/L	
Pesticides	Dichlorvos	8141A, 1657A	GW	41-113	21	0.002	mg/L	
Pesticides	Dimethoate	8141A, 1657A	GW	18-104	34	0.001	mg/L	
Pesticides	Disulfoton	8141A, 1657A	GW	45-123	23	0.001	mg/L	
Pesticides	Epn	8141A, 1657A	GW	51-130	27	0.001	mg/L	
Pesticides	Ethoprop	8141A, 1657A	GW	42-125	21	0.001	mg/L	
Pesticides	Ethyl Parathion	8141A, 1657A	GW	55-122	24	0.001	mg/L	
Pesticides	Fensulfothion	8141A, 1657A	GW	23-133	35	0.001	mg/L	
Pesticides	Fenthion	8141A, 1657A	GW	42-128	24	0.001	mg/L	
Pesticides	Malathion	8141A, 1657A	GW	53-120	24	0.001	mg/L	

App. VII, Ver. 10.0 Date: April 15, 2012 Page 27 of 44

# Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs

This table is subject to revision without notice								
Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit	
Pesticides	Merphos	8141A, 1657A	GW	10-177	34	0.001	mg/L	
Pesticides	Methyl Parathion	8141A, 1657A	GW	47-126	25	0.001	mg/L	
Pesticides	Mevinphos	8141A, 1657A	GW	41-134	23	0.001	mg/L	
Pesticides	Naled	8141A, 1657A	GW	17-155	25	0.001	mg/L	
Pesticides	Phorate	8141A, 1657A	GW	30-139	22	0.001	mg/L	
Pesticides	Ronnel	8141A, 1657A	GW	45-120	23	0.001	mg/L	
Pesticides	Stirophos	8141A, 1657A	GW	47-127	26	0.001	mg/L	
Pesticides	Sulfotep	8141A, 1657A	GW	51-122	23	0.001	mg/L	
Pesticides	Терр	8141A, 1657A	GW	10-137	40	0.0083	mg/L	
Pesticides	Tokuthion (Prothiofos)	8141A, 1657A	GW	47-122	24	0.001	mg/L	
Pesticides	Trichloronate	8141A, 1657A	GW	41-122	24	0.001	mg/L	
Pesticides	Azinphos-Methyl	8141A	SS	50-127	37	0.1	mg/Kg	
Pesticides	Bolstar (Sulprofos)	8141A	SS	55-120	33	0.1	mg/Kg	
Pesticides	Chlorpyrifos	8141A	SS	56-117	28	0.1	mg/Kg	
Pesticides	Coumaphos	8141A	SS	50-126	36	0.1	mg/Kg	
Pesticides	Demeton,-O And -S	8141A	SS	19-153	25	0.1	mg/Kg	
Pesticides	Diazinon	8141A	SS	43-133	28	0.1	mg/Kg	
Pesticides	Dichlorvos	8141A	SS	22-116	32	0.1	mg/Kg	
Pesticides	Dimethoate	8141A	SS	10-142	36	0.1	mg/Kg	
Pesticides	Disulfoton	8141A	SS	52-115	24	0.1	mg/Kg	
Pesticides	Epn	8141A	SS	48-139	35	0.1	mg/Kg	
Pesticides	Ethoprop	8141A	SS	53-112	25	0.1	mg/Kg	
Pesticides	Ethyl Parathion	8141A	SS	52-133	29	0.1	mg/Kg	
Pesticides	Fensulfothion	8141A	SS	26-120	40	0.1	mg/Kg	
Pesticides	Fenthion	8141A	SS	54-121	29	0.1	mg/Kg	
Pesticides	Malathion	8141A	SS	52-123	29	0.1	mg/Kg	
Pesticides	Merphos	8141A	SS	10-193	33	0.1	mg/Kg	
Pesticides	Methyl Parathion	8141A	SS	55-119	29	0.1	mg/Kg	
Pesticides	Mevinphos	8141A	SS	34-114	29	0.1	mg/Kg	
Pesticides	Naled	8141A	SS	10-132	40	0.1	mg/Kg	
Pesticides	Phorate	8141A	SS	54-115	24	0.1	mg/Kg	
Pesticides	Ronnel	8141A	SS	53-112	27	0.1	mg/Kg	
Pesticides	Stirophos	8141A	SS	51-120	35	0.1	mg/Kg	
Pesticides	Sulfotep	8141A	SS	52-124	23	0.1	mg/Kg	
Pesticides	Терр	8141A	SS	10-85	40	0.1	mg/Kg	
Pesticides	Tokuthion (Prothiofos)	8141A	SS	52-124	31	0.1	mg/Kg	
Pesticides	Trichloronate	8141A	SS	50-118	33	0.1	mg/Kg	

App. VII, Ver. 10.0 Date: April 15, 2012 Page 28 of 44

# Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
Pesticides	Alachlor	507	DW	70-130	25	0.0002	mg/L
Pesticides	Atrazine	507	DW	70-130	25	0.0001	mg/L
Pesticides	Butachlor	507	DW	70-130	25	0.0001	mg/L
Pesticides	Metolachlor	507	DW	70-130	25	0.0002	mg/L
Pesticides	Metribuzin	507	DW	70-130	25	0.0002	mg/L
Pesticides	Simazine	507	DW	70-130	25	7.00E-05	mg/L
Pesticides	Aldrin	508	DW	70-130	25	0.0005	mg/L
Pesticides	Dieldrin	508	DW	70-130	25	0.0005	mg/L
Pesticides	Endrin	508	DW	70-130	25	0.0005	mg/L
Pesticides	Gamma BHC	508	DW	70-130	25	0.0005	mg/L
Pesticides	Heptachlor	508	DW	70-130	25	0.0005	mg/L
Pesticides	Heptachlor Epoxide	508	DW	70-130	25	0.0005	mg/L
Pesticides	Hexachlorobenzene	508	DW	70-130	25	0.0005	mg/L
Pesticides	Methoxychlor	508	DW	70-130	25	0.0005	mg/L
Pesticides	4,4-DDD	608/8081A/B, 6630C	GW, WW	60-123	20	0.00005	mg/L
Pesticides	4,4-DDE	608/8081A/B, 6630C	GW, WW	50-120	22	0.00005	mg/L
Pesticides	4,4-DDT	608/8081A/B, 6630C	GW, WW	61-121	20	0.00005	mg/L
Pesticides	Aldrin	608/8081A/B, 6630C	GW, WW	10-136	33	0.00005	mg/L
Pesticides	Alpha BHC	608/8081A/B, 6630C	GW, WW	58-114	21	0.00005	mg/L
Pesticides	Beta BHC	608/8081A/B, 6630C	GW, WW	61-120	20	0.00005	mg/L
Pesticides	Alpha Chlordane	608/8081A/B, 6630C	GW, WW	51-117	21	0.005	mg/L
Pesticides	Delta BHC	608/8081A/B, 6630C	GW, WW	57-120	21	0.00005	mg/L
Pesticides	Dieldrin	608/8081A/B, 6630C	GW, WW	62-123	20	0.00005	mg/L
Pesticides	Endosulfan I	608/8081A/B, 6630C	GW, WW	63-123	20	0.00005	mg/L
Pesticides	Endosulfan II	608/8081A/B, 6630C	GW, WW	63-124	20	0.00005	mg/L
Pesticides	Endosulfan Sulfate	608/8081A/B, 6630C	GW, WW	59-125	21	0.00005	mg/L
Pesticides	Endrin	608/8081A/B, 6630C	GW, WW	60-123	20	0.00005	mg/L
Pesticides	Endrin Aldehyde	608/8081A/B, 6630C	GW, WW	42-92	21	0.00005	mg/L

App. VII, Ver. 10.0 Date: April 15, 2012 Page 29 of 44

	is subject to revision without		Mat	Accuracy	Prec.	DI	TT •4
Class	Analyte	Method	Matrix	(%)	(RPD)	RL	Unit
Pesticides	Endrin Ketone	608/8081A/B, 6630C	GW, WW	60-117	20	0.00005	mg/L
Pesticides	Gamma BHC	608/8081A/B, 6630C	GW, WW	59-116	20	0.00005	mg/L
Pesticides	Heptachlor	608/8081A/B, 6630C	GW, WW	10-131	28	0.00005	mg/L
Pesticides	Heptachlor Epoxide	608/8081A/B, 6630C	GW, WW	61-118	20	0.00005	mg/L
Pesticides	Hexachlorobenzene	608/8081A/B, 6630C	GW, WW	28-116	27	0.00005	mg/L
Pesticides	Methoxychlor	608/8081A/B, 6630C	GW, WW	66-122	20	0.00005	mg/L
Pesticides	Toxaphene	608/8081A/B, 6630C	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1016	608, 6431B, 8082/A	GW, WW	32-126	22	0.0005	mg/L
PCBs	PCB 1221	608, 6431B, 8082/A	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1232	608, 6431B, 8082/A	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1242	608, 6431B, 8082/A	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1248	608, 6431B, 8082/A	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1254	608, 6431B, 8082/A	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1260	608, 6431B, 8082/A	GW, WW	58-128	20	0.0005	mg/L
PCBs	PCB 1016	8082/A	SS	64-120	20	0.017	mg/Kg
PCBs	PCB 1221	8082/A	SS	-	-	0.017	mg/Kg
PCBs	PCB 1232	8082/A	SS	-	-	0.017	mg/Kg
PCBs	PCB 1242	8082/A	SS	-	-	0.017	mg/Kg
PCBs	PCB 1248	8082/A	SS	-	-	0.017	mg/Kg
PCBs	PCB 1254	8082/A	SS	-	-	0.017	mg/Kg
PCBs	PCB 1260	8082/A	SS	72-130	20	0.017	mg/Kg
Pesticides	4,4-DDD	8081A/B	SS	74-114	20	0.02	mg/Kg
Pesticides	4,4-DDE	8081A/B	SS	74-115	20	0.02	mg/Kg
Pesticides	4,4-DDT	8081A/B	SS	62-124	20	0.02	mg/Kg
Pesticides	Aldrin	8081A/B	SS	69-110	20	0.02	mg/Kg
Pesticides	Alpha BHC	8081A/B	SS	68-111	20	0.02	mg/Kg
Pesticides	Beta BHC	8081A/B	SS	74-112	20	0.02	mg/Kg
Pesticides	Delta BHC	8081A/B	SS	71-110	20	0.02	mg/Kg

App. VII, Ver. 10.0 Date: April 15, 2012 Page 30 of 44

# Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs

This table is subject to revision without notice									
Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit		
Pesticides	Dieldrin	8081A/B	SS	76-115	20	0.02	mg/Kg		
Pesticides	Endosulfan I	8081A/B	SS	76-119	20	0.02	mg/Kg		
Pesticides	Endosulfan II	8081A/B	SS	75-116	20	0.02	mg/Kg		
Pesticides	Endosulfan Sulfate	8081A/B	SS	70-118	20	0.02	mg/Kg		
Pesticides	Endrin	8081A/B	SS	68-115	20	0.02	mg/Kg		
Pesticides	Endrin Aldehyde	8081A/B	SS	48-92	20	0.02	mg/Kg		
Pesticides	Endrin Ketone	8081A/B	SS	71-112	20	0.02	mg/Kg		
Pesticides	Gamma BHC	8081A/B	SS	70-112	20	0.02	mg/Kg		
Pesticides	Heptachlor	8081A/B	SS	69-111	20	0.02	mg/Kg		
Pesticides	Heptachlor Epoxide	8081A/B	SS	72-115	20	0.02	mg/Kg		
Pesticides	Hexachlorobenzene	8081A/B	SS	64-111	20	0.02	mg/Kg		
Pesticides	Methoxychlor	8081A/B	SS	65-123	20	0.02	mg/Kg		
Pesticides	Chlordane	8081A/B	SS	-	-	0.2	mg/Kg		
Pesticides	Toxaphene	8081A/B	SS	-	-	0.4	mg/Kg		
Herbicides	2,4,5-TP (SILVEX)	515.1	DW	70-130	25	0.0001	mg/L		
Herbicides	2,4-D	515.1	DW	70-130	25	0.0001	mg/L		
Herbicides	Dalapon	515.1	DW	70-130	25	0.001	mg/L		
Herbicides	Dicamba	515.1	DW	70-130	25	0.0001	mg/L		
Herbicides	Dinoseb	515.1	DW	70-130	25	0.0001	mg/L		
Herbicides	Pentachlorophenol	515.1	DW	70-130	25	0.00004	mg/L		
Herbicides	Picloram	515.1	DW	70-130	25	0.0001	mg/L		
Herbicides	2,4,5-T	1658, 8151A, 6640C	GW, WW	47-120	22	0.002	mg/L		
Herbicides	2,4,5-TP (SILVEX)	1658, 8151A, 6640C	GW, WW	46-125	25	0.002	mg/L		
Herbicides	2,4-D	1658, 8151A, 6640C	GW, WW	39-112	23	0.002	mg/L		
Herbicides	2,4-DB	1658, 8151A, 6640C	GW, WW	29-133	34	0.002	mg/L		
Herbicides	Dalapon	1658, 8151A, 6640C	GW, WW	34-97	35	0.002	mg/L		
Herbicides	Dicamba	1658, 8151A, 6640C	GW, WW	47-119	22	0.002	mg/L		
Herbicides	Dichloroprop	1658, 8151A, 6640C	GW, WW	35-110	23	0.002	mg/L		
Herbicides	Dinoseb	1658, 8151A, 6640C	GW, WW	29-111	27	0.002	mg/L		
Herbicides	МСРА	1658, 8151A, 6640C	GW, WW	34-120	31	0.1	mg/L		
Herbicides	МСРР	1658, 8151A,	GW, WW	16-189	31	0.1	mg/L		

App. VII, Ver. 10.0 Date: April 15, 2012 Page 31 of 44

This table is subject to revision without notice								
Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit	
		6640C						
Herbicides	2,4,5-T	8151A	SS	34-103	22	0.07	mg/Kg	
Herbicides	2,4,5-TP (SILVEX)	8151A	SS	30-123	28	0.07	mg/Kg	
Herbicides	2,4-D	8151A	SS	28/-98	24	0.07	mg/Kg	
Herbicides	2,4-DB	8151A	SS	26-109	32	0.07	mg/Kg	
Herbicides	Dalapon	8151A	SS	28-92	23	0.07	mg/Kg	
Herbicides	Dicamba	8151A	SS	38-109	20	0.07	mg/Kg	
Herbicides	Dichloroprop	8151A	SS	28-91	24	0.07	mg/Kg	
Herbicides	Dinoseb	8151A	SS	10-61	40	0.07	mg/Kg	
Herbicides	МСРА	8151A	SS	22-101	37	6.5	mg/Kg	
Herbicides	МСРР	8151A	SS	13-181	31	6.5	mg/Kg	
PAH	1-Methylnaphthalene	8310, 610, 6440B	GW, WW	37-89	27	0.0001	mg/L	
РАН	2-Methylnaphthalene	8310, 610, 6440B	GW, WW	34-88	28	0.0001	mg/L	
РАН	Acenaphthene	8310, 610, 6440B	GW, WW	45-90	24	0.0001	mg/L	
РАН	Acenaphthylene	8310, 610, 6440B	GW, WW	49-93	24	0.0001	mg/L	
РАН	Anthracene	8310, 610, 6440B	GW, WW	55-101	20	0.0001	mg/L	
РАН	Benzo(a)Anthracene	8310, 610, 6440B	GW, WW	65-112	20	0.0001	mg/L	
РАН	Benzo(a)Pyrene	8310, 610, 6440B	GW, WW	58-105	20	0.0001	mg/L	
РАН	Benzo(b)Fluoranthene	8310, 610, 6440B	GW, WW	63-103	20	0.0001	mg/L	
РАН	Benzo(g,h,i)Perylene	8310, 610, 6440B	GW, WW	47-116	20	0.0001	mg/L	
РАН	Benzo(k)Fluoranthene	8310, 610, 6440B	GW, WW	61-102	20	0.0001	mg/L	
РАН	Chrysene	8310, 610, 6440B	GW, WW	67-106	20	0.0001	mg/L	
РАН	Dibenz(a,h)Anthracene	8310, 610, 6440B	GW, WW	39-115	23	0.0001	mg/L	
PAH	Fluoranthene	8310, 610, 6440B	GW, WW	69-107	20	0.0001	mg/L	
РАН	Fluorene	8310, 610, 6440B	GW, WW	48-95	21	0.0001	mg/L	
РАН	Indeno(1,2,3-cd)Pyrene	8310, 610, 6440B	GW, WW	59-103	20	0.0001	mg/L	
РАН	Naphthalene	8310, 610, 6440B	GW, WW	33-84	29	0.0001	mg/L	
РАН	Phenanthrene	8310, 610, 6440B	GW, WW	58-95	20	0.0001	mg/L	
РАН	Pyrene	8310, 610, 6440B	GW, WW	62-108	20	0.0001	mg/L	
РАН	1-Methylnaphthalene	8310	SS	33-102	25	0.02	mg/Kg	
РАН	2-Methylnaphthalene	8310	SS	32-101	26	0.02	mg/Kg	
PAH	Acenaphthene	8310	SS	39-102	22	0.02	mg/Kg	
PAH	Acenaphthylene	8310	SS	40-104	23	0.02	mg/Kg	
РАН	Anthracene	8310	SS	64-102	20	0.02	mg/Kg	
PAH	Benzo(a)Anthracene	8310	SS	79-100	20	0.02	mg/Kg	

App. VII, Ver. 10.0 Date: April 15, 2012 Page 32 of 44

# Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
РАН	Benzo(a)Pyrene	8310	SS	66-109	20	0.02	mg/Kg
PAH	Benzo(b)Fluoranthene	8310	SS	79-109	20	0.02	mg/Kg
PAH	Benzo(g,h,i)Perylene	8310	SS	75-113	20	0.02	mg/Kg
РАН	Benzo(k)Fluoranthene	8310	SS	75-103	20	0.02	mg/Kg
РАН	Chrysene	8310	SS	79-109	20	0.02	mg/Kg
РАН	Dibenz(a,h)Anthracene	8310	SS	75-105	20	0.02	mg/Kg
РАН	Fluoranthene	8310	SS	80-110	20	0.02	mg/Kg
РАН	Fluorene	8310	SS	48-109	20	0.02	mg/Kg
РАН	Indeno(1,2,3-cd)Pyrene	8310	SS	75-104	20	0.02	mg/Kg
РАН	Naphthalene	8310	SS	28-99	28	0.02	mg/Kg
РАН	Phenanthrene	8310	SS	61-101	20	0.02	mg/Kg
РАН	Pyrene	8310	SS	75-104	20	0.02	mg/Kg
BNA	PYRIDINE	8270C/D 625	GW,WW	11-52	36	0.01	mg/L
BNA	PYRENE	8270C/D 625	GW,WW	65-116	20	0.001	mg/L
BNA	PHENOL	8270C/D 625	GW,WW	10-53	20	0.01	mg/L
BNA	PHENANTHRENE	8270C/D 625	GW,WW	61-110	20	0.001	mg/L
BNA	PENTACHLOROPHENOL	8270C/D 625	GW,WW	10-101	40	0.01	mg/L
BNA	N-OCTADECANE	8270C/D 625	GW,WW	27-136	20	0.01	mg/L
BNA	N- NITROSODIPHENYLAMINE	8270C/D 625	GW,WW	55-98	20	0.01	mg/L
BNA	N-NITRODIPHENYLAMINE	8270C/D 625	GW,WW	10-186	20	0.01	mg/L
BNA	N-NITROSODI-N- PROPYLAMINE	8270C/D 625	GW,WW	50-115	20	0.01	mg/L
BNA	N- NITROSODIMETHYLAMINE	8270C/D 625	GW,WW	12-68	31	0.01	mg/L
BNA	NITROBENZENE	8270C/D 625	GW,WW	39-102	20	0.01	mg/L
BNA	N-DECANE	8270C/D 625	GW,WW	10-96	27	0.01	mg/L
BNA	NAPHTHALENE	8270C/D 625	GW,WW	42-103	20	0.001	mg/L
BNA	ISOPHORONE	8270C/D 625	GW,WW	55-108	20	0.01	mg/L
BNA	INDENO(1,2,3-CD)PYRENE	8270C/D 625	GW,WW	56-129	20	0.01	mg/L
BNA	HEXACHLOROETHANE	8270C/D 625	GW,WW	24-93	25	0.01	mg/L
BNA	HEXACHLOROCYCLOPENT ADIENE	8270C/D 625	GW,WW	20-121	27	0.00	mg/L
BNA	HEXACHLOROBENZENE	8270C/D 625	GW,WW	55-117	20	0.001	mg/L
BNA	HEXACHLORO-1,3- BUTADIENE	8270C/D 625	GW,WW	34-115	22	0.01	mg/L
BNA	FLUORENE	8270C/D 625	GW,WW	58-110	20	0.001	mg/L
BNA	FLUORANTHENE	8270C/D 625	GW,WW	66-120	20	0.001	mg/L
BNA	DI-N-OCTYL PHTHALATE	8270C/D 625	GW,WW	59-143	20	0.001	mg/L

App. VII, Ver. 10.0 Date: April 15, 2012 Page 33 of 44

This table	is subject to revision without notic	e	-			•	-
Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	DI-N-BUTYL PHTHALATE	8270C/D 625	GW,WW	56-133	20	0.001	mg/L
BNA	DIMETHYL PHTHALATE	8270C/D 625	GW,WW	10-152	22	0.001	mg/L
BNA	DIETHYL PHTHALATE	8270C/D 625	GW,WW	33-136	20	0.001	mg/L
BNA	DIBENZOFURAN	8270C/D 625	GW,WW	53-109	20	0.01	mg/L
BNA	DIBENZ(A,H)ANTHRACENE	8270C/D 625	GW,WW	54-130	20	0.001	mg/L
BNA	CHRYSENE	8270C/D 625	GW,WW	65-114	20	0.001	mg/L
BNA	CARBAZOLE	8270C/D 625	GW,WW	62-114	20	0.01	mg/L
BNA	CAPROLACTAM	8270C/D 625	GW,WW	10-30	24	0.01	mg/L
BNA	BIS(2- ETHYLHEXYL)PHTHALATE	8270C/D 625	GW,WW	61-147	20	0.001	mg/L
BNA	BIS(2- CHLOROISOPROPYL)ETHER	8270C/D 625	GW,WW	43-108	20	0.01	mg/L
BNA	BIS(2- CHLOROETHYL)ETHER	8270C/D 625	GW,WW	39-109	23	0.01	mg/L
BNA	BIS(2- CHLORETHOXY)METHANE	8270C/D 625	GW,WW	56-116	20	0.01	mg/L
BNA	BIPHENYL	8270C/D 625	GW,WW	48-105	20	0.01	mg/L
BNA	BENZYLBUTYL PHTHALATE	8270C/D 625	GW,WW	12-166	20	0.001	mg/L
BNA	BENZYL ALCOHOL	8270C/D 625	GW,WW	32-91	20	0.01	mg/L
BNA	BENZOIC ACID	8270C/D 625	GW,WW	10-62	37	0.01	mg/L
BNA	BENZO(K)FLUORANTHENE	8270C/D 625	GW,WW	62-116	20	0.001	mg/L
BNA	BENZO(G,H,I)PERYLENE	8270C/D 625	GW,WW	52-132	20	0.001	mg/L
BNA	BENZO(B)FLUORANTHENE	8270C/D 625	GW,WW	67-114	20	0.001	mg/L
BNA	BENZO(A)PYRENE	8270C/D 625	GW,WW	68-115	20	0.001	mg/L
BNA	BENZO(A)ANTHRACENE	8270C/D 625	GW,WW	68-113	20	0.001	mg/L
BNA	BENZIDINE	8270C/D 625	GW,WW	10-31	40	0.01	mg/L
BNA	BENZALDEHYDE	8270C/D 625	GW,WW	10-56	26	0.01	mg/L
BNA	AZOBENZENE	8270C/D 625	GW,WW	52-113	20	0.01	mg/L
BNA	ATRAZINE	8270C/D 625	GW,WW	61-116	20	0.01	mg/L
BNA	ANTHRACENE	8270C/D 625	GW,WW	65-114	20	0.001	mg/L
BNA	ANILINE	8270C/D 625	GW,WW	30-78	24	0.01	mg/L
BNA	ACETOPHENONE	8270C/D 625	GW,WW	44-98	20	0.01	mg/L
BNA	ACENAPHTHYLENE	8270C/D 625	GW,WW	55-119	20	0.001	mg/L
BNA	ACENAPHTHENE	8270C/D 625	GW,WW	52-107	20	0.001	mg/L
BNA	4-NITROPHENOL	8270C/D 625	GW,WW	10-53	40	0.01	mg/L
BNA	4-NITROANILINE	8270C/D 625	GW,WW	53-135	20	0.01	mg/L
BNA	4-CHLOROPHENYL- PHENYLETHER	8270C/D 625	GW,WW	58-115	20	0.01	mg/L
BNA	4-CHLOROANILINE	8270C/D 625	GW,WW	43-104	20	0.01	mg/L
BNA	4-CHLORO-3-	8270C/D 625	GW,WW	50-105	20	0.01	mg/L

App. VII, Ver. 10.0 Date: April 15, 2012 Page 34 of 44

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
	METHYLPHENOL			(,,,)	()		
BNA	4-BROMOPHENYL- PHENYLETHER	8270C/D 625	GW,WW	63-120	20	0.01	mg/L
BNA	4,6-DINITRO-2- METHYLPHENOL	8270C/D 625	GW,WW	21-119	40	0.01	mg/L
BNA	3-NITROANILINE	8270C/D 625	GW,WW	49-116	20	0.01	mg/L
BNA	3,3-DICHLOROBENZIDINE	8270C/D 625	GW,WW	58-116	20	0.01	mg/L
BNA	3&4-METHYLPHENOL	8270C/D 625	GW,WW	33-94	20	0.01	mg/L
BNA	2-NITROPHENOL	8270C/D 625	GW,WW	40-112	22	0.01	mg/L
BNA	2-NITROANILINE	8270C/D 625	GW,WW	56-122	20	0.01	mg/L
BNA	2-METHYLPHENOL	8270C/D 625	GW,WW	35-84	20	0.01	mg/L
BNA	2-METHYLNAPHTHALENE	8270C/D 625	GW,WW	46-105	20	0.001	mg/L
BNA	2-CHLOROPHENOL	8270C/D 625	GW,WW	37-90	21	0.01	mg/L
BNA	2-CHLORONAPHTHALENE	8270C/D 625	GW,WW	47-106	20	0.001	mg/L
BNA	2,6-DINITROTOLUENE	8270C/D 625	GW,WW	57-110	20	0.01	mg/L
BNA	2,4-DINITROTOLUENE	8270C/D 625	GW,WW	59-117	20	0.01	mg/L
BNA	2,4-DINITROPHENOL	8270C/D 625	GW,WW	10-121	40	0.01	mg/L
BNA	2,4-DIMETHYLPHENOL	8270C/D 625	GW,WW	47-108	20	0.01	mg/L
BNA	2,4-DICHLOROPHENOL	8270C/D 625	GW,WW	46-105	20	0.01	mg/L
BNA	2,4,6-TRICHLOROPHENOL	8270C/D 625	GW,WW	38-113	29	0.01	mg/L
BNA	2,4,5-TRICHLOROPHENOL	8270C/D 625	GW,WW	41-125	27	0.01	mg/L
BNA	1-METHYLNAPHTHALENE	8270C/D 625	GW,WW	45-100	20	0.001	mg/L
BNA	1,4-DICHLOROBENZENE	8270C/D 625	GW,WW	28-94	25	0.01	mg/L
BNA	1,3-DICHLOROBENZENE	8270C/D 625	GW,WW	27-94	25	0.01	mg/L
BNA	1,2-DICHLOROBENZENE	8270C/D 625	GW,WW	30-96	24	0.01	mg/L
BNA	1,2,4-TRICHLOROBENZENE	8270C/D 625	GW,WW	34-97	21	0.01	mg/L
BNA	1,2,4,5- TETRACHLOROBENZENE	8270C/D 625	GW,WW	40-109	20	0.01	mg/L
BNA	PYRIDINE	8270C/D	SS	17-79	27	0.33	mg/Kg
BNA	PYRENE	8270C/D	SS	54-104	20	0.33	mg/Kg
BNA	PHENOL	8270C/D	SS	49-99	20	0.33	mg/Kg
BNA	PHENANTHRENE	8270C/D	SS	55-103	20	0.33	mg/Kg
BNA	PENTACHLOROPHENOL	8270C/D	SS	10-89	28	0.33	mg/Kg
BNA	N-OCTADECANE	8270C/D	SS	33-122	20	0.33	mg/Kg
BNA	N- NITROSODIPHENYLAMINE	8270C/D	SS	48-90	20	0.33	mg/Kg
BNA	N-NITRODIPHENYLAMINE	8270C/D	SS	57-121	20	0.33	mg/Kg
BNA	N-NITROSODI-N- PROPYLAMINE	8270C/D	SS	52-103	20	0.33	mg/Kg
BNA	N-	8270C/D	SS	31-107	23	0.33	mg/Kg

App. VII, Ver. 10.0 Date: April 15, 2012 Page 35 of 44

This table	e is subject to revision without notice			1. 1			
Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
	NITROSODIMETHYLAMINE						
BNA	NITROBENZENE	8270C/D	SS	47-92	20	0.33	mg/Kg
BNA	N-DECANE	8270C/D	SS	31-93	21	0.33	mg/Kg
BNA	NAPHTHALENE	8270C/D	SS	55-91	20	0.33	mg/Kg
BNA	3&4-METHYLPHENOL	8270C/D	SS	60-104	20	0.33	mg/Kg
BNA	ISOPHORONE	8270C/D	SS	51-99-110	20	0.033	mg/Kg
BNA	INDENO(1,2,3-CD)PYRENE	8270C/D	SS	50-83	20	0.33	mg/Kg
BNA	HEXACHLOROETHANE	8270C/D	SS	45-117	20	0.033	mg/Kg
BNA	HEXACHLOROCYCLOPENT ADIENE	8270C/D	SS	36-108	20	0.33	mg/Kg
BNA	HEXACHLOROBENZENE	8270C/D	SS	50-106	20	0.33	mg/Kg
BNA	HEXACHLORO-1,3- BUTADIENE	8270C/D	SS	53-100	20	0.33	mg/Kg
BNA	FLUORENE	8270C/D	SS	59-108	20	0.33	mg/Kg
BNA	FLUORANTHENE	8270C/D	SS	59-119	20	0.33	mg/Kg
BNA	DI-N-OCTYL PHTHALATE	8270C/D	SS	51-114	22	0.33	mg/Kg
BNA	DI-N-BUTYL PHTHALATE	8270C/D	SS	59-106	20	0.33	mg/Kg
BNA	DIMETHYL PHTHALATE	8270C/D	SS	60-105	20	0.33	mg/Kg
BNA	DIETHYL PHTHALATE	8270C/D	SS	61-105	20	0.33	mg/Kg
BNA	DIBENZOFURAN	8270C/D	SS	56-98	20	0.33	mg/Kg
BNA	DIBENZ(A,H)ANTHRACENE	8270C/D	SS	49-111	20	0.33	mg/Kg
BNA	CHRYSENE	8270C/D	SS	55-102	20	0.33	mg/Kg
BNA	CARBAZOLE	8270C/D	SS	51-103	20	0.33	mg/Kg
BNA	CAPROLACTAM	8270C/D	SS	43-104	20	0.33	mg/Kg
BNA	BIS(2- ETHYLHEXYL)PHTHALATE	8270C/D	SS	56-120	20	0.33	mg/Kg
BNA	BIS(2- CHLOROISOPROPYL)ETHER	8270C/D	SS	56-95	20	0.33	mg/Kg
BNA	BIS(2- CHLOROETHYL)ETHER	8270C/D	SS	51-103	20	0.33	mg/Kg
BNA	BIS(2- CHLORETHOXY)METHANE	8270C/D	SS	58-104	20	0.33	mg/Kg
BNA	BIPHENYL	8270C/D	SS	55-93	20	0.33	mg/Kg
BNA	BENZYLBUTYL PHTHALATE	8270C/D	SS	61-118	20	0.33	mg/Kg
BNA	BENZYL ALCOHOL	8270C/D	SS	48-96	20	0.033	mg/Kg
BNA	BENZOIC ACID	8270C/D	SS	10-110	41	0.033	mg/Kg
BNA	BENZO(K)FLUORANTHENE	8270C/D	SS	53-104	20	0.33	mg/Kg
BNA	BENZO(G,H,I)PERYLENE	8270C/D	SS	47-112	20	0.33	mg/Kg
BNA	BENZO(B)FLUORANTHENE	8270C/D	SS	52-106	20	0.33	mg/Kg
BNA	BENZO(A)PYRENE	8270C/D	SS	57-103	20	0.33	mg/Kg

App. VII, Ver. 10.0 Date: April 15, 2012 Page 36 of 44

This table	is subject to revision without notic	ce					
Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	BENZO(A)ANTHRACENE	8270C/D	SS	56-103	20	0.33	mg/Kg
BNA	BENZIDINE	8270C/D	SS			0.033	mg/Kg
BNA	BENZALDEHYDE	8270C/D	SS	10-30	23	0.33	mg/Kg
BNA	AZOBENZENE	8270C/D	SS	49-105	20	0.33	mg/Kg
BNA	ATRAZINE	8270C/D	SS	55-101	20	0.33	mg/Kg
BNA	ANTHRACENE	8270C/D	SS	58-105	20	0.33	mg/Kg
BNA	ANILINE	8270C/D	SS	32-79	23	0.33	mg/Kg
BNA	ACETOPHENONE	8270C/D	SS	49-88	20	0.33	mg/Kg
BNA	ACENAPHTHYLENE	8270C/D	SS	61-107	20	0.033	mg/Kg
BNA	ACENAPHTHENE	8270C/D	SS	55-96	20	0.033	mg/Kg
BNA	4-NITROPHENOL	8270C/D	SS	34-101	26	0.033	mg/Kg
BNA	4-NITROANILINE	8270C/D	SS	41-105	20	0.033	mg/Kg
BNA	4-CHLOROPHENYL- PHENYLETHER	8270C/D	SS	59-103	20	0.033	mg/Kg
BNA	4-CHLOROANILINE	8270C/D	SS	38-89	20	0.33	mg/Kg
BNA	4-CHLORO-3- METHYLPHENOL	8270C/D	SS	58-98	20	0.33	mg/Kg
BNA	4-BROMOPHENYL- PHENYLETHER	8270C/D	SS	58-111	20	0.33	mg/Kg
BNA	4,6-DINITRO-2- METHYLPHENOL	8270C/D	SS	24-98	32	0.33	mg/Kg
BNA	3-NITROANILINE	8270C/D	SS	42-91	20	0.33	mg/Kg
BNA	3,3-DICHLOROBENZIDINE	8270C/D	SS	36-84	20	0.33	mg/Kg
BNA	2-NITROPHENOL	8270C/D	SS	55-106	20	0.33	mg/Kg
BNA	2-NITROANILINE	8270C/D	SS	55-110	20	0.33	mg/Kg
BNA	2-METHYLPHENOL	8270C/D	SS	52-90	20	0.33	mg/Kg
BNA	2-METHYLNAPHTHALENE	8270C/D	SS	57-94	20	0.033	mg/Kg
BNA	2-CHLOROPHENOL	8270C/D	SS	52-88	20	0.33	mg/Kg
BNA	2-CHLORONAPHTHALENE	8270C/D	SS	55-96	20	0.33	mg/Kg
BNA	2,6-DINITROTOLUENE	8270C/D	SS	53-99	20	0.33	mg/Kg
BNA	2,4-DINITROTOLUENE	8270C/D	SS	54-103	20	0.033	mg/Kg
BNA	2,4-DINITROPHENOL	8270C/D	SS	10-109	39	0.33	mg/Kg
BNA	2,4-DIMETHYLPHENOL	8270C/D	SS	52-101	20	0.33	mg/Kg
BNA	2,4-DICHLOROPHENOL	8270C/D	SS	56-96	20	0.33	mg/Kg
BNA	2,4,6-TRICHLOROPHENOL	8270C/D	SS	50-98	20	0.33	mg/Kg
BNA	2,4,5-TRICHLOROPHENOL	8270C/D	SS	48-103	20	0.33	mg/Kg
BNA	1-METHYLNAPHTHALENE	8270C/D	SS	54-90	20	0.33	mg/Kg
BNA	1,4-DICHLOROBENZENE	8270C/D	SS	47-84	20	0.33	mg/Kg
BNA	1,3-DICHLOROBENZENE	8270C/D	SS	47-84	20	0.33	mg/Kg
BNA	1,2-DICHLOROBENZENE	8270C/D	SS	48-86	20	0.33	mg/Kg

App. VII, Ver. 10.0 Date: April 15, 2012 Page 37 of 44

	is subject to revision without notic	, <b>C</b>					
Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	1,2,4-TRICHLOROBENZENE	8270C/D	SS	48-87	20	0.33	mg/Kg
BNA	1,2,4,5- TETRACHLOROBENZENE	8270C/D	SS	52-99	20	0.33	mg/Kg
BNA	PYRIDINE	8270C/D RV	GW,WW	10-74	40	0.01	mg/L
BNA	PYRENE	8270C/D RV	GW,WW	45-176	28	0.001	mg/L
BNA	PHENOL	8270C/D RV	GW,WW	10-69	40	0.01	mg/L
BNA	PHENANTHRENE	8270C/D RV	GW,WW	46-163	29	0.001	mg/L
BNA	PENTACHLOROPHENOL	8270C/D RV	GW,WW	10-128	40	0.01	mg/L
BNA	N-OCTADECANE	8270C/D RV	GW,WW	37-183	39	0.01	mg/L
BNA	N- NITROSODIPHENYLAMINE	8270C/D RV	GW,WW	41-168	37	0.01	mg/L
BNA	2-NITRODIPHENYLAMINE	8270C/D RV	GW,WW	39-156	38	0.01	mg/L
BNA	N-NITROSODI-N- PROPYLAMINE	8270C/D RV	GW,WW	27-157	31	0.01	mg/L
BNA	N- NITROSODIMETHYLAMINE	8270C/D RV	GW,WW	10-96	36	0.01	mg/L
BNA	NITROBENZENE	8270C/D RV	GW,WW	22-154	37	0.01	mg/L
BNA	N-DECANE	8270C/D RV	GW,WW	10-141	36	0.01	mg/L
BNA	NAPHTHALENE	8270C/D RV	GW,WW	26-147	31	0.001	mg/L
BNA	ISOPHORONE	8270C/D RV	GW,WW	36-166	35	0.01	mg/L
BNA	INDENO(1,2,3-CD)PYRENE	8270C/D RV	GW,WW	42-184	32	0.01	mg/L
BNA	HEXACHLOROETHANE	8270C/D RV	GW,WW	10-130	39	0.01	mg/L
BNA	HEXACHLOROCYCLOPENT ADIENE	8270C/D RV	GW,WW	10-142	40	0.00	mg/L
BNA	HEXACHLOROBENZENE	8270C/D RV	GW,WW	38-163	35	0.001	mg/L
BNA	HEXACHLORO-1,3- BUTADIENE	8270C/D RV	GW,WW	18-136	30	0.01	mg/L
BNA	FLUORENE	8270C/D RV	GW,WW	39-163	36	0.001	mg/L
BNA	FLUORANTHENE	8270C/D RV	GW,WW	46-171	37	0.001	mg/L
BNA	DI-N-OCTYL PHTHALATE	8270C/D RV	GW,WW	40-170	28	0.001	mg/L
BNA	DI-N-BUTYL PHTHALATE	8270C/D RV	GW,WW	33-175	39	0.001	mg/L
BNA	DIMETHYL PHTHALATE	8270C/D RV	GW,WW	10-165	37	0.001	mg/L
BNA	DIETHYL PHTHALATE	8270C/D RV	GW,WW	10-182	35	0.001	mg/L
BNA	DIBENZOFURAN	8270C/D RV	GW,WW	35-149	34	0.01	mg/L
BNA	DIBENZ(A,H)ANTHRACENE	8270C/D RV	GW,WW	43-187	31	0.001	mg/L
BNA	CHRYSENE	8270C/D RV	GW,WW	46-170	30	0.001	mg/L
BNA	CARBAZOLE	8270C/D RV	GW,WW	49-165	35	0.01	mg/L
BNA	CAPROLACTAM	8270C/D RV	GW,WW	10-39	37	0.01	mg/L
BNA	BIS(2- ETHYLHEXYL)PHTHALATE	8270C/D RV	GW,WW	42-191	33	0.001	mg/L
BNA	BIS(2-	8270C/D RV	GW,WW	26-149	34	0.01	mg/L

App. VII, Ver. 10.0 Date: April 15, 2012 Page 38 of 44

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
	CHLOROISOPROPYL)ETHER						
BNA	BIS(2- CHLOROETHYL)ETHER	8270C/D RV	GW,WW	22-149	38	0.01	mg/L
BNA	BIS(2- CHLORETHOXY)METHANE	8270C/D RV	GW,WW	34-155	31	0.01	mg/L
BNA	BIPHENYL	8270C/D RV	GW,WW	33-151	32	0.01	mg/L
BNA	BENZYLBUTYL PHTHALATE	8270C/D RV	GW,WW	10-178	40	0.001	mg/L
BNA	BENZYL ALCOHOL	8270C/D RV	GW,WW	22-140	34	0.01	mg/L
BNA	BENZOIC ACID	8270C/D RV	GW,WW	10-75	20	0.01	mg/L
BNA	BENZO(K)FLUORANTHENE	8270C/D RV	GW,WW	42-178	33	0.001	mg/L
BNA	BENZO(G,H,I)PERYLENE	8270C/D RV	GW,WW	42-181	30	0.001	mg/L
BNA	BENZO(B)FLUORANTHENE	8270C/D RV	GW,WW	39-173	32	0.001	mg/L
BNA	BENZO(A)PYRENE	8270C/D RV	GW,WW	39-167	29	0.001	mg/L
BNA	BENZO(A)ANTHRACENE	8270C/D RV	GW,WW	46-167	29	0.001	mg/L
BNA	BENZIDINE	8270C/D RV	GW,WW	10-86	40	0.01	mg/L
BNA	BENZALDEHYDE	8270C/D RV	GW,WW	24-115	34	0.01	mg/L
BNA	AZOBENZENE	8270C/D RV	GW,WW	39-156	31	0.01	mg/L
BNA	ATRAZINE	8270C/D RV	GW,WW	50-149	38	0.01	mg/L
BNA	ANTHRACENE	8270C/D RV	GW,WW	48-167	26	0.001	mg/L
BNA	ANILINE	8270C/D RV	GW,WW	24-120	30	0.01	mg/L
BNA	ACETOPHENONE	8270C/D RV	GW,WW	35-130	32	0.01	mg/L
BNA	ACENAPHTHYLENE	8270C/D RV	GW,WW	34-162	31	0.001	mg/L
BNA	ACENAPHTHENE	8270C/D RV	GW,WW	37-159	30	0.001	mg/L
BNA	4-NITROPHENOL	8270C/D RV	GW,WW	10-61	40	0.01	mg/L
BNA	4-NITROANILINE	8270C/D RV	GW,WW	41-174	36	0.01	mg/L
BNA	4-CHLOROPHENYL- PHENYLETHER	8270C/D RV	GW,WW	39-155	33	0.01	mg/L
BNA	4-CHLOROANILINE	8270C/D RV	GW,WW	37-158	28	0.01	mg/L
BNA	4-CHLORO-3- METHYLPHENOL	8270C/D RV	GW,WW	14-158	40	0.01	mg/L
BNA	4-BROMOPHENYL- PHENYLETHER	8270C/D RV	GW,WW	40-166	36	0.01	mg/L
BNA	4,6-DINITRO-2- METHYLPHENOL	8270C/D RV	GW,WW	10-164	40	0.01	mg/L
BNA	3-NITROANILINE	8270C/D RV	GW,WW	38-153	33	0.01	mg/L
BNA	3,3-DICHLOROBENZIDINE	8270C/D RV	GW,WW	42-150	29	0.01	mg/L
BNA	3&4-METHYLPHENOL	8270C/D RV	GW,WW	11-132	40	0.01	mg/L
BNA	2-NITROPHENOL	8270C/D RV	GW,WW	14-158	40	0.01	mg/L
BNA	2-NITROANILINE	8270C/D RV	GW,WW	38-169	31	0.01	mg/L
BNA	2-METHYLPHENOL	8270C/D RV	GW,WW	19-122	36	0.01	mg/L

App. VII, Ver. 10.0 Date: April 15, 2012 Page 39 of 44

This table	is subject to revision without notic	ce				-	-
Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	2-METHYLNAPHTHALENE	8270C/D RV	GW,WW	27-151	32	0.001	mg/L
BNA	2-CHLOROPHENOL	8270C/D RV	GW,WW	16-129	40	0.01	mg/L
BNA	2-CHLORONAPHTHALENE	8270C/D RV	GW,WW	29-149	34	0.001	mg/L
BNA	2,6-DINITROTOLUENE	8270C/D RV	GW,WW	32-163	30	0.01	mg/L
BNA	2,4-DINITROTOLUENE	8270C/D RV	GW,WW	30-168	32	0.01	mg/L
BNA	2,4-DINITROPHENOL	8270C/D RV	GW,WW	10-135	40	0.01	mg/L
BNA	2,4-DIMETHYLPHENOL	8270C/D RV	GW,WW	19-160	40	0.01	mg/L
BNA	2,4-DICHLOROPHENOL	8270C/D RV	GW,WW	10-157	40	0.01	mg/L
BNA	2,4,6-TRICHLOROPHENOL	8270C/D RV	GW,WW	12-147	40	0.01	mg/L
BNA	2,4,5-TRICHLOROPHENOL	8270C/D RV	GW,WW	12-154	40	0.01	mg/L
BNA	2,3,4,6- TETRACHLOROPHENOL	8270C/D RV	GW,WW	10-152	40	0.001	mg/L
BNA	1-METHYLNAPHTHALENE	8270C/D RV	GW,WW	29-144	31	0.01	mg/L
BNA	1,4-DICHLOROBENZENE	8270C/D RV	GW,WW	15-129	34	0.01	mg/L
BNA	1,3-DICHLOROBENZENE	8270C/D RV	GW,WW	14-127	34	0.01	mg/L
BNA	1,2-DICHLOROBENZENE	8270C/D RV	GW,WW	16-134	33	0.01	mg/L
BNA	1,2,4-TRICHLOROBENZENE	8270C/D RV	GW,WW	18-130	32	0.01	mg/L
BNA	1,2,4,5- TETRACHLOROBENZENE	8270C/D RV	GW,WW	29-121	31	0.01	mg/L
Explosives	1,3,5-Trinitrobenzene	8330A/B	SS	82-105	20	0.5	mg/Kg
Explosives	1,3-Dinitrobenzene	8330A/B	SS		20	0.5	mg/Kg
Explosives	2,4,6-Trinitrotoluene	8330A/B	SS	75-90	20	0.5	mg/Kg
Explosives	2,4-Dinitrotoluene	8330A/B	SS	77-101	20	0.5	mg/Kg
Explosives	2,6-Dinitrotoluene	8330A/B	SS	84-101	20	0.5	mg/Kg
Explosives	2-Nitrotoluene	8330A/B	SS	83-99	20	0.5	mg/Kg
Explosives	3-Nitrotoluene	8330A/B	SS	79-103	20	0.5	mg/Kg
Explosives	4-Nitrotoluene (4-NT)	8330A/B	SS	83-104	20	0.5	mg/Kg
HVDLOCIVAC	Hexahydro-1,3,5-Trinitro-1,3,5- Triazine	8330A/B	SS	81-101	20	0.5	mg/Kg
Explosives	Methyl-2,4,6- Trinitrophenylnitramine	8330A/B	SS	74-101	20	0.5	mg/Kg
-	Nitrobenzene	8330A/B	SS	79-103	20	0.5	mg/Kg
Explosives	Octahydro - 1,3,5,7 -tetranitro- 1,3,5,7-tetrazocine (HMX)	8330A/B	SS	86-108	20	0.0005	mg/Kg

App. VII, Ver. 10.0 Date: April 15, 2012 Page 40 of 44

# Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
Explosives	Pentaerythritol Tetranitrate (PETN)	8330A/B	SS	72-121	21	2	mg/Kg
	Nitroglycerine	8330A/B	SS	63-127	20	2	mg/Kg
Explosives	Nitroguanidine	8330A/B	SS		20	8	mg/Kg
Explosives	1,3,5-Trinitrobenzene	8330A/B	GW	82-105	20	0.0005	mg/L
Explosives	1,3-Dinitrobenzene	8330A/B	GW		20	0.0005	mg/L
Explosives	2,4,6-Trinitrotoluene	8330A/B	GW	75-90	20	0.0005	mg/L
Explosives	2,4-Dinitrotoluene	8330A/B	GW	77-101	20	0.0005	mg/L
Explosives	2,6-Dinitrotoluene	8330A/B	GW	84-101	20	0.0005	mg/L
Explosives	2-Nitrotoluene	8330A/B	GW	83-99	20	0.0005	mg/L
Explosives	3-Nitrotoluene	8330A/B	GW	79-103	20	0.0005	mg/L
Explosives	4-Nitrotoluene (4-NT)	8330A/B	GW	46-87	20	0.0005	mg/L
Explosives	Hexahydro-1,3,5-Trinitro-1,3,5- Triazine	8330A/B	GW	81-101	20	0.0005	mg/L
Explosives	Methyl-2,4,6- Trinitrophenylnitramine	8330A/B	GW	74-101	20	0.0005	mg/L
Explosives	Nitrobenzene	8330A/B	GW	79-103	20	0.0005	mg/L
Explosives	Octahydro - 1,3,5,7 -tetranitro- 1,3,5,7-tetrazocine (HMX)	8330A/B	GW	86-108	20	0.0005	mg/L
Explosives	Pentaerythritol Tetranitrate (PETN)	8330A/B	GW	72-121	20	0.0005	mg/L
Explosives	Nitroglycerine	8330A/B	GW	67-123	20	0.0005	mg/L
GC	1, 2 Dibromoethane (EDB)	504/8011	DW,GW, WW	70 - 130	<30	0.00002	mg/L
GC	1, 2 Dibromo-3-chloropropane	504/8011	DW,GW, WW	70 - 130	<30	0.00002	mg/L
GC	1,2,3-Trichloropropane	504/8011	DW,GW, WW	70 - 130	<30	0.0005	mg/L
THAA	Bromoacetic Acid	552.2	DW	70 - 130	<30	0.001	mg/L
THAA	Chloroacetic Acid	552.2	DW	70 - 130	<30	0.002	mg/L
THAA	Dibromoacetic Acid	552.2	DW	70 - 130	<30	0.001	mg/L
THAA	Dichloroacetic Acid	552.2	DW	70 - 130	<30	0.001	mg/L
THAA	Trichloroacetic Acid	552.2	DW	70 - 130	<30	0.001	mg/L
TPH	Petroleum Range Organics (TRPH)	FL-PRO	GW,	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH)	FL-PRO	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics (TRPH)	EPH TN	GW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH)	EPH TN	SS	50 - 150	<20	4.0	mg/Kg

App. VII, Ver. 10.0 Date: April 15, 2012 Page 41 of 44

	e is subject to revision without notion			Accuracy	Prec.		
Class	Analyte	Method	Matrix	(%)	(RPD)	RL	Unit
ТРН	Petroleum Range Organics (TRPH) - C9-C18, C19-C36, C11-C22	MADEP EPH	GW, WW	50 - 150	<20	0.1	mg/L
ТРН	Petroleum Range Organics (TRPH) - C9-C18, C19-C36, C11-C22	MADEP EPH	SS	50 - 150	<20	5.5	mg/Kg
TPH	Petroleum Range Organics (TRPH) - C10-C28	DRO, 8015Mod	GW, WW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH) - C10-C28	DRO, 8015Mod	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics (TRPH) – C10-C20, C20-C34	OHIO DRO	GW, WW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH) – C10-C20, C20-C34	OHIO DRO	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics (TRPH) – gas, diesel, motor oil, etc.	OA2	GW, WW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH) – gas, diesel, motor oil, etc.	OA2	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics - C10-C28, C28-C40	DRORLA	GW, WW	50 - 150	<20	0.1	mg/L
ТРН	Petroleum Range Organics - C10-C28, C28-C40	DRORLA	SS	50 - 150	<20	4.0	mg/Kg
ТРН	Petroleum Range Organics – C10-C32	DROWY	GW, WW	50 - 150	<20	0.1	mg/L
ТРН	Petroleum Range Organics – C10-C32	DROWY	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics – gas, diesel, motor oil, etc.	NWTPH-Dx	GW, WW	50 - 150	<20	0.25	mg/L
TPH	Petroleum Range Organics – gas, diesel, motor oil, etc.	NWTPH-Dx	SS	50 - 150	<20	25	mg/Kg
TPH	Petroleum Range Organics – C10-C28	DROWM	GW, WW	75 - 115	<20	0.1	mg/L
TPH	Petroleum Range Organics – C10-C28	DROWM	SS	70 - 120	<20	10	mg/Kg
TPH	Petroleum Range Organics – C10-C22	TPHAZ	SS	70-130	<20	30	mg/Kg
TPH	Petroleum Range Organics – C22-C32	TPHAZ	SS	70-130	<20	100.	mg/Kg
ТРН	Petroleum Range Organics – C10-C32	TPHAZ	SS	70-130	<20	130.	mg/Kg
ТРН	Petroleum Range Organics - C6- C12, C12-C28, C28-C35, C6- C35	TX TPH	SS	75 - 125	<20	50	mg/Kg
ТРН	Petroleum Range Organics - C10-C21, C21-C35	DROMO	GW, WW	75 - 125	<20	1.0	mg/L

App. VII, Ver. 10.0 Date: April 15, 2012 Page 42 of 44

	Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RLs         This table is subject to revision without notice									
Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit			
ТРН	Petroleum Range Organics - C10-C21, C21-C35	DROMO	SS	75 - 125	<20	20	mg/Kg			
IH	Aromatic Hydrocarbons	NIOSH 1501	Air	85-115	<20	10	ug/samp le			

# **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 QA 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 will take precedence.

13.2.2 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

<u>Rejection Criteria</u> - Blank reading is more than twice the background absorbance or more than 1/2 RL.

<u>Corrective Action</u> - 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 Manager or QA Department 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)

<u>Rejection Criteria</u> - 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 excedence allowance is surpassed (see section 12.2).

<u>Corrective Action</u> - 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 group leader, Department Manager, or QA Department is consulted for further action.

#### 13.2.4 Out Of Control Matrix Spike Samples

<u>Rejection Criteria</u> - If sample is outside of lab-generated control limits from accuracy charts on matrix spike samples from a similar matrix (i.e., water, solid, etc). Limits are calculated as the mean of at least 20 data points  $\pm 3$  times the standard deviation of those points.

<u>Corrective Action</u> - Spiking technique is assessed to ascertain if the sample has been spiked correctly. The spiked sample should be 1 - 5 times the client sample concentration; otherwise, the percent recovery (%R) or relative percent difference (%RPD) of the MS/MSD is flagged as not meaningful or usable. The sample is re-spiked and re-analyzed, along with several other similar samples in subset. If an out of control situation persists, sample matrix interference is indicated. Samples to be analyzed by standard additions are prepared (where appropriate), and the group leader, Department Manager, or QA Department is notified.

#### 13.2.5 Out Of Control Duplicate Samples

<u>Rejection Criteria</u> - Lab-generated maximum RPD limit (as listed under precision in Section 12)

<u>Corrective Action</u> - 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 before and just after the duplicated sample are re-checked. If problem still exists, Department Manager, or QA Department is notified to review the analytical techniques.

13.2.6 Out Of Control Matrix Spike Duplicates

Rejection Criteria - These QC samples can be out of control for accuracy, precision, or both.

<u>Corrective Action</u> - The appropriate corrective actions listed for either matrix spikes, duplicate samples, or both are followed.

**NOTE**: Some samples cannot be duplicated. This is the case for wipe samples, filters, and some water samples. When possible, sampling personnel should collect duplicate samples.

13.2.7 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

<u>Corrective Action</u> - 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 group leader, Department Manager, or QA Department will be consulted for further action.

## 14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* 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 8.0.

App. VIII, Ver. 10.0 Date: April 15, 2012 Page 1 of 20

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

ESC LAB SCIENCES **12065 LEBANON ROAD MT. JULIET, TENNESSEE 37122** (615) 758-5858

NOTE: The QAM has been approved by the following people. A signed cover page is available upon request

Morgan, M.S., VP of Technical and Regulatory Affairs 615-773-9657

Eric Johnson, B.S., Laboratory Director 615-773-9654

arlis

Dixie Marlin, B.S. QC Manager, 615-773-9681

uckley, B.S., Organis/Wet Chemistry Department Manager, 615-773-9686

# 2.0 APPENDIX TABLE OF CONTENTS

Section	Section Title			Rev. Date	<i>Rev.</i> #
1.0	Approval and Signature Page	Page	1	4/15/11	2
2.0	Table of Contents	Page	2	4/15/11	2
3.0	Scope and Application	Page	3	4/15/11	2
4.0	Laboratory Organization and Responsibility	Page	3	4/15/11	2
5.0	Personnel and Training	Page	3	4/15/11	2
6.0	Facilities and Laboratory Safety	Page	3	4/15/11	2
7.0	Sampling Procedures	Page	4	4/15/11	2
8.0	Equipment	Page	5	4/15/11	2
9.0	Laboratory Practices	Page	11	4/15/11	2
10.0	Analytical Procedures	Page	13	4/15/11	2
11.0	Quality Control Checks	Page	14	4/15/11	2
12.0	Data Reduction, Validation and Reporting	Page	15	4/15/11	2
13.0	Corrective Actions	Page	18	4/15/11	2
14.0	Record Keeping	Page	19	4/15/11	2
15.0	Quality Audits	Page	20	4/15/11	2
	TABLES				
8.1	Equipment	Page	5	4/15/11	2
8.2	Equipment Preventative Maintenance, Equipment Calibration	Page	5	4/15/11	2
8.3A	Standards and Reagents	Page	6	4/15/11	2
8.3B	Working Standards	Page	6	4/15/11	2
8.5	Instrument Calibration	Page	10	4/15/11	2
10.1	Semi-Volatile Department SOPs	Page	13	4/15/11	2
12.1	Data Reduction Formulas	Page	15	4/15/11	2
12.3	QC Targets and RLs	Page	16	4/15/11	2

## **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 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 8.0*.

## 5.0 PERSONNEL AND TRAINING

## 5.1 **PERSONNEL**

Kenneth W. Buckley, with a B.S. degree in General Science, is the Department Manager of Organics and Wet Chemistry laboratories. Mr. Buckley reviews and approves all data reduction associated with analyses in these areas and is responsible for the overall production of these laboratories; including the management of the staff and scheduling. Mr. Buckley has 12 years of environmental laboratory experience. In his absence, J. D. Gentry, with a B.S. degree in Chemistry and 11 years of environmental laboratory experience, assumes responsibility for Air Department decisions.

### 5.2 TRAINING

The primary analyst or Manager 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). 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 and Safety 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 for air analysis are collected in four ways:
  - Ø Samples may be collected directly in evacuated Summa canisters fit with the appropriately adjusted regulator that controls sampling flow to fill the canister over a given time period.
  - Ø Summa canisters may also be collected as "grab" samples by simply opening the canister without the aid of a flow regulator and allowing the canister to fill quickly by virtue of the canister vacuum.
  - Ø 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 to the bag to sample the air over the appropriate timeframe needed by the client.
  - Ø The headspace of containers housing water samples may also be analyzed for specific volatile components.

App. VIII, Ver. 10.0 Date: April 15, 2012 Page 5 of 20

- 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 shipping. 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 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

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Air Analysis This table is subject to revision without notice						
Item	n wunout notice Manufacturer	Model	Instrument Name	#	Serial #	Location
Gas Chromatograph	HP	6890N TCD	AIRGC1	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
Preconcentrator	Entech	7200			0197	Air Lab
Canister Autosampler	Entech	7016C			0203	Air Lab
Preconcentrator	Entech	7100A			1089	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	7016CA			1137	Air Lab
Tedlar Autosampler	Entech	7032A-L			1017	Air Lab
GC/FID	Agilent	6890N	AIRGC2	2	US10137006	Air Lab

#### **LABORATORY EQUIPMENT LIST:** MAJOR ITEMS - Air Analysis This table is subject to revision without notice

This indie is subject to revision without notice						
Item	Manufacturer	Model	Instrument Name	#	Serial #	Location
Headspace Autosampler	Tekmar	7000			9507018	Air Lab
TO Canister	Restek/Entech	TO-Can/ SiloniteCan	1120 cans owned		N/A	Air Lab
Passive Sampling Kit	Restek		505 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 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
0 1	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/Mass Spectrometer & Gas Chromatographs	•Replace column	When separation begins to degrade

# 8.3 STANDARDS AND REAGENTS

Table 8.3A: Standard stock sources, description and calibration information.         This table is subject to revision without notice					
Method	Vendor	Description	Conc.	Storage Req.	Expiration
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>6</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) 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

App. VIII, Ver. 10.0 Date: April 15, 2012 Page 7 of 20

,	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 ppbv	225 cc	45L in 15L Canister	1 year		

\* see analytes listed in Table 12.3.

# 8.4 INSTRUMENT CALIBRATION

### <u>TO-15, 8260B(Ohio VAP Air), Gasoline Range Components (Method 18) – Volatiles in</u> <u>Air by GC/MS – SOP Numbers 330367, 330368, & 330369</u>

Detector mass calibration is performed daily 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 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 24 hours for method TO-15 and Method 18 and every 12 hours for method 8260B.

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 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 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).

SPCCs:			
Analyte	Minimum Relative		
Allaryte	Response Factor		
Chloromethane	0.10		
1,1-Dichloroethane	0.10		
Bromoform	0.10		
Chlorobenzene	0.30		
1,1,2,2-Tetrachloroethane	0.30		

CO	CCs:
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. Initial 8260B calibration that does not meet these requirements is not accepted and re-calibration must be performed. Linear regression can be used for any target compound exceeding the 15% RSD criteria providing that the correlation coefficient is 0.990 or better.

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 retuned and either recalibrated or the existing calibration may be verified prior to further sample analysis.

App. VIII, Ver. 10.0 Date: April 15, 2012 Page 9 of 20

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 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.

### Landfill Gases (Carbon Dioxide, Carbon Monoxide, Methane, Nitrogen, Oxygen) – SOP Number 330366

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 three concentration levels from purchased certified standards. Generation of the initial calibration is performed using Chrom-Perfect Spirit software and a linear regression model. The correlation coefficient must be 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 30% of the expected concentration. Each sample is analyzed in triplicate and the average sample area for each compound is calculated. The sample results are considered acceptable when the injections agree within 5% of their average. If this criteria is not met, additional injections are analyzed until consistent area data is obtained.

# Methane, Ethane, Ethene, Propane 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 three concentration levels. The 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. 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, 100 uL 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.

# 8.5 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	Massm/z Abundance Criteria508-40% of mass 957530-66% of mass 9595Base peak, 100%965-9% of mass 95173<2% of mass 174	TO-15/ M-18: Every 24 hours 8260 VAP: Every 12 hours
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	Average Response Factor: Target analytes in the calibration standards must be <15% RSD, CCCs must	As needed

	TABLE 8.5: INSTRUMENT CALIBRATION & QC				
Analysis/ Instrument	Calibration Type	Number of Standards	Acceptance/ Rejection Criteria	Frequency	
			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		
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	< <sup>1</sup> / <sub>2</sub> 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	< <sup>1</sup> / <sub>2</sub> 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	
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	< <sup>1</sup> / <sub>2</sub> RL, concentrations of common laboratory contaminants shall not exceed the reporting limit	Following init. calibration or daily cal. verification	

	TABLE 8.5: INSTRUMENT CALIBRATION & QC					
Analysis/ Instrument	Calibration Type	Number of Standards	Acceptance/ Rejection Criteria	Frequency		
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		

# 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. Conductivity and pH are checked continuously or with each use.

# 9.2 SAMPLER 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 = $120^{\circ}C$
1	Initial evacuation of canister to 1000 mtorr
2	Refill canister to 20psi
3	Evacuate the canister to 50 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 may be 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 precleaned from approved providers.

# 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 = $100^{\circ}$ C	Module 2 Desorb = $180^{\circ}$ C
Bulk Head $1 = 30^{\circ}$ C	Module 2 Bake = $190^{\circ}$ C
Bulk Head $2 = 30^{\circ}$ C	Module 2 Desorb Time = 3.5 min
Module 1 Trap = $-150^{\circ}$ C	Module 3 Trap = $-180^{\circ}$ C
Module 1 Preheat = $20^{\circ}$ C	Module 3 Inject = $2 \min$
Module 1 Desorb = $20^{\circ}$ C	Module 3 Bake Time = 2 min
Module 1 Bake = $130^{\circ}$ C	Module 3 Event = 3
Module 1 Bake Time = 5 min	Module 3 Wait Time = 25 min.
Module 2 Trap = $-30^{\circ}$ 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:

This Table is subject to revision without notice				
SOP #	Title/Description			
330366	Determination of Carbon Dioxide, Carbon Monoxide, Methane,			
330300	Nitrogen and Oxygen in Air Samples.			
330367	Measurement of Volatile Organic Compound in Ambient Air by GC/MS (EPA TO-15)			
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			
330309	(with provisions for GRO determination based on 8015B)			
330370	Method for Determination of Methane, Ethane, and Ethene (Based on RSK-175)			

#### TABLE 10.1: AIR DEPARTMENT SOPs

#### 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-l0mL) 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. 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 and RPD of 25%. 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 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 SOP #030201, *Data Handling and Reporting*. The Quality Control Department performs the secondary review of the data package using the ESC SOP #030227, *Data Review*. The QC Reviewer verifies that the analysis has 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/MS – Analyte Response Factor	response of analyte primary ion { <i>area</i> } x concentration of analyte (ug/L) response of ISTD primary ion { <i>area</i> }_x concentration of ISTD (ug/L)			
	Calculations performed by HP Enviroquant Software			
GC/MS – Sample Analyte Concentration	response of primary ion in analyte x int. std concentration. {ppbv} x dilution factor response factor {area/(mg/ml)} x initial volume-mass {ml or g} x int. std cal. {area}			

# **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.

<u>Organic Control Limits -</u> The organic QC targets are statutory in nature; warning and control limits for organic analyses are initially set 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 Air Accuracy (LCS), Precision and RLs         This table is subject to revision without notice						
Analyte	Method	Matrix	Accuracy (%)	Prec. (% RPD)	RL	Unit
1,1,1-Trichloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,1,2,2-Tetrachloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,1,2,2-Tetrachloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,1,2-Trichloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,1-Dichloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,1-Dichloroethene	TO-15	Air	70-130	25	0.2	ppbv
1,2,4-Trichlorobenzene	TO-15	Air	70-130	25	0.63	ppbv

App. VIII, Ver. 10.0 Date: April 15, 2012 Page 17 of 20

# Table 12.3: QC Targets for Air Accuracy (LCS), Precision and RLs This table is subject to revision without notice

Analyte	Method	Matrix	Accuracy (%)	Prec. (% RPD)	RL	Unit
1,2,4-Trimethylbenzene	TO-15	Air	70-130	25	0.2	ppbv
1,2-Dibromoethane	TO-15	Air	70-130	25	0.2	ppbv
1,2-Dichlorobenzene	TO-15	Air	70-130	25	0.2	ppbv
1,2-Dichloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,2-Dichloropropane	TO-15	Air	70-130	25	0.2	ppbv
1,3,5-Trimethylbenzene	TO-15	Air	70-130	25	0.2	ppbv
1,3-Butadiene	TO-15	Air	70-130	25	0.2	ppbv
1,3-Dichlorobenzene	TO-15	Air	70-130	25	0.2	ppbv
1,4-Dichlorobenzene	TO-15	Air	70-130	25	0.2	ppbv
1,4-Dioxane	TO-15	Air	70-130	25	0.2	ppbv
1,1,1-Trichloroethane	TO-15	Air	70-130	25	0.2	ppbv
2,2,4-Trimethylpentane	TO-15	Air	70-130	25	0.2	ppbv
2-Chlorotoluene	TO-15	Air	70-130	25	0.2	ppbv
2-Propanol	TO-15	Air	70-130	25	0.2	ppbv
4-Ethyltoluene	TO-15	Air	70-130	25	0.2	ppbv
Acetone	TO-15	Air	70-130	25	1.25	ppbv
Allyl Chloride	TO-15	Air	70-130	25	0.2	ppbv
Benzene	TO-15	Air	70-130	25	0.2	ppbv
Benzyl Chloride	TO-15	Air	70-130	25	0.2	ppbv
Bromomethane	TO-15	Air	70-130	25	0.2	ppbv
Bromodichloromethane	TO-15	Air	70-130	25	0.2	ppbv
Bromoform	TO-15	Air	70-130	25	0.6	ppbv
Carbon Disulfide	TO-15	Air	70-130	25	0.2	ppbv
Carbon Tetrachloride	TO-15	Air	70-130	25	0.2	ppbv
Chlorobenzene	TO-15	Air	70-130	25	0.2	ppbv
Chloroethane	TO-15	Air	70-130	25	0.2	ppbv
Chloroform	TO-15	Air	70-130	25	0.2	ppbv
Chloromethane	TO-15	Air	70-130	25	0.2	ppbv
Cis-1,2-Dichloroethene	TO-15	Air	70-130	25	0.2	ppbv
Cis-1,3-Dichloropropene	TO-15	Air	70-130	25	0.2	ppbv
Cyclohexane	TO-15	Air	70-130	25	0.2	ppbv
Dibromochloromethane	TO-15	Air	70-130	25	0.2	ppbv
Ethanol	TO-15	Air	70-130	25	0.63	ppbv
Ethyl Acetate	TO-15	Air	70-130	25	0.2	ppbv
Ethylbenzene	TO-15	Air	70-130	25	0.2	ppbv

### **ESC Lab Sciences Air Quality Assurance Manual** Appendix VIII to the ESC QAM

App. VIII, Ver. 10.0 Date: April 15, 2012 Page 18 of 20

# Table 12.3: QC Targets for Air Accuracy (LCS), Precision and RLs This table is subject to revision without notice

This table is subject to revision w	ithout notice			1		
Analyte	Method	Matrix	Accuracy (%)	Prec. (% RPD)	RL	Unit
Freon-11	TO-15	Air	70-130	25	0.2	ppbv
Freon-12	TO-15	Air	70-130	25	0.2	ppbv
Freon-113	TO-15	Air	70-130	25	0.2	ppbv
Freon-114	TO-15	Air	70-130	25	0.2	ppbv
Gasoline Range Organics	TO-15	Air	70-130	25	50	ppbv
Heptane	TO-15	Air	70-130	25	0.2	ppbv
Hexachloro-1,3-Butadiene	TO-15	Air	70-130	25	0.63	ppbv
Hexane	TO-15	Air	70-130	25	0.2	ppbv
Isopropylbenzene	TO-15	Air	70-130	25	0.2	ppbv
M&P-Xylene	TO-15	Air	70-130	25	0.4	ppbv
Methyl Butyl Ketone	TO-15	Air	70-130	25	1.25	ppbv
Methyl Ethyl Ketone	TO-15	Air	70-130	25	1.25	ppbv
Methyl Isobutyl Ketone	TO-15	Air	70-130	25	1.25	ppbv
Methyl Methacrylate	TO-15	Air	70-130	25	0.2	ppbv
Methyl tert Butyl Ether	TO-15	Air	70-130	25	0.31	ppbv
Methylene Chloride	TO-15	Air	70-130	25	0.63	ppbv
Naphthalene	TO-15	Air	70-130	25	0.63	ppbv
o-Xylene	TO-15	Air	70-130	25	0.2	ppbv
Propene	TO-15	Air	70-130	25	0.4	ppbv
Styrene	TO-15	Air	70-130	25	0.2	ppbv
t-Butyl Alcohol	TO-15	Air	70-130	25	0.2	ppbv
Tetrachloroethylene	TO-15	Air	70-130	25	0.2	ppbv
Tetrahydrofuran	TO-15	Air	70-130	25	0.2	ppbv
Toluene	TO-15	Air	70-130	25	0.2	ppbv
Trans-1,3-Dichloropropene	TO-15	Air	70-130	25	0.2	ppbv
Trans-1,2-Dichloroethene	TO-15	Air	70-130	25	0.2	ppbv
Trichloroethylene	TO-15	Air	70-130	25	0.2	ppbv
Vinyl Acetate	TO-15	Air	70-130	25	0.2	ppbv
Vinyl Bromide	TO-15	Air	70-130	25	0.2	ppbv
Vinyl Chloride	TO-15	Air	70-130	25	0.2	ppbv
Methane	RSK-175	Air/ Headspace	70-130	25	0.01	ppmv
Ethane	RSK-175	Air/ Headspace	70-130	25	0.129	ppbmv
Ethene	RSK-175	Air/ Headspace	70-130	25	0.127	ppmv
Propane	RSK-175	Air/	70-130	25		ppmv

App. VIII, Ver. 10.0 Date: April 15, 2012 Page 19 of 20

Table 12.3: QC Targets for Air Accuracy (LCS), Precision and RLs         This table is subject to revision without notice						
Analyte	Method	Matrix	Accuracy (%)	Prec. (% RPD)	RL	Unit
		Headspace				
Carbon Dioxide	Method 3C	Air	70-130	25	0.50 / 200	% / ppmv
Carbon Monoxide	Method 3C	Air	70-130	25	0.50 / 200	% / ppmv
Methane	Method 3C	Air	70-130	25	0.50 / 200	% / ppmv
Nitrogen	Method 3C	Air	70-130	25	0.50 / 200	% / ppmv
Oxygen	Method 3C	Air	70-130	25	0.50 / 200	% / ppmv

# **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 reason for the nonconformance 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 QA department. Corrective action procedures are documented in SOP #030208, Corrective and Preventive Action
  - **Required Corrective Action** 13.2

All samples and procedures are governed by ESC's quality assurance program. Designated corrective actions are as follows.

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 will take 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 1/2 the RL.

<u>Corrective Action</u> - 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)

<u>Rejection Criteria</u> - If the performance is outside of lab-generated control (Listed in Table 12.3).

<u>Corrective Action</u> - 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 #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* 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 8.0.

ESC Lab Sciences Aquatic Toxicity Lab Quality Assurance Manual Appendix IX to the ESC QAM

App. IX, Ver. 10.0 Date: April 15, 2012 Page 1 of 20

**1.0** SIGNATORY APPROVALS

# Aquatic Toxicity Laboratory QUALITY ASSURANCE MANUAL

# APPENDIX IX 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. A signed cover page is available upon request

chnical and Regulatory Affairs, 615-773-9657 rgan

ric Johnson, B.S., Laboratory Director, 615-773-9654

arla Dixie Marlin, B.S., OC Manager, 615-773-9681

Disie marini, D.S., QC Matager, 013-775-9081

NAT

Kim Johnson, B.S., Aquatic Toxicity Department Manager, 615-773-9687

App. IX, Ver. 10.0 Date: April 15, 2012 Page 2 of 20

# 2.0 APPENDIX TABLE OF CONTENTS

Section	Section Title			Rev. Date	<i>Rev.</i> #
1.0	Approval and Signature Page	Page	1	4/15/11	2
2.0	Table of Contents	Page	2	4/15/11	2
3.0	Scope and Application	Page	3	4/15/11	2
4.0	Laboratory Organization and Responsibility	Page	3	4/15/11	2
5.0	Personnel and Training	Page	3	4/15/11	2
6.0	Facilities and Laboratory Safety	Page	3	4/15/11	2
7.0	Sampling Procedures	Page	4	4/15/11	2
8.0	Equipment	Page	5	4/15/11	2
9.0	Laboratory Practices	Page	10	4/15/11	2
10.0	Analytical Procedures	Page	12	4/15/11	2
11.0	Quality Control Checks	Page	13	4/15/11	2
12.0	Data Reduction, Validation and Reporting	Page	15	4/15/11	2
13.0	Corrective Actions	Page	16	4/15/11	2
14.0	Record Keeping	Page	17	4/15/11	2
15.0	Quality Audits	Page	17	4/15/11	2
	TABLES				
8.1	Equipment	Page	5	4/15/11	2
8.2	Equipment Preventative Maintenance,	Page	6	4/15/11	2
0.2	Equipment Calibration	rage	0		
8.3A	Stock Solutions and Storage	Page	7	4/15/11	2
8.3B	Working Solutions and Storage	Page	7	4/15/11	2
10.1	Aquatic Toxicity Department SOPs	Page	12	4/15/11	2
12.1	Data Reduction Formulas	Page	15	4/15/11	2

# **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 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 8.0*.

# 5.0 PERSONNEL AND TRAINING

### 5.1 **PERSONNEL**

Kim Johnson, with a B.S. degree in Biological Sciences, is the Department Manager of the Aquatic Toxicity laboratory. Ms. Johnson reviews and approves all data reduction associated with Aquatic Toxicity analysis. Her responsibilities include the coordination with clients regarding sample analysis for regulatory compliance, scheduling of testing and personnel, and data reduction, interpretation and validation for Toxicity analyses. Ms. Johnson is also involved in microbiological assessments of wastewater, sludges and drinking water and oversees the Protozoan laboratory. She is also a certified mold analyst. In her absence, Shain Schmitt assumes responsibility for departmental decisions.

### 5.2 TRAINING

All new analysts to the laboratory will be 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*.

### 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 Siemans Elga UltraPure deionizer system. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's hazardous waste disposal company. 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 chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods, where applicable.

ESC's laboratory safety guidelines are detailed in *the ESC Chemical Hygiene and Safety 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. 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*.

#### ESC Lab Sciences Aquatic Toxicity Lab Quality Assurance Manual Appendix IX to the ESC QAM

• 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:

- Ø Temperature recorded up to twice daily.
- Ø pH initial and final measurements recorded
- Ø D.O. initial and final measurements recorded
- Ø Specific Conductance
- **Ø** Alkalinity
- Ø Hardness
- Ø Total Residual Chlorine
- Samples must be immediately cooled and maintained at 0-6°C during shipment and prior to testing.

### **Residual Chlorine Treatment**

**§** Residual chlorine in biomonitoring samples are monitored using a pocket colorimeter and these checks are documented. Chlorine removal is not performed.

### 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  $\leq 4.0$  mg/L are aerated until the sample reaches 90% saturation.

# 8.0 EQUIPMENT

# 8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Aquatic Toxicity Lab This table is subject to revision without notice.					
Item	Manufacturer	Model	Location		
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		
pH Meter	Beckman	pH/Temp/mV/ISE	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	Siemans	Elga Purelab	Aquatic Tox Lab		
Refrigerator	Fridgidaire	FRC 445GB	Aquatic Tox Lab		
Freezer	Kenmore	198130582	Aquatic Tox Lab		

# 8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

PREVENTATIVE MAINTENANCE FOR LABORATORY EQUIPMENT					
INSTRUMENT	P. M. DESCRIPTION	FREQUENCY			
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			
Conductivity Meter	•Check probe cables	As needed			
Conductivity Meter	•Clean probe	Daily			
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	Each Use			
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)			
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			

# 8.3 STANDARDS, REAGENTS AND ORGANISM CULTURES

All reagents and standards must meet the requirements listed in the analytical methods.

Table 8.3A: Stock solution sources, description and related information.         (subject to revision as needed)					
Description	Vendor	Storage Req.	Expiration		
Conductivity standard 100	Fisher	Ambient	1 yr		
Conductivity standard 1000	Fisher	Ambient	1 yr		
pH buffer 7	Fisher	Ambient	1 yr		
pH buffer 10	Fisher	Ambient	1 yr		
Bromothymol blue solution	Fisher	Ambient	1 yr		
Potassium phosphate monobasic	Fisher	Ambient	1 yr		
Magnesium chloride	JT Baker	Ambient in dessicator	1 yr		
Potassium Chloride	EMD	Ambient in dessicator	1 yr		
Brine shrimp eggs	Argentemia	Ambient, tightly sealed.	1 yr		
Calcium sulfate	EM	Ambient in dessicator	1 yr		
EDTA	Fisher	Ambient in dessicator	1 yr		
Sodium thiosulfate	JT Baker	Ambient in dessicator	1 yr		
pH buffer 4	Fisher	Ambient.	1 yr		
YCT	Made in-house	-10 to -20°C	14 days after thawing		
Selenastrum capricornatum	Aq. Biosystems	1-6°C	NA		
Vitamin B12	Fisher	1-6°C	NA		

TABLE 8.3B: Working Solution Descriptions and Related Information.							
	(subject to change)						
Solution	Concentrations	Storage Requirements	Expiration				
KCl stock solution	31.237g KCl to 2L of 20% DMW	1-4°C	14 days				

#### Source and *Maintenance of in-house cultures*:

**B12** Solution

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 regards to the taxonomic identification of the bioassay species. The packing slips for bioassay test organisms are kept on file.

0.01125g to 1L of DI Water

 $1-4^{\circ}C$ 

NA

The amount of food added to culture vessels will depend 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 daily as needed. The date, time and the amount the organisms are fed are documented. All brewers yeast purchased is at least food grade and has passed FDA standards. All 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 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 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.

The water used for culturing is dilute mineral water prepared by diluting (6) 750mL bottles of Perrier to 20 Liters with deionized water and aerating for 24 hours. The physical and chemical parameters for each new tank of water prepared are recorded and should fall within the following acceptable range:

- 1. pH 7.9 to 8.3 units
- 2. D.O. greater than 80% saturation in mg/L
- 3. Specific Conductance ~215 micromhos/cm
- 4. Alkalinity 80-100 mg CaCO<sub>3</sub>/L
- 5. Hardness 80 to  $100 \text{ mg CaCO}_3/\text{L}$
- 6. Total Residual Chlorine <0.1 mg/L

*Pimephales promelas* are taxonomically identified to species on a quarterly basis. All taxonomy information is documented and kept on file for a year.

### 8.4 INSTRUMENT CALIBRATION

### <u>Lighting</u>

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 and is verified and documented semi-annually. All incubators are monitored at least weekly for proper light intensity.

### <u>pH Meter</u>

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 95-105%. 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. Volumetric equipment that is not disposed of after use is calibrated on an annual basis. The error of calibration must not exceed 2.5%.

### 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.

### <u>Stereoscope</u>

All glass surfaces are kept clean using a 3:7 mixture of alcohol and ether or a small amount of xylene. Maintenance is performed by a trained technician on an annual basis.

### **Conductivity Meter**

With each use of conductivity meters, calibrate the instrument according to manufacturer's instructions.

### Dissolved Oxygen Meter

With each use of the DO meter, calibrated according to manufacturer's instructions. The probe membrane is changed every two to four weeks to maintain accurate readings.

### 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 the instrument is accurate.

# 9.0 LABORATORY PRACTICES

### 9.1 REAGENT GRADE WATER

Deionized water or reverse-osmosis produces water free from bactericidal and inhibitory substances and shall be used in the preparation of media, solutions and buffers. The quality of the water shall be 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 or Use Test (to determine presence of toxic agents or growth promoting substances) shall be performed annually. Results of these analyses shall meet the specifications of the required method and records of analyses shall be maintained for five years. (An exception to performing the Bacteriological Water Quality Test shall 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 SPECÖ SECONDARY STANDARDS

Standards are used for retrieval and verification of the factory calibrated colorimeter and is used to verify consistent instrument calibration.

### 9.4 LABORATORY CONTROL WATER

Control water (20% dilute mineral water) is prepared by diluting (6) 750mL bottles of Perrier to 20 Liters with deionized water and aerating for 24 hours. The physical and chemical parameters for each new tank of water prepared are recorded and should fall within the following acceptable range:

- 1. pH 7.9 to 8.3 units
- 2. D.O. greater than 80% saturation in mg/L
- 3. Specific Conductance ~215 micromhos/cm
- 4. Alkalinity 57 to 64 80-100 mg  $CaCO_3/L$
- 5. Hardness 80 to 100 mg  $CaCO_3/L$
- 6. Total Residual Chlorine <0.1 mg/L

Control water (10% dilute mineral water) is prepared by diluting (3) 750mL bottles of Perrier to 20 Liters with deionized water and aerating for 24 hours. 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 ~215 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 <0.1 mg/L

A given batch of control water is not used for more than 14 days following preparation.

### 9.5 BRINE SHRIMP

Artemia cysts are of platinum or gold grade, certified brine shrimp eggs from ARGENT chemical Laboratories. To determine the quality of the new lots of Brine shrimp, a sideby-side comparison test is performed using the new food and the food of known acceptable quality.

# 9.6 YCT

YCT 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 ALGAE

Algae is 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 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 350334 Sterilization, Sanitization and Residue Testing of Microbiological Glassware and Equipment.* 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 will be cleaned according to the same procedure as listed above except the first step will be 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 presterilized. 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

A list of laboratory SOPs associated with the microbiology laboratory can be found in the 10.1 following table:

SOP #	This Table is subject to revision without notice 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, Ceriodaphnia dubia, Chronic Survival and Reproduction Test, EPA Method		
	1002.0		
350303	Pimephales promelas Acute Toxicity Testing		
350303NC	North Carolina Pimephales promelas Acute Toxicity Testing		
350304	Ceriodaphnia dubia Acute Toxicity Testing		
350304NC	North Carolina Ceriodaphnia dubia Acute Toxicity Testing		
350305	Fecal Coliform Membrane Filter Technique		
350315	Fecal Coliform Determination in Biosolids: Membrane Filter Technique		
350316	Total Coliform: Membrane Filter Technique		
350317	WET Reference toxicant testing		
350318	Mini Chronic C. dubia 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>		
350331	Salmonella Determination and Enumeration in Biosolids by Most Probable Number		
350332	Laboratory Maintenance of Bacteria Reference Cultures		
350333	Quality Control and Quality Assurance of Microbiological Equipment and Testing Materials		
350343	Total Coliform and <i>E. Coli</i> Enumeration by the Enzyme Substrate Method		
350344	M-Coliblue: Membrane Filter Technique		
350345	Receipt and Maintenance of Pimephalas Promelas (Fathead Minnow)		
350346	Ceriodaphnia Dubia Culture Maintenance, Food Preparation, and Food Maintenance		
350348	Enterococci Enumeration by the Defined Substrate Method Enterolert		
350354	Heterotropic Plate Count – Pour Plate Method		
350355	Technical Training and Personnel Qualifications for Biomonitoring – Aquatic Toxicity,		
	Mold, and Microbiology		
350356	Water Bath and Incubator Temperature Stability and Load Testing		
350359	Calibration and Maintenance of Autoclaves		
350362	Analytical Balance Operation and Verification in the Aquatic Toxicity Microbiology Laboratory		
1	Total Hardness Kit Operation		

# **TABLE 10.1: AQUATIC TOXICITY DEPARTMENT SOPs**

SOP #	Title/Description	
1350364	North Carolina Phase II Chronic Whole Effluent Toxicity Test Procedure for Ceriodaphnia Dubia	
350369	Sterilization, Sanitation and Residue Testing of Microbiological Glassware and Equipment	

10.2 Additional information regarding microbiological 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**

- <u>7-Day Fathead Minnow Larval Survival and Growth Test</u> 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.
- <u>3-Brood Ceriodaphnia dubia Survival and Reproduction Test</u> test organisms are fed 0.15mL of Yeast, Cereal leaves, Trout chow (YCT) and 0.15mL *Selenastrum capricornutum* algal suspension once daily.

- <u>24 and 48 Hour Acute Toxicity Tests</u> organisms are fed 2-5 hours prior to introduction into sample but are not fed for the duration of the test.
- <u>96-Hour Acute Toxicity Tests</u> organisms are fed at the 48 hour renewal period.
- <u>3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test for North Carolina</u>test organisms are fed .05mL of YCT/15mL test solution and .05 Selanastrum capricornutum algal concentrate once daily (1.7x10 to the 7th power cells/mL).

# **11.3 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. In respect to FDER related samples ESC will perform acute and chronic reference toxicant tests for all in-house cultures done with each batch.

# 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 formulas 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 formulas pertaining to Chronic Toxicity tests and EPA/821/R-02/012 for formulas pertaining to Acute Toxicity tests.

TABLE 12.1 Data Reduction Formulas			
PARAMETER	FORMULA		
IC25, NOEC, LC50, AEC	Toxcalc 5.0 Software		

 TABLE 12.1
 Data Reduction Formulas

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.

# **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 reason for the nonconformance will be 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 will be kept on file by the QA 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 will take precedence.

- 13.2.2 Out of control acute toxicity tests.
- <u>Rejection Criteria</u> –More than 10% mortality occurs in the control organisms within the specified time frame of the test.

<u>Corrective Action</u> – The test will be considered invalid and must be repeated using fresh control water.

13.2.3 Out of control 3-Brood Ceriodaphnia dubia Survival and Reproduction Test.

<u>Rejection Criteria</u> –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)

 $\underline{\text{Corrective Action}}$  – The test will be considered invalid and must be repeated using fresh control water.

13.2.4 Out of control 3-Brood Ceriodaphnia dubia Survival and Reproduction Test.

<u>Rejection Criteria</u> – If the average number of young produced in the control is less than 15 per organism

 $\underline{\text{Corrective Action}}$  – The test will be considered invalid and must be repeated using fresh control water.

13.2.5 Out of control 3-Brood Ceriodaphnia dubia Survival and Reproduction Test.

<u>Rejection Criteria</u> – A test will be considered invalid if or 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).

<u>Corrective Action</u> – The test will be considered invalid and must be repeated using fresh control water.

13.2.6 Out of control 7-Day Pimephales promelas Larval Survival and Growth Test.

<u>Rejection Criteria</u> –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.

<u>Corrective Action</u> – The test will be considered invalid and must be repeated using fresh control water.

13.2.7 Out of control 7-Day Pimephales promelas Larval Survival and Growth Test.

<u>Rejection Criteria</u> – The average weight of the control minnows is less than 0.2500 mg.

<u>Corrective Action</u> – The test will be considered invalid and must be repeated using fresh control water.

13.2.8 Out of control Monthly Reference Toxicant:

<u>Rejection Criteria</u> – 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 and median).

<u>Corrective Action</u> – 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.

<u>Rejection Criteria</u> – The PMSD value is greater than the upper value of 30.

<u>Corrective Action</u> - 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.

<u>Rejection Criteria</u> – The PMSD value is greater than the upper value of 47.

<u>Corrective Action</u> - 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.

<u>Rejection Criteria</u> – The %CV value is greater than the upper value of 40%.

<u>Corrective Action</u> - The test is deemed invalid and must be repeated.

# 14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103 *Document Control and Distribution*, SOP #030203 *Reagent Logs and Records* 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 8.0.

App. X, Ver. 10.0 Date: April 15, 2012 Page 1 of 15

**1.0** SIGNATORY APPROVALS

# Microbiology Laboratory QUALITY ASSURANCE MANUAL

# APPENDIX X 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. A signed cover page is available upon request

chnical and Regulatory Affairs, 615-773-9657 Aorgan, M.S

ric Johnson, B.S., Laboratory Director, 615-773-9654

Willia

Dixie Marlin, B.S., Qd Manager, 615-773-9681

2Nd

Kim Johnson, B.S., Microbiology Department Manager, 615-773-9687

# 2.0 APPENDIX TABLE OF CONTENTS

Section	Section Title			Rev. Date	<i>Rev.</i> #
1.0	Approval and Signature Page	Page	1	4/15/11	2
2.0	Table of Contents	Page	2	4/15/11	2
3.0	Scope and Application	Page	3	4/15/11	2
4.0	Laboratory Organization and Responsibility	Page	3	4/15/11	2
5.0	Personnel and Training	Page	3	4/15/11	2
6.0	Facilities and Laboratory Safety	Page	4	4/15/11	2
7.0	Sampling Procedures	Page	4	4/15/11	2
8.0	Equipment	Page	5	4/15/11	2
9.0	Laboratory Practices	Page	9	4/15/11	2
10.0	Analytical Procedures	Page	12	4/15/11	2
11.0	Quality Control Checks	Page	13	4/15/11	2
12.0	Data Reduction, Validation and Reporting	Page	13	4/15/11	2
13.0	Corrective Actions	Page	14	4/15/11	2
14.0	Recording Keeping	Page	15	4/15/11	2
15.0	Quality Audits	Page	15	4/15/11	2
	TABLES				
8.1	Equipment	Page	5	4/15/11	2
8.2	Equipment Preventative Maintenance, Equipment Calibration	Page	6	4/15/11	2
8.3A	Commercially Prepared Agars and Storage	Page	6	4/15/11	2
8.3B	In-house Prepared Agars and Storage	Page	7	4/15/11	2
10.1	Microbiology Department SOPs	Page	12	4/15/11	2

# **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 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 8.0*.

## 5.0 PERSONNEL AND TRAINING

#### 5.1 **PERSONNEL**

Kimberly Johnson, with a B.S. degree in Biological Sciences, is the Department Manager of the Microbiology laboratory. Ms. Johnson reviews and approves all data reduction associated with Microbiological analyses. Her responsibilities include the coordination with clients regarding sample analysis for regulatory compliance, scheduling of testing and personnel, and data reduction, interpretation and validation. Ms. Johnson is also involved in biological assessments of aquatic habitats and Toxicity Identification Evaluations. Additionally, Ms. Johnson oversees the Protozoan laboratory and is also a certified mold analyst. In her absence, Shain Schmitt assumes responsibility for Microbiological and Aquatic Toxicity departmental decisions.

# 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 ERA 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 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 Siemans Elga UltraPure deionizer system. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's hazardous waste disposal company. 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 chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods, where applicable.

ESC's laboratory safety guidelines are detailed in *the ESC Chemical Hygiene and Safety 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 for bacterial analysis are collected directly into pre-sterilized highdensity 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 30 hours (except HPC which has a 6 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 6-hour hold time.

#### ESC Lab Sciences Microbiology Quality Assurance Manual Appendix X to the ESC QAM

App. X, Ver. 10.0 Date: April 15, 2012 Page 5 of 15

- 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

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Microbiological Analysis This table is subject to revision without notice					
Item	Manufacturer	Model	Location		
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	SterlieMax	Harvey	Microbiology Lab		
Stereoscope	Olympus	SZX-ILLK100	Microbiology Lab		
Water Purifier	Siemans	Elga Purelab Plus	Microbiology Lab		

# 8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

PREVENTATIVE MAINTENANCE FOR LABORATORY EQUIPMENT				
INSTRUMENT	P. M. DESCRIPTION	FREQUENCY		
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, and Water Baths	•Maintenance service	As needed - determined by twice daily temperature performance checks @ least 4 hours apart		
Water Bath	•Check thermometer vs. N.B.S.	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	Daily – Chemical Indicator Strip		
Conductivity Meter	•Calibrate and clean probe	Daily		
Conductivity Meter	•Replace or replatinize probe	Poor response not corrected by above		
Stereoscope	Clean optics and stage	Each Use		
pH Meters	•Reference junction & electrode replacement	As needed		
pH Meters	•Probe stored in 4 pH standard	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 (acceptable range of 95-105 %)	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		
Stereoscope	• Clean optics and stage; microscope alignment (annual maintenance contract)	Annually		

# 8.3 STANDARDS AND REAGENTS

All reagents and standards must meet the requirements listed in the analytical methods.

Table 8.3A: Commercially prepared agar/broth, reagent sources, and storage information. (subject to revision as needed)				
Agar Type	Source	Storage		
M-FC Broth w/ Rosolic acid	Millipore	$4 \pm 2^{\circ}C$		
mColiBlue Broth	Millipore	$4 \pm 2^{\circ}C$		
A-1 Media (broth)	Hach	$4 \pm 2^{\circ}C$		
mEndo Broth	Hach	$4 \pm 2^{\circ}C$		
Lauryl Tryptose Broth	Hach	$4 \pm 2^{\circ}C$		
Brilliant Green Lactose Broth	Hach	$4 \pm 2^{\circ}C$		
EC media w/ mug broth	Hach	$4 \pm 2^{\circ}C$		
HPC	Hach	$4 \pm 2^{\circ}C$		
Colilert reagent powder	IDEXX	Room temp		
Enterolert reagent powder	IDEXX	Room temp		
Xylose Lysisne Deoxycholate Agar (XLD)	HealthLink	$4 \pm 2^{\circ}C$		
Brilliant Green (BG) Agar	HealthLink	$4 \pm 2^{\circ}C$		
Phosphate Buffer Solution	Weber Scientific	Room temp		

All stock agar expirations are per manufacturer specification.

# Table 8.3B: In-house prepared agar/broth, reagent sources, and storage information. (subject to revision as needed)

Agar Type-Stock	Source	Stock Storage	Stock Expiration	Preparation Components Media	Prepared Storage	Prepared Expiration
Xylose Lysisne Deoxycholate Agar (XLD)	Fisher/Difco	Room Temp	As specified by Manufacturer	XLD + Water	$4 \pm 2^{\circ}C$	2 weeks
Brilliant Green (BG) Agar	Fisher/Difco	Room Temp	As specified by Manufacturer	BG + Water	$4 \pm 2^{\circ}C$	2 weeks
Plate Count Agar	Fisher/Difco	Room Temp	As specified by Manufacturer	PCA + Water	$4 \pm 2^{\circ}C$	3 months
Tryptic Soy Agar	Fisher/Difco	Room Temp	As specified by Manufacturer	TSA + Water	$4 \pm 2^{\circ}C$	3 months
Triple Sugar Iron (TSI)	Fisher/Difco	Room Temp	As specified by Manufacturer	TSI + Water	$4 \pm 2^{\circ}C$	3 months
Lysine Iron Agar (LIA)	Fisher/Difco	Room Temp	As specified by Manufacturer	LIA + Water	$4 \pm 2^{\circ}C$	3 months
Tetrathionate Broth (TTB)	Fisher/Difco	Room Temp	As specified by Manufacturer	TTB +Water + 1 drops Iodine	$4 \pm 2^{\circ}C$	24 hrs
Tryptic Soy Broth (TSB)	Fisher/Difco	Room Temp	As specified by Manufacturer	TSB + Water	$4 \pm 2^{\circ}C$	3 months
Lauryl Tryptose Broth (LTB)	Fisher/Difco	Room Temp	As specified by Manufacturer	LTB + Water	$4 \pm 2^{\circ}C$	3 months
Buffered Rinse Water	Fisher/Difco	$4 \pm 2^{\circ}C$	As specified by Manufacturer	KH <sub>2</sub> PO <sub>4</sub> + MgCl <sub>2</sub> +Water	Room temp.	1 year

#### <u>Membrane Filters and Pads</u>

Membrane filters and pads are purchased and certified to meet the following specifications:

- Filter diameter 47 mm, mean pore diameter 0.45 µm. 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-µm-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 15 s with no dry spots on the filters.
- Flow rates are at least 55 mL/min/cm2 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  $2.0 \pm 0.2$ mL Endo broth.
- Pads release less than 1mg total acidity calculated as CaCO3 when titrated to the phenolphthalein endpoint with 0.02*N* 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  $35 \pm 0.5$ °C for 24h.

# 8.4 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 daily to verify that supplies and materials have been sterilized. Records of autoclave operations shall be maintained for every cycle. Records shall 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.

#### <u>Quebecor Colony counter</u>

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 bromcresol 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 95-105% acceptable range prior to use.

#### 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 semiannually.

#### 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.

#### **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.

# **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.

# 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 350334 Sterilization, Sanitization and Residue Testing of Microbiological Glassware and Equipment.* 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 will be cleaned according to the same procedure as listed above except the first step will be 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 presterilized. 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 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 nontarget 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:

	This Table is subject to revision without notice				
SOP #	Title/Description				
350305	Fecal Coliform: Membrane Filter Technique				
350334	HPC, Method 9215 B				
350315	Fecal Coliform Determination in Biosolids: Membrane Filter Technique (SM9222D)				
350316	Total Coliform				
350325	PH Meter Maintenance and Calibration				
350326	Thermometer Operation, Maintenance and Calibration Procedure				
350328	Conductivity Meter Maintenance and Calibration				
350331	Salmonella in Sludge				
350332	Laboratory Maintenance of Bacteria Reference Cultures				
350333	QA/QC of Microbiological Equipment and Testing Materials				
350369	Sterilization, Sanitization and Residue Testing of Microbiological Glassware and Equipment				
350359	Calibration and Maintenance of Autoclaves				
350343	Colilert				
350344	m-ColiBlue				
350355	Technical Training and Personnel Qualification for Biomonitoring-Microbiology				
350356	Water bath and Incubator Temperature Stability and Load Testing				
350348	Enterolert				

### **TABLE 10.1: MICROBIOLOGICAL DEPARTMENT SOPs**

- 10.2 Additional information regarding microbiological testing can be found in:
  - Standard Methods for the Examination of Water and Wastewater, 20<sup>th</sup> Edition, Section 9000.
  - **§** Heterotrophic Plate Count, SM 9215B
  - § Fecal Coliform Direct Test (A-1 Media), SM9221E
  - **§** Standard Total Coliform Membrane Filter Procedure, SM9222B.
  - § Fecal Coliform Membrane Filter Procedure, SM9222D.
  - **§** Enzyme Substrate Test, SM 9223B.
  - § Quantitative Salmonella Procedures, SM9260D.§ Environmental Regulations and Technology, Con
  - **§** Environmental Regulations and Technology, Control of Pathogens and Vector Attraction in Sewage Sludge, Appendix F.

# **11.0 QUALITY CONTROL CHECKS**

11.1 ESC participates in microbiological proficiency testing (PTs) by analyzing samples provided by Environmental Resource Associates (ERA). Unknowns are received and analyzed according to instructions from ERA 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 analysis is 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 blank 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 or qualified.

# 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

# **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*. 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) must be completed. The reason for the nonconformance will be 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 will be kept on file by the QA 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 will take precedence.

13.2.2 Out of control plate count comparisons between analysts.

<u>Rejection Criteria</u> – Comparisons must be within  $\pm 10\%$  for monthly plate count comparisons.

<u>Corrective Action</u> – 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*.

<u>Rejection Criteria</u> – Duplicate RPDs must not exceed 20% for total and/or fecal coliform or *E.coli*.

<u>Corrective Action</u> – 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.

<u>Rejection Criteria</u> – Blank analyses performed either at the beginning or end of the analytical sequence is positive.

<u>Corrective Action</u> – The analytical sequence may be rejected and reprocessed or qualified based on the nature of the contamination.

# **14.0 RECORD KEEPING**

Record keeping is outlined in SOP #010103 Document Control and Distribution, SOP #030203 Reagent Logs and Records 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 8.0.

App. XI, Ver. 10.0 Date: April 15, 2012 Page 1 of 19

**1.0** SIGNATORY APPROVALS

# Mold Laboratory QUALITY ASSURANCE MANUAL

# APPENDIX XI 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. A signed cover page is available upon request

hnical and Regulatory Affairs, 615-773-9657 rgan

Eric Johnson, B.S., Laboratory Director, 615-773-9654

Dixie Marlin, B.S., QC Manager, 615-773-9681

eiro nano

Christabel Fernandes-Monteiro, PhD, Mold/BOD Department Manager 615-773-9683

# 2.0 APPENDIX TABLE OF CONTENTS

Section	Section Title			Rev. Date	<i>Rev.</i> #
1.0	Approval and Signature Page	Page	1	4/15/11	2
2.0	Table of Contents	Page	2	4/15/11	2
3.0	Scope and Application	Page	3	4/15/11	2
4.0	Laboratory Organization and Responsibility	Page	3	4/15/11	2
5.0	Personnel and Training	Page	3	4/15/11	2
6.0	Facilities and Laboratory Safety	Page	4	4/15/11	2
7.0	Sampling Procedures	Page	4	4/15/11	2
8.0	Equipment	Page	5	4/15/11	2
9.0	Laboratory Practices	Page	8	4/15/11	2
10.0	Analytical Procedures	Page	10	4/15/11	2
11.0	Quality Control Checks	Page	10	4/15/11	2
12.0	Data Reduction, Validation and Reporting	Page	11	4/15/11	2
13.0	Corrective Actions	Page	12	4/15/11	2
14.0	Record Keeping	Page	13	4/15/11	2
15.0	Quality Audits	Page	13	4/15/11	2
	TABLES				
8.1	Equipment	Page	5	4/15/11	2
0.0	Equipment Preventative Maintenance,		6	4/15/11	2
8.2	Equipment Calibration	Page	6		
8.3A	Commercially Prepared Agars and Storage	Page	6	4/15/11	2
8.3B	In-house Prepared Agars and Storage	Page	7	4/15/11	2
10.1	Mold Department SOPs	Page	10	4/15/11	2
12.1	Data Reduction Formulas	Page	12	4/15/11	2

# **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 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 8.0*.

## 5.0 PERSONNEL AND TRAINING

#### 5.1 **PERSONNEL**

Dr. Christabel Fernandes-Monteiro, with a Ph.D. in Applied Biology, is the Department Manager of the Mold and BOD laboratory. 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. Her responsibilities include sample analysis, protocol development and quality control. Dr. Fernandes-Monteiro oversees the review and approval processes of all data associated with the Mold and BOD laboratory. She also reviews AIHA and EPA online training modules related to the methods being performed in the Mold and BOD Laboratory. In her absence, David Cooper assumes responsibility for departmental decisions.

David Cooper, with a BS degree in Biological Sciences, is the Primary Analyst in the Mold and BOD laboratory. He is proficient in Mold analytical methods as per AIHA guidelines. David has gained analytical experience at ESC, an AIHA accredited laboratory, and obtained additional training in Mold analysis at the McCrone Research Institute. He reviews AIHA and EPA online training modules related to the methods being performed in the Mold and BOD Laboratory.

# 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 Biomonitoring-Mold*. Performance for BOD analysis 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), Round Robin analysis and daily Quality Control sample analysis. On-going acceptable capability in BOD analysis is demonstrated by acceptable participation in the WP 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 Commodore 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 Commodore 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:
  - Ø Closed-toe shoes are worn in the laboratory
  - Ø Floors and work surfaces are cleaned on a regular basis
  - Ø Emergency numbers are posted in the laboratory
  - Ø Biological safety hoods are tested and certified annually
  - Ø 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 and Safety 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

LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Mold/ BOD Analysis This table is subject to revision without notice					
Item	Manufacturer	Model	Serial #	Location	
Analytical Balance	Mettler	PL602-S	1125081657	Bacteriology Lab	
Analytical Balance	Ohaus	Adventure Pro	8029211055	Bacteriology Lab	
Autoclave	Tuttnauer	2540EK	2906170	Mold 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	El05PPXMQ	EEP3524864	Mold Lab	
Refrigerator	Whirlpool	EL7ATRRMQ07	EWR4973976	Mold Lab	
Refrigerator	Frigidaire	FRT17G4BW9	BA703306	Mold Lab	
Stereoscope	VWR Scientific	VWRS1	V168430	Mold Lab	
Incubator	Precision Scientific	FV199LRW2	WB02401046	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	
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	

App. XI, Ver. 10.0 Date: April 15, 2012 Page 7 of 19

LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Mold/ BOD Analysis This table is subject to revision without notice					
Item	Manufacturer	Model	Serial #	Location	
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	Thermo	Orion 3 star	BOD pH	BOD	
Spectrophotometer	Hach	DR 2700	1388224	BOD	

# 8.2 EQUIPMENT PREVENTIVE MAINTENANCE

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
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 atleast 4 hours apart
Water Bath	•Check thermometer vs. NIST	Once each year
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 – G. stearothermophilus
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 95-	Daily
pH meters	Reference junction & electrode replacement	As needed
pH meters	Probe stored in KCl	At all times when not in use

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
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 3 months or as needed
	Replace tubing for dispenser, diluent pump, and rinsing vessel	Annually or as needed

# 8.3 STANDARDS 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 ranges may change depending on regulatory requirements, procedural changes, or project needs.

Table 8.3A: Commercially prepared agar sources and storage information.         (subject to revision as needed)					
Agar Type	gar Type Source				
Malt Extract Agar w/chloramphenicol (MEA)	HealthLink	4 <u>+</u> 2°C			
DG18 Agar	HealthLink	$4 \pm 2^{\circ}C$			
Modified Cellulose Agar	HealthLink	4 <u>+</u> 2°C			
Potato Dextrose Agar w/chloramphenicol (PDA)	HealthLink	$4 \pm 2^{\circ}C$			
Tryptic Soy Agar w/Sheep Blood	HealthLink	$4 \pm 2^{\circ}C$			
R2A w/cycloheximide	HealthLink	4 <u>+</u> 2°C			
2 % Malt Extract	Biolog	$4 \pm 2^{\circ}C$			
Biolog Universal Agar (BUG)	Biolog	4 <u>+</u> 2°C			
BUG w/BL	Biolog	4 <u>+</u> 2°C			
Biolog Universal Anaerobic Agar (BUA)	Biolog	4 <u>+</u> 2°C			
BUA w/BL	Biolog	$4 \pm 2^{\circ}C$			
Biolog Universal Yeast Agar (BUY)	Biolog	$4 \pm 2^{\circ}C$			
TSA w/SB contact	HealthLink	$4 \pm 2^{\circ}C$			
BUG w/0.25% Maltose	Biolog	$4 \pm 2^{\circ}C$			
Malt Extract Agar w/chloramphenicol contact	HealthLink	$4 \pm 2^{\circ}C$			
Chocolate Agar	Biolog	$4 \pm 2^{\circ}C$			
Czapek Yeast Extract Agar	HealthLink	4 <u>+</u> 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)							
Agar Type-Stock	Source	Stock Storage	Stock Expiration	Preparation Components Media	Prepared Storage	Prepared Expiration	
Malt Extract Agar (MEA)	Fisher/Difco	Room Temp	As specified by Manufacturer	MEA + Water	$4 \pm 2^{\circ}C$	3 weeks	
Potato Dextrose Agar (PDA)	Fisher/Difco	Room Temp	As specified by Manufacturer	PDA + Water	$4 \pm 2^{\circ}C$	3 weeks	
Modified Saboraud's Agar (MSA)	Fisher/Difco	Room Temp	As specified by Manufacturer	M-SAB Dex + Water	4 <u>+</u> 2°C	3 weeks	
R2A	Fisher/Difco	Room Temp	As specified by Manufacturer	R2A + Water	$4 \pm 2^{\circ}C$	3 weeks	
2 % Malt Extract	Fisher/Oxoid	Room Temp	As specified by Manufacturer	Bacteriological Agar + Malt	$4 \pm 2^{\circ}C$	3 weeks	
Biolog Universal Agar (BUG)	Biolog	Room Temp	As specified by Manufacturer	BUG + Water	$4 \pm 2^{\circ}C$	3 weeks	
Biolog Universal Anaerobic Agar (BUA)	Biolog	Room Temp	As specified by Manufacturer	BUA + Water	$4 \pm 2^{\circ}C$	3 weeks	
Biolog Universal Yeast Agar (BUY)	Biolog	Room Temp	As specified by Manufacturer	BUY + Water	$4 \pm 2^{\circ}C$	3 weeks	
Biolog Universal Agar (BUG) with 0.25%	Biolog	Room Temp	As specified by Manufacturer	BUG + Water + Maltose	$4 \pm 2^{\circ}C$	3 weeks	
Anaerobic Agar (ANA)	Hi Media	Room Temp	As specified by Manufacturer	ANA + water	$4 \pm 2^{\circ}C$	3 weeks	

Table 8.3C: Standard sources, description and calibration information.         (This table is subject to revision without notice)							
Instrument Group	Standard Source	How Received	Source/Storage	Preparation from Source	Lab Stock Storage	Preparation Frequency	
BOD	Lab preparation	As dry glucose and glutamic acid	Dessicator	150mg each/L	$4 \pm 2^{\circ}C$	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	

Table 8.3D: Working Standard Calibration				
Analysis	Calibration Standard			
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  $4 \pm 2^{\circ}$ 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  $4 \pm 2^{\circ}$ C.

#### 8.4 INSTRUMENT CALIBRATION

#### <u>Autoclave</u>

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 use 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 semiannually.

#### <u>Microscope</u>

App. XI, Ver. 10.0 Date: April 15, 2012 Page 11 of 19

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 calibrated annually by an external calibration 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 pressure. 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 recalibrates 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 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, one for each probe to verify that the analytical process is performing accurately.

#### <u>pH meter</u>

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 95-105%.

#### <u>Turbidimeter</u>

With each use, calibrate 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 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 off after use is calibrated on an annual basis. The error of calibration must not exceed 2.5%.

# 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.

Prior to first use, a sterility check with non-selective growth media is performed on each batch of dilution and rinse water prepared in the laboratory and on each batch of commercially prepared water.

## 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 will be 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.

App. XI, Ver. 10.0 Date: April 15, 2012 Page 13 of 19

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 being 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 nontarget 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 pre-prepared media are evaluated for suitability using manufacturer QC data.

# **10.0** ANALYTICAL PROCEDURES

A list of laboratory SOPs associated with the Mold and BOD laboratory can be found in the following table:

This Table is subject to revision without notice					
SOP #	Title				
340303	Biochemical Oxygen Demand				
340303A	Biochemical Oxygen Demand, Automated				
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 Ecoli/Coliforms				
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				
350357	Actinomycetes Identification				
350379	Mold Lab Reference Culture Maintenance				
350367	Labconco Flaskscrubber Operation and Maintenance				
350371	Mold lab Autoclave Maintenance and Operation				
350372	Mold Lab Balance Calibration and Verification				
350373	Preparation of Culture media				

# TABLE 10.1: MOLD DEPARTMENT SOPs

# **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. The samples are received and analyzed by method according to the vendor's instructions and according to the ESC SOP.

For BOD analysis, environmental PTs are purchased from Environmental Resource Associates (ERA). 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.  $\pm 3$ 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 by the lab supervisor and is identified by each 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 and by a second qualified analyst is 5%.
- 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 Capabilility (IDOCs) are performed during new analyst training and/or prior to acceptance and use of any new 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, 1 laboratory control sample, 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.
- 11.11 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. Only one acceptable method blank is required for each batch. If all method blanks fail, data is qualified. The depletion of the method blank should be between 0.2 and + 0.2mg DO/L.
- 11.12 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
- 11.13 The CCV should not vary more than 0.2g DO/L within a run.
- 11.14 The BOD value for the LCS must be within 167.5 and 228.5.
- 11.15 The RPD for the sample duplicate must be  $\leq 5\%$ .

# 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 is followed
- Sample preparation is correct and complete
- Analytical results are correct and complete
- QC is within criteria and complete

For BOD analysis, the Quality Control Department performs the secondary review of the data package using the ESC SOP#030227, *Data Review*. The QC Reviewer verifies that the analysis has 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.)

PARAMETER	FORMULA					
Non-viable (Spore Traps) Mold	$\frac{SporeCount}{m^3} = \frac{\text{number on trace} \times 1000}{\text{Volume of air sampled in liters}}$					
Andersen Fungal Viable (Culturable) Mold Spore Andersen Bacterial Viable (Culturable) Bacteria	$\frac{CFU}{m^3} = \frac{\text{raw counts} \times 1000}{\text{Volume of air sampled in liters}}$ $P_c = N [1/N+1/N-1+1/N-2+1/N-r+1]$					
Quantitative Fungal/Bacterial	$\frac{\text{CFU}}{\text{gm}} \text{ or } \frac{\text{CFU}}{\text{Swab}} = \frac{\# \text{ of Colonies} \times \text{Dilution Factor}}{\text{Sample Amount}}$					
BOD, 5-DAY	Initial D.O. –Final D.O. –CF % Dilution Sample Calculations are performed by computer software					

#### TABLE 12.1 Mold Data Reduction Formulas

## **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, <20% 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 will conform to the provider's certified ranges for accuracy and precision.

Table 12.3: QC Targets for BOD Lab Accuracy (LCS), Precision and RLs         This table is subject to revision without notice						
Analyte	Analysis Method Matrix Accuracy Range (%)		Precision (RPD)	RL (ppb)		
Biochemical Oxygen Demand	SM5210B	W	85-115	<u>&lt;</u> 5	5000	
Biochemical Oxygen Demand - Carbonaceous	SM5210B	W	85-115	<u>&lt;</u> 5	5000	

# 13.0 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 reason for the nonconformance 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 will be kept on file by the QA 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. 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 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 will take precedence.

13.2.2 Out of Control RPD for inter- and/or intra-analyst reanalysis.

<u>Rejection Criteria</u> - RPD value of the original analysis is calculated and must be below the current control limit.

<u>Corrective Action</u> - 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.

<u>Rejection Criteria</u> – All organisms must be accurately identified.

<u>Corrective Action</u> - 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

<u>Corrective Action</u>- 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 QA 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.

<u>Corrective action</u>- nly one acceptable method blank is required for each batch. If both blanks fail, all data must be reported with a qualifier.

13.2.6 Out of Control Laboratory Control Standards (LCS)

<u>Rejection Criteria-</u> 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).

<u>Corrective Action</u>- All samples bracketed by the failed LCS must be reported with a qualifier.

13.2.7 Out of Control Duplicate Samples

<u>Rejection Criteria-</u> Lab-generated maximum RPD limit (as listed under precision in Section12)

Corrective Action- The sample and duplicate are reported with a qualifier.

#### **14.0 RECORD KEEPING**

Record keeping is outlined in SOP #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* 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 8.0.

App. XII, Ver. 10. 0 Date: April 15, 2012 Page 1 of 10

**1.0** SIGNATORY APPROVALS

# Protozoa Laboratory QUALITY ASSURANCE MANUAL

# APPENDIX XII 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. A signed cover page is available upon request

Judith R. Morgan, M.S., VP of Technical and Regulatory Affairs 615-773-9657

Eric Johnson, B.S., Laboratory Director 615-773-9654

なんしょん

Dixie Marlin, B.S, QC Manager, 615-773-9681

Kimberly Johnson, B.S., Protozoan Department Manager 615-773-9687

# 2.0 APPENDIX TABLE OF CONTENTS

Section	Section Title			Rev. Date	<i>Rev.</i> #
1.0	Approval and Signature Page	Page	1	4/15/11	2
2.0	Table of Contents	Page	2	4/15/11	2
3.0	Scope and Application	Page	3	4/15/11	2
4.0	Laboratory Organization and Responsibilities	Page	3	4/15/11	2
5.0	Personnel and Training	Page	3	4/15/11	2
6.0	Facilities and Laboratory Safety	Page	4	4/15/11	2
7.0	Sampling Procedures	Page	4	4/15/11	2
8.0	Equipment	Page	5	4/15/11	2
9.0	Laboratory Practices	Page	7	4/15/11	2
10.0	Analytical Procedures	Page	7	4/15/11	2
11.0	Quality Control Checks	Page	8	4/15/11	2
12.0	Data Reduction, Validation and Reporting	Page	9	4/15/11	2
13.0	Corrective Actions	Page	9	4/15/11	2
14.0	Recording Keeping	Page	10	4/15/11	2
15.0	Quality Audits	Page	11	4/15/11	2
	TABLES				
8.1	Equipment	Page	5	4/15/11	2
8.3A	Standards and Reagents	Page	5	4/15/11	2
8.3B	Working Standards	Page	6	4/15/11	2
10.1	Protozoan Department SOPs	Page	7	4/15/11	2

# **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 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 8.0*.

## 5.0 PERSONNEL AND TRAINING

## 5.1 **PERSONNEL**

Kasey Raley, with a B.S. degree in Biological Sciences, is the Principal Analyst for the Protozoan laboratory. Ms. Raley is proficient in performing EPA Methods 1622 and 1623. She gained analytical experience from an accredited Protozoan laboratory and obtained additional training on microscopic techniques. Also, she frequently reviews EPA online training modules related to the methods being performed. In her absence, Nacole Jinks assumes her responsibilities.

#### 5.2 TRAINING

The certified analyst trains all new analysts to 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*. 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 Siemans® 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:
  - Ø Closed-toe shoes are worn in the laboratory
  - Ø Floors and work surfaces are cleaned on a regular basis
  - Ø Emergency numbers are posted in the laboratory
  - Ø Biological safety hoods are tested and certified annually
  - Ø 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 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 is 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			
Item	Manufacturer	Model	
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 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*.

## 8.3 STANDARDS AND REAGENTS

Table 8.3A: Stock solution sources, description and related information.						
(subject to revision as needed)           Description         Vendor         Concentration         Storage Req.         Expiration						
Sodium Hydroxide (NaOH)	Fisher	Concentrated	ambient	1 year		
Hydrochloric Acid (HCl)	Fisher	Concentrated	ambient	1 year		
Laureth-12	VWR		ambient	1 year		
Tris Stock	Fisher		ambient	NA		
EDTA	Sigma	0.5 M, pH 8.0	1-10°C	1 year		
Antifoam A	Sigma Chemical		ambient	NA		
Dynabeads <sup>®</sup> GC-Combo/Crypto	Dynal		$0 \pm 8^{\circ}C$	2 years		
Direct labeling kit for det. of oocysts and cysts, Merifluor Cryptosporidium/Giardia	Meridian Diagnostics		$0 \pm 8^{\circ}C$	1 year		
Phosphate Buffered Saline (PBS) Solution, pH 7.4	Sigma Chemical		ambient	1 year		
4', 6-diamidino-2-phenylindole (DAPI) stain	Waterborne, Inc	2mg/mL	0 ± 8°C/Darkness	When positive control fails		
Purified, live <i>Cryptosporidium</i> oocysts stock suspension	WSLH		$0 \pm 8^{\circ}C$	1 month		
Purified, live <i>Giardia</i> cysts stock suspension	WSLH		$0\pm8^{\circ}C$	1 month		

Solution	Concentrations	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	-10°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		$0 \pm 8^{\circ}C$	1 week
Staining 1X wash buffer		ambient	3 months
Phosphate Buffered Saline (PBS) Solution, pH 7.4		ambient	1 week
Working DAPI stain	10mL Stock/50ml Phosphate Buffer	Ambient/Dark container	1 day

## 9.0 LABORATORY PRACTICES

## 9.1 **REAGENT GRADE WATER**

ASTM Type I grade water: Siemans® supplies reagent grade water. Reagent water is analyzed for total chlorine, heterotrophic bacteria and specific conductance on a monthly basis. Reagent water is tested for metals: Lead, Cadmium, Chromium, Copper, Nickel, and Zinc on an annual basis.

## 9.2 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 plastic ware are checked for acceptability prior to use. Glassware acceptance criteria are documented in SOP #350412, *Cryptosporidium Laboratory Equipment Cleaning*.

## 9.3 FILTER ACCEPTANCE

Each new lot of filters is checked for acceptability prior to use by performing method blanks (MB) and ongoing precision and recovery testing (OPR) on the lot.

## **10.0** ANALYTICAL PROCEDURES

10.1 A list of laboratory SOPs associated with the protozoan laboratory can be found in the following table:

SOP #	Title	
350401	Isolation & Identification of Giardia and/or Cryptosporidium 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	
350407	Microscope Analyst Verification	
350408	Biosafety Guidelines for Cryptosporidium 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	Cryptosporidium Laboratory Equipment Cleaning	
350413	Olympus BX40 Microscope Operation and Maintenance	
350414	Steamscrubber Dishwasher Operation and Maintenance	

 TABLE 10.1: PROTOZOAN DEPARTMENT SOPs

- 10.2 The following references are used for analytical procedures conducted in the laboratory:
  - EPA. Method 1623: *Cryptosporidium* and *Giarda* 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.

## 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 US Environmental Protection Agency (EPA) who issues the testing results as either a "pass" or "fail". If the laboratory fails a PT round, a follow-up test is performed in an attempt to meet the necessary requirements. If the follow-up test results in a second failure, the laboratory takes part in a re-training program offered by the EPA or another accredited laboratory.
- 11.2 An Ongoing Precision and Recovery sample (OPR) is analyzed once weekly or per 20 samples. 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: Oocyts = 22-100% and Cysts = 14-100%.
- 11.3 A Method Blank is also analyzed once weekly or per 20 samples. The Method Blank must be free of other 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 for 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.

## 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 #350401, *Isolation and Identification of Cryptosporidium and/or Giardia in Water* and SOP #350406, *Data Collection and Verification for Method* 1622/1623.

## **12.2 VALIDATION**

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 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

## **12.3 Reporting**

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, and finally by the QC Department. The final review person 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) 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 QA Department. Corrective action procedures are documented in the SOP #350409, *IPR (Initial Precision and Recovery), OPR (Ongoing Precision and Recovery) and MS (Matrix Spike sample), Spiking Procedures.* 

## **ESC Lab Sciences Protozoa Quality Assurance Manual** *Appendix XII to the ESC OAM*

App. XII, Ver. 10. 0 Date: April 15, 2012 Page 10 of 10

13.2 Required Corrective Action

13.2.1 If a spiked sample or set of samples fails to meet quality control limits

<u>Rejection Criteria</u> - Recoveries from the OPR fall beyond the approved QC limits: Oocyts = 22-100% and Cysts = 14-100%.

<u>Corrective Action</u> - 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%. 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.

<u>Rejection Criteria</u> – The Method Blank must be free of test organisms and serves as a sterility control on the analytical system.

<u>Corrective Action</u> - 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. 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  $\pm 10\%$ .

<u>Rejection Criteria</u> – 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.

## 14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* 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 8.0.

Section 2.0, Ver. 10.0 Date: April 15, 2012 Page: 1 of 1

End Of Document



THE LEADER IN ENVIRONMENTAL TESTING

# **Quality Assurance Manual**

# **TestAmerica Nashville**

2960 Foster Creighton Drive Nashville, TN 37204 Phone: 615-726-0177/ 800-765-0980 Fax: 615-726-3404

www.testamericainc.com

Effective Date of Revision 21: August 1, 2011



# Vision

TestAmerica will be the industry leader for environmental testing and data deliverables.

# **Mission Statement**

TestAmerica is committed to providing exceptional client service, highest quality legally defensible data, and the most comprehensive range of capabilities in the environmental testing industry.

## **Core Values**

**Integrity:** We adhere to the highest moral and ethical principles in all that we do.

**Client Service:** We ensure our clients' satisfaction and success.

**Performance:** We set challenging goals, hold one another accountable, and reward results.

**Technical Excellence:** We continually invest in new technologies to provide exceptional data quality and credibility.

**Teamwork:** We help each other succeed through a cooperative team effort, in an atmosphere of civility and respect.

**People:** We invest in the long term professional development and success of our employees.

**Growth:** We manage our business for profitable growth.



# **Quality Assurance Manual**

TestAmerica Nashville 2960 Foster Creighton Drive Nashville, TN 37204 Phone: 615-726-0177/ 800-765-0980 Fax: 615-726-3404

www.testamericainc.com

Effective Date of Revision 21: August 1, 2011

# **Approval Signatures**

amirez boratory Directo

7-19-11

Date

Quality Assurance Manager - Michael H. Dunn

Technical Manager - Jamey Carmichael

<u> 7 - 19- 11</u> Date

Date

**Copyright Information:** 

This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2011 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. 0001 Distributed To: QA Server, QA, 02, 03, 04, 05, 06, 07, 12

## **SECTION 2**

## TABLE OF CONTENTS

Section No.	Title	Page No.	Effective Date
1	APPROVAL SIGNATURES	1-3	8/1/2011
2	TABLE OF CONTENTS	2-1	8/1/2011
3	INTRODUCTION	3-1	8/1/2011
3.1	Introduction And Compliance References	3-1	8/1/2011
3.2	Terms And Definitions	3-2	8/1/2011
3.3	Scope / Fields Of Testing	3-2	8/1/2011
3.4	Management Of The Manual	3-2	8/1/2011
4	ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)	4-1	8/1/2011
4.1	Overview	4-1	8/1/2011
4.2	Roles And Responsibilities	4-1	8/1/2011
4.3	Deputies	4-9	8/1/2011
5	QUALITY SYSTEM (NELAC 5.4.2)	5-1	8/1/2011
5.1	Quality Policy Statement	5-1	8/1/2011
5.2	Ethics And Data Integrity	5-1	8/1/2011
5.3	Quality System Supporting Documentation	5-2	8/1/2011
5.4	QA/QC Objectives for the Measurement of Data	5-3	8/1/2011
5.5	Criteria For Quality Indicators	5-5	8/1/2011
5.6	Statistical Quality Control	5-5	8/1/2011
5.7	Quality System Metrics	5-6	8/1/2011
6	DOCUMENT CONTROL (NELAC 5.4.3)	6-1	8/1/2011
6.1	Overview	6-1	8/1/2011
6.2	Document Approval And Issue	6-2	8/1/2011
6.3	Procedures For Document Control Policy	6-2	8/1/2011
6.4	Obsolete Documents	6-3	8/1/2011
7	REVIEW OF WORK REQUEST	7-1	8/1/2011
7.1	Overview	7-1	8/1/2011
7.2	Review Sequence And Key Personnel	7-2	8/1/2011
7.3	Documentation	7-3	8/1/2011
8	SUBCONTRACTING OF TESTS (NELAC 5.4.5)	8-1	8/1/2011
8.1	Overview	8-1	8/1/2011
8.2	Qualifying And Monitoring Subcontractors	8-1	8/1/2011
8.3	Oversight And Reporting	8-3	8/1/2011
8.4	Contingency Planning	8-4	8/1/2011
9	PURCHASING SERVICES AND SUPPLIES (NELAC 5.4.6)	9-1	8/1/2011
9.1	Overview	9-1	8/1/2011
9.2	Glassware	9-1	8/1/2011
9.3	Reagents, Standards & Supplies	9-2	8/1/2011
9.4	Purchase Of Equipment/Instruments/Software	9-4	8/1/2011

Section No.	Title	Page No.	Effective Date
9.5	Services	9-4	8/1/2011
9.6	Suppliers	9-4	8/1/2011
10	SERVICE TO THE CLIENT (NELAC 5.4.7)	10-1	8/1/2011
10.1	Overview	10-1	8/1/2011
10.2	Special Services	10-1	8/1/2011
10.3	Client Communication	10-1	8/1/2011
10.4	Reporting	10-1	8/1/2011
10.5	Client Surveys	10-2	8/1/2011
11	COMPLAINTS (NELAC 5.4.8)	11-1	8/1/2011
11.1	Overview	11-1	8/1/2011
11.2	External Complaints	11-1	8/1/2011
11.3	Internal Complaints	11-2	8/1/2011
11.4	Management Review	11-2	8/1/2011
12	CONTROL OF NON-CONFORMING WORK (NELAC 5.4.9)	12-1	8/1/2011
12.1	Överview	12-1	8/1/2011
12.2	Responsibilities And Authorities	12-1	8/1/2011
12.3	Evaluation Of Significance And Actions Taken	12-2	8/1/2011
12.4	Prevention Of Nonconforming Work	12-3	8/1/2011
12.5	Method Suspension/Restriction (Stop Work Procedures)	12-3	8/1/2011
13	CORRECTIVE ACTION (NELAC 5.4.10)	13-1	8/1/2011
13.1	Overview	13-1	8/1/2011
13.2	General	13-1	8/1/2011
13.3	Closed Loop Corrective Action Process	13-2	8/1/2011
13.4	Technical Corrective Actions	13-3	8/1/2011
13.5	Basic Corrections	13-4	8/1/2011
14	PREVENTIVE ACTION (NELAC 5.4.11)	14-1	8/1/2011
14.1	Overview	14-1	8/1/2011
14.2	Management Of Change	14-2	8/1/2011
15	CONTROL OF RECORDS (NELAC 5.4.12)	15-1	8/1/2011
15.1	Overview	15-1	8/1/2011
15.2	Technical And Analytical Records	15-4	8/1/2011
15.3	Laboratory Support Activities	15-5	8/1/2011
15.4	Administrative Records	15-6	8/1/2011
15.5	Records Management, Storage And Disposal	15-6	8/1/2011
16	AUDITS (NELAC 5.4.13)	16-1	8/1/2011
16.1	Internal Audits	16-1	8/1/2011
16.2	External Audits	16-3	8/1/2011
16.3	Audit Findings	16-4	8/1/2011
17	MANAGEMENT REVIEWS (NELAC 5.4.14)	17-1	8/1/2011
17.1	Quality Assurance Report	17-1	8/1/2011
17.2	Annual Management Review	17-1	8/1/2011
17.3	Potential Integrity Related Managerial Reviews	17-2	8/1/2011
18	PERSONNEL (NELAC 5.5.2)	18-1	8/1/2011
18.1	Overview	18-1	8/1/2011

Section No.	Title	Page No.	Effective Date
18.2	Education And Experience Requirements For Technical Personnel	18-1	8/1/2011
18.3	Training	18-3	8/1/2011
18.4	Data Integrity And Ethics Training Program	18-4	8/1/2011
19	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)	19-1	8/1/2011
19.1	Overview	19-1	8/1/2011
19.2	Environment	19-1	8/1/2011
19.3	Work Areas	19-2	8/1/2011
19.4	Floor Plan	19-2	8/1/2011
19.5	Building Security	19-2	8/1/2011
20	TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)	20-1	8/1/2011
20.1	Overview	20-1	8/1/2011
20.2	Standard Operating Procedures (SOPs)	20-1	8/1/2011
20.3	Laboratory Methods Manual	20-1	8/1/2011
20.4	Selection Of Methods	20-2	8/1/2011
20.5	Laboratory Developed Methods And Non-Standard Methods	20-5	8/1/2011
20.6	Validation Of Methods	20-5	8/1/2011
20.7	Method Detection Limits (MDL) / Limits of Detection (LOD)	20-7	8/1/2011
20.8	Instrument Detection Limits (IDL)	20-9	8/1/2011
20.9	Verification Of Detection And Reporting Limits	20-9	8/1/2011
20.10	Retention Time Windows	20-10	8/1/2011
20.11	Evaluation Of Selectivity	20-11	8/1/2011
20.12	Estimation Of Uncertainty Of Measurement	20-11	8/1/2011
20.13	Control Of Data	20-12	8/1/2011
21	EQUIPMENT (AND CALIBRATIONS) (NELAC 5.5.5)	21-1	8/1/2011
21.1	Overview	21-1	8/1/2011
21.2	Preventive Maintenance	21-1	8/1/2011
21.3	Support Equipment	21-2	8/1/2011
21.4	Instrument Calibrations	21-3	8/1/2011
21.5	Tentatively Identified Compounds (TICs) – GC/MS Analysis	21-12	8/1/2011
21.6	GC/MS Tuning	21-14	8/1/2011
22	MEASUREMENT TRACEABILITY (NELAC 5.5.6)	22-1	8/1/2011
22.1	Overview	22-1	8/1/2011
22.2	NIST-Traceable Weights and Thermometers	22-1	8/1/2011
22.3	Reference Standards / Materials	22-2	8/1/2011
22.4	Documentation And Labeling Of Standards, Reagents, And Reference Materials	22-3	8/1/2011
23	SAMPLING (NELAC 5.5.7)	23-1	8/1/2011
23.1	Overview	23-1	8/1/2011
23.2	Sampling Containers	23-1	8/1/2011
23.3	Field Quality Control (QC)	23-2	8/1/2011

#### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Table of Contents Page 4 of 8

Section No.	Title	Page No.	Effective Date
23.4	Definition Of Holding Time	23-2	8/1/2011
23.5	Sampling Containers, Preservation Requirements, Holding Times	23-3	8/1/2011
23.6	Sample Aliquots / Subsampling	23-3	8/1/2011
24	HANDLING OF SAMPLES (NELAC 5.5.8)	24-1	8/1/2011
24.1	Chain of Custody (COC)	24-1	8/1/2011
24.2	Sample Receipt	24-2	8/1/2011
24.3	Sample Acceptance Policy	24-4	8/1/2011
24.4	Sample Storage	24-5	8/1/2011
24.5	Hazardous Samples And Foreign Soils	24-5	8/1/2011
24.6	Sample Shipping	24-5	8/1/2011
24.7	Sample Disposal	24-6	8/1/2011
25	ASSURING THE QUALITY OF TEST RESULTS (NELAC 5.5.9)	25-1	8/1/2011
25.1	Överview	25-1	8/1/2011
25.2	Controls	25-1	8/1/2011
25.3	Negative Controls	25-1	8/1/2011
25.4	Positive Controls	25-2	8/1/2011
25.5	Sample Matrix Controls	25-4	8/1/2011
25.6	Acceptance Criteria (Control Limits)	25-6	8/1/2011
25.7	Method Detection Limits (MDLs)	25-8	8/1/2011
25.8	Additional Procedures To Assure Quality Control	25-8	8/1/2011
26	REPORTING RESULTS (NELAC 5.5.10)	26-1	8/1/2011
26.1	Overview	26-1	8/1/2011
26.2	Test Reports	26-1	8/1/2011
26.3	Reporting Level Or Report Type	26-3	8/1/2011
26.4	Electronic Reporting And Signature Policy	26-4	8/1/2011
26.5	Supplemental Information For Test	26-5	8/1/2011
26.6	Environmental Testing Obtained From Subcontractors	26-6	8/1/2011
26.7	Client Confidentiality	26-6	8/1/2011
26.8	Format Of Reports	26-7	8/1/2011
26.9	Amendments To Test Reports	26-7	8/1/2011
26.10	Policies On Client Requests For Amendments	26-7	8/1/2011

Table No.	Title	Page	Effective Date
9-1	Laboratory Glassware Cleaning Procedures	9-6	8/1/2011
9-2	Storage of Reagents and Chemicals	9-7	8/1/2011
13-1	Example – General Corrective Action Procedures	13-10	8/1/2011
15-1	Record Index	15-1	8/1/2011
15-2	Special Record Retention Requirements	15-3	8/1/2011
16-1	Types of Internal Audits and Frequency	16-1	8/1/2011
21-1	Types of Internal Audits and Frequency	21-16	8/1/2011
21-2	Preventive Maintenance Procedures for Laboratory Equipment	21-28	8/1/2011
21-3	Periodic Calibration	21-31	8/1/2011

# LIST OF FIGURES

Figure No.	Title	Page	Effective Date
3-1	Laboratory QA/QC Policy Memorandum	3-4	8/1/2011
4-1	Corporate Organization Chart	4-10	8/1/2011
8-1	Client-Approved Subcontractor Form	8-5	8/1/2011
8-2	Subcontracted Sample Form	8-6	8/1/2011
9-1	Example – JD Edwards Vendor Add Request Form	9-8	8/1/2011
13-1	Analysis Non-Conformance Report	13-5	8/1/2011
13-2	Sample Non-conformance Report	13-6	8/1/2011
13-3	Corrective Action Report	13-8	8/1/2011
18-1	Training Summary	18-6	8/1/2011
20-1	Example - Demonstration of Capability Documentation	20-21	8/1/2011
20-2	Work Flow	20-22	8/1/2011
24-1	Example: Chain of Custody (COC)	24-7	8/1/2011
24-2	Example: Custody Seal	24-8	8/1/2011
24-3	Example: Internal Chain of Custody (COC)	24-9	8/1/2011
24-4	Sample Disposal Record	24-10	8/1/2011
24-5	Cooler Receipt Form	24-11	8/1/2011

# LIST OF APPENDICES

Appendix No.	Title	Page	Effective Date
1	(Reserved)		
2	Laboratory Organization Chart	Appendix 2-1	8/1/2011
3	Laboratory Floor Plan	Appendix 3-1	8/1/2011
4	(Reserved)		8/1/2011
5	Glossary/Acronyms	Appendix 5-1	8/1/2011
6	Laboratory Certifications, Accreditations, Validations	Appendix 6-1	8/1/2011
7	Data Qualifiers	Appendix 7-1	8/1/2011
8	Federal Appendix I - Constituents for Assessment Monitoring	Appendix 8-1	8/1/2011
9	Federal Appendix II - Constituents for Assessment Monitoring	Appendix 9-1	8/1/2011
10	Federal Appendix IX – Groundwater Monitoring List	Appendix 10-1	8/1/2011
11	Federal Target Compound and Analyte List	Appendix 11-1	8/1/2011
12	Federal Priority Pollutant List	Appendix 12-1	8/1/2011
13	Industrial Hygiene Specific Information	Appendix 13-1	8/1/2011

# **REFERENCED CORPORATE SOPs AND POLICIES**

SOP/Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Internal Auditing
CA-Q-S-005	Calibration Curves (General)
CA-Q-S-008	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-002	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-004	Controlled Purchases Policy

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 3-1 of 3-4

#### **SECTION 3**

#### INTRODUCTION (NELAC 5.1 - 5.3)

#### 3.1 INTRODUCTION AND COMPLIANCE REFERENCES

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

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Standard 17025 (2005). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 6. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

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

- EPA 600/4-88/039, Methods for the Determination of Organic Compounds in Drinking Water, EPA, Revised July 1991.
- EPA 600/R-95/131, Methods for the Determination of Organic Compounds in Drinking Water, Supplement III, EPA, August 1995.
- EPA 600/4-79-019, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA, March 1979.
- EPA SW-846, *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)*, Third Edition, September 1986, Final Update 1, July 1992; Final Update IIA, August 1993; Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IIB, June 2005, and Final Update IV, January 2008.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- USEPA Contract Laboratory Program, Statement of Work for Inorganics & Organics Analysis, Multi-Media, Multi-Concentration, SOM and ISM, current revisions.
- APHA, Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup> Edition, 19<sup>th</sup>, 20<sup>th</sup> and 21<sup>st</sup> Edition.
- U.S. Department of Energy Order 414.1B, Quality Assurance, Approved April 29, 2004.
- US Department of Energy Order 414.1 C, Quality Assurance, June 17, 2005.
- U.S. Department of Energy, Quality Systems for Analytical Services, Revision 2.4, October 28, 2008.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- U.S. Department of Defense, Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP), Version 4.0.02, May 2006.

## 3.2 TERMS AND DEFINITIONS

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

Refer to Appendix 5 for the Glossary/Acronyms.

## 3.3 SCOPE / FIELDS OF TESTING

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

The methods covered by this manual include the most frequently requested water, air, industrial waste, and soil methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in the Control Limits Manual. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by TestAmerica Nashville meet these criteria, as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory abides by the requested criteria following review and acceptance of the requirements by the Laboratory Director, the Technical Manager, and the Quality Assurance Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director, the Technical Stringent requirements.

## 3.4 MANAGEMENT OF THE MANUAL

## 3.4.1 <u>Review Process</u>

This manual is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of TestAmerica Nashville's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager reviews the changes in the normal course of business and incorporate changes into revised sections of the document. The updates are reviewed by the QA Manager, Laboratory Director, Technical Manager, relevant operational staff and Corporate Quality Assurance (if a change is made to the Corporate template) and then incorporated into the document in periodic updates. The QAM is based on a Corporate QAM

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 3-3 of 3-4

Template that is prepared and reviewed annually by the Corporate Quality Department. Necessary changes are coordinated by the Corporate Quality Department and distributed electronically to each laboratory for inclusion in the laboratory specific QA Manuals.

Policies in the QAM that require immediate attention are addressed through the use of a Laboratory QA/QC Policy Memoranda. QA/QC Policy Memoranda are published from time to time to facilitate immediate changes to QA/QC Policy. QA/QC Policy Memoranda supersede the QAM and all other SOPs (refer to Section 5.3). All policy memoranda are dated, archived and distributed by their placement into the front of the QAM between the signature page and Section 2. At a minimum, each policy memorandum is approved by the same authorized signatories as shown on the cover page of the QA Manual. The QA/QC Policy Memoranda are incorporated into the QAM during the periodic updates. Policy memorandum may also include an expiration date if appropriate. An example format can be found in Figure 3-1.

Laboratory-specific QAM changes are approved and documented through the Management of Change process. (Refer to SOP NV08-161, Management of Change System.)

## 3.4.2 <u>Control</u>

Copyright information pertaining to this QA Manual is located on the Cover Page of this manual. The procedure for control of distribution of the QA manual is incorporated by reference to laboratory SOP NV08-152: Document Control.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 3-4 of 3-4

#### Figure 3-1.

## Laboratory QA/QC Policy Memorandum #\_\_\_\_\_

Effective Date: \_\_\_\_\_ Expiration Date: When Appropriate QAM Section is Revised

Technical Manager Approval	Date	Quality Assurance Approval	Date
Laboratory Director Approval	Date		

#### 1. Purpose

## 2. Procedure

## 3. Documentation

#### 4. Attachments

## 5. <u>References/Cross References</u>

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 4-1 of 4-10

#### **SECTION 4**

## ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)

## 4.1 <u>OVERVIEW</u>

TestAmerica Nashville is a local operating unit of TestAmerica Laboratories, Inc. This Quality Assurance Manual (QAM) is applicable to the TestAmerica Nashville laboratory only.

#### TestAmerica Nashville 2960 Foster Creighton Drive Nashville, TN 37204

## EPA ID: TN00032

The organizational structure, responsibilities, and authorities of the corporate staff of TestAmerica Laboratories, Inc are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, General Managers, VP of Client and Technical Services, Corporate Director of Technical Services, Legal and Contracts Director, National Accounts Director, Ethics and Compliance Directors, Director of Quality and Client Advocacy, Corporate Quality Directors, Corporate Quality Assurance, etc.). The laboratory operational and support staff work under direction of the Laboratory Director. The organizational structure for Corporate is presented in Figure 4-1 and the laboratory's organization chart can be found in Appendix 2.

## 4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions define each role in its relationship to the Quality Assurance Program. Due to various state terminology, the use of Director and Manager should be considered equivalent. When necessary, more extensive job descriptions may be maintained by laboratory management.

## 4.2.1 Quality Assurance Program

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

## 4.2.2 <u>Laboratory Director</u>

TestAmerica Nashville's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and

reports to their respective General Manager. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical managers for the appropriate fields of testing. The name(s) of the Technical Manager is (are) included in the national database. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director. Once all corrective actions have been made, with the approval of the Quality Manager, the Laboratory Director has the responsibility to resume suspended/restricted work.
- Ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Manager, the Operations Manager(s), Human Resources, and EH&S Manager as direct reports.

## 4.2.3 Quality Assurance Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (i. e., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintains records of all ethics-related training, including the type and proof of attendance.
- Maintains, improves, and evaluates the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 13.
- Evaluation of the thoroughness and effectiveness of training.
- Monitoring standards of performance in quality control and quality assurance to ensure that systems are in place to produce the level of quality as defined in this document.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Reviews a percentage of all final data reports for internal consistency, including Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, calculations, format, holding time, sensibility and completeness of the project file contents.
- Reviews of external audit reports and data validation requests.
- Follows up with audits to ensure client QAPP requirements are met.
- Ensuring communication with and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.

## 4.2.4 <u>Technical Manager</u>

The Technical Manager reports directly to the Laboratory Director. For Ohio VAP, this position is the Technical Director. He/she is accountable for all analyses and analysts with respect to ISO 17025. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and

second- and third-generation instrumentation. Specific responsibilities include, but are not limited to:

- Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This
  activity begins with reviewing and supporting new business contracts, insuring data quality,
  analyzing internal and external non-conformances to identify root cause issues and
  implementing the resulting corrective and preventive actions, facilitating the data review
  process (training, development, and accountability at the bench), and providing technical
  and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, manual integration, and instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Works with operations managers and department supervisors to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with operations managers, department supervisors and QA Manager.

## 4.2.5 <u>Analytical Operations Manager(s)</u>

The Analytical Operations Managers manage and direct the analytical production sections of the laboratory. For Ohio VAP, this position is known as a Department Operations Manager. He/She

reports directly to the Technical Manager. He/She assists the Technical Manager in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Manager and QA Manager and in compliance with regulatory requirements.
- Ensures that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Develops, reviews, and provides comment on technical SOPs.
- Works with department supervisors to ensure departmental compliance with the quality system.

#### 4.2.6 <u>Hazardous Waste Coordinator</u>

The Hazardous Waste Coordinator reports directly to the EH&S Manager. The duties consist of:

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

## 4.2.7 <u>Department Supervisors</u>

Department supervisors report to the Operations Managers. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, oversees management of the selection, training (as documented in Section 18.3), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, Operations Managers, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/she is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

## 4.2.8 <u>Laboratory Analysts</u>

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or department supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Completion of initial demonstration of capability and maintenance of ongoing demonstration of capability.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.

- Suggest method improvements to their department supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, are incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

## 4.2.9 Environmental Health & Safety (EH&S) Manager

The EH&S Manager reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. For Ohio VAP, this is position is the Health & Safety Manager/Coordinator. The EH&S Manager is responsible to:

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

## 4.2.10 Sample Control Manager

The Sample Control Manager reports to the Client Services Manager. The responsibilities are outlined below:

- Direct the logging of incoming samples into the LIMS.
- Ensure the verification of data entry from login.

## 4.2.11 <u>Client Services Manager</u>

The Client Services Manager reports to the Laboratory Director and serves as the interface between the laboratory's analytical departments and the laboratory's clients. For Ohio VAP, this

position is the Project Management Operations Manager. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Technical training and growth of the PM team.
- Technical liaison for the PM team.
- Human resource management of the PM team.
- Responsible to ensure that clients receive the proper sampling supplies.
- Coordinating status meetings with departmental supervisors.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the department supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress/status.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

## 4.2.12 Project Managers

The Project Management team consists of Project Managers who report to the Client Services Manager. The Project Managers are responsible to:

- Monitor analytical and QA project requirements for a specified project
- Ensure that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notify laboratory personnel of incoming projects and sample delivery schedules.
- Be accountable to clients for communicating sample progress/status.
- Ensure that clients receive the proper sampling supplies.
- Coordinate subcontract work
- Inform clients of project related problems, resolving service issues, and coordinating technical issues with the laboratory staff.

- Provide assistance to clients regarding resolution of problems concerning COC.
- Monitor the status of all projects in-house to ensure timely delivery of reports.
- Perform review of projects for completeness and compliance to client needs prior to release to client.
- Generate final laboratory reports and has signature authority (where approved).

## 4.2.13 Purchasing / Facilities Maintenance Manager

The Purchasing / Facilities Maintenance Manager reports to the Laboratory Director. He/She is responsible for:

- Coordinating facility maintenance, and
- Oversight of purchasing and receiving of supplies.

## 4.2.14 Shipping Manager / Sample Archiving and Disposal

The Shipping Manager reports to the Technical Manager. He/She is responsible for ensuring the timely and correct shipment of sample containers, including proper preservatives and instructions, to clients. He/She maintains accurate records of sample container shipments. In addition, he/she:

- Supervises the organized storage and appropriate climate control of samples.
- Supervises the disposal of samples in accordance with the Waste Disposal SOP, the Hazardous Waste Contingency Plan in the Chemical Hygiene/Safety Manual, and the U. S. Department of Agriculture requirements.

## 4.3 DEPUTIES

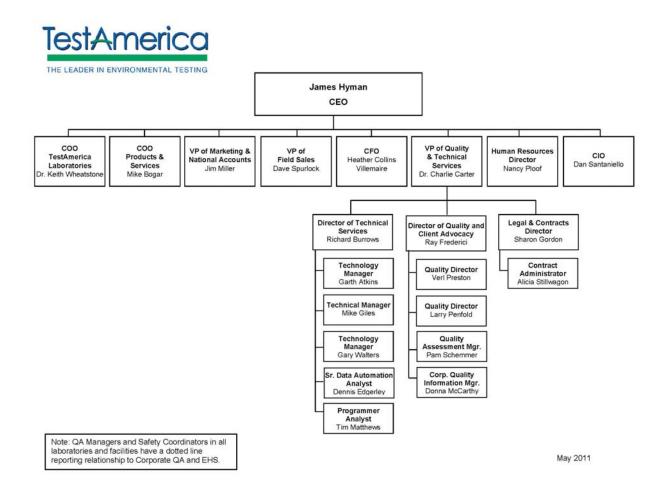
The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Primary Deputy	Support Deputy
Laboratory Director	Technical Manager	QA Manager PM Operations Manager
QA Manager	Laboratory Director	Technical Manager
Technical Manager	Laboratory Director	QA Manager Operations Managers
EHS Manager	Purchasing Manager	Laboratory Director

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 4-10 of 4-10

## Figure 4-1.

## **Corporate Organization Chart**



Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 5-1 of 5-6

#### **SECTION 5**

## QUALITY SYSTEM (NELAC 5.4.2)

#### 5.1 QUALITY POLICY STATEMENT

It is TestAmerica's Policy to:

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

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

## 5.2 ETHICS AND DATA INTEGRITY

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

- An Ethics Policy (Corporate Policy No. CA-L-P-001) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A training program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CA-L-S-001)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CA-L-S-001).
- An effective external and internal monitoring system that includes procedures for internal audits (Section 16).

- Produce results, which are accurate and include QA/QC information that meets client predefined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the ethical and quality standards of our industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

## 5.3 QUALITY SYSTEM SUPPORTING DOCUMENTATION

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

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

## 5.3.1 Order of Precedence

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

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

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 5-3 of 5-6

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

## 5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

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

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

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

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

## 5.4.1 <u>Precision</u>

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples. The calculation of precision is described in Section 25.

## 5.4.2 <u>Accuracy</u>

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery. The calculation of accuracy is described in Section 25.

## 5.4.3 <u>Representativeness</u>

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 5-4 of 5-6

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

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

# 5.4.4 <u>Comparability</u>

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

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

# 5.4.5 <u>Completeness</u>

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

# 5.4.6 <u>Selectivity</u>

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

# 5.4.7 <u>Sensitivity</u>

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

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 5-5 of 5-6

# 5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory prepares a Control Limits Manual containing tables that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Nashville. The Control Limits Manual is considered to be part of the QA Manual and therefore carries the same revision number as the QA Manual. Letter suffixes are added to the revision number if it becomes necessary to update the Control Limits Manual during the course of the year prior to a QA Manual update. This summary includes an effective date, is updated each time new limits are generated and is distributed in the same manner as the QA Manual to the analytical departments. It also is available on the laboratory server. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are not required, TestAmerica Nashville has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in Section 25.

### 5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. TestAmerica Nashville routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Manager and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. (See SOP Archiving / SA08-194.) If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

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

# 5.6.1 <u>QC Charts</u>

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 5-6 of 5-6

# 5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 17). These metrics are used to drive continuous improvement in the laboratory's Quality System.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 6-1 of 6-3

#### **SECTION 6**

# DOCUMENT CONTROL (NELAC 5.4.3)

### 6.1 <u>OVERVIEW</u>

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

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

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedures for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in the following lab SOPs: Document Control of Internal Technical Forms, Work Instructions, Information Sheets / NV08-152, Archiving of Technical and Quality Department Paper Records / NV 08-194, and Organization of Quality Department Documents / NV08-220.

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

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports. Discussion on records control is described in Section 15.

The maintenance of purchasing data is discussed in Section 9.

The maintenance of sales and marketing contracts is discussed in Section 7.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 6-2 of 6-3

## 6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a control system for each document include a unique document name and number, the number of pages of the item, the effective date, revision number and the laboratory's name. The QA department is responsible for the maintenance of the system and maintains the items in the QA Office.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a manager submits a paper or electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain the official document on file. The official document is provided as needed to those using it. Controlled documents are available at all locations where the operational activity described in the document is performed (this may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

Handwritten edits to controlled documents are not allowed, except by QA staff. Necessary changes to controlled documents must be brought to the attention of the QA department by using the appropriate notification and amendment procedures for controlled documents.

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

Quality System Policies and Procedures are reviewed at a minimum of every two years except for drinking water SOPs which are reviewed every year and revised as appropriate. Changes to documents occur when a procedural change warrants a revision of the document.

#### 6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP Document Control / NV08-152. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the QA server.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure (SOP) and SOP Document Control / NV08-152. The QA Department has a complete file of all current and previous versions, showing changes, of each SOP. Additionally, there are controlled notebooks of current SOPs in the lab. These are updated by the QA department. There is a table of contents. Electronic versions of current, previous, and in-transition SOPs are maintained on a QA hard drive that is backed up weekly. Current, unchangeable versions are stored on the QA server/SOPs.

Changes to facilities, the QA Manual, certifications, personnel, safety/health, capabilities are documented in the Management of Change log as prescribed in Section 14.

Forms, worksheets, work instructions and information are organized by department in the QA office. There is a table of contents. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP Document Control / NV08-152.

Reference books, regulations, and other external protocols are listed, with location, in the QA office. This list is updated as needed.

Logbooks and preparation worksheets are initialized and stored in an archiving system described in SOP NV08-194 for easy tracking and retrieval.

Certification correspondence, audit reports and responses, control charts, MDLs, training files, subcontractor credentials, and PT studies are stored by date in the QA office in appropriate files. These documents are not uniquely identified.

# 6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in Section 15.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 7-1 of 7-4

#### **SECTION 7**

#### **REVIEW OF WORK REQUEST**

#### 7.1 <u>OVERVIEW</u>

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

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

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

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

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

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

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

contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

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

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

# 7.2 <u>REVIEW SEQUENCE AND KEY PERSONNEL</u>

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

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements, and that the lab has the capacity to meet the clients turn around needs. Project Managers contact the appropriate laboratory management or corporate personnel if any of the details are unknown, differ from what standard payment policy and insurance coverage includes, or if the task appears to fall outside the Project Manager's job responsibilities.

For complex or large projects, the proposed contract is given to the Director of National Accounts, who decides which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in SOP No. CA-L-P-002, Contract Compliance Policy.

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

- Legal & Contracts Director
- General Manager
- The Laboratory Client Services Manager
- Laboratory and/or Corporate Technical Directors
- Regional and/or national account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Director of National Accounts, Legal & Contracts Director, or local account representative then submits the final proposal to the client.

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

The Legal & Contracts Director maintains copies of all signed contracts.

# 7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes.

The contract is distributed to and maintained by the appropriate sales/marketing personnel. A copy of the contract and formal quote is filed with the laboratory PM and the Lab Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps correspondence records (i. e., e-mail) and a phone log of conversations with the client. These records are archived by the laboratory facility for a minimum of five years.

### 7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, TestAmerica Nashville assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom requirements.

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

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

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

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers/supervisors at these

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 7-4 of 7-4

meetings. The laboratory staff is then introduced to the modified requirements via the individual laboratory operations managers/department supervisors. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

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

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 8-1 of 8-6

#### **SECTION 8**

# SUBCONTRACTING OF TESTS (NELAC 5.4.5)

# 8.1 <u>OVERVIEW</u>

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

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the SOP on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

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

Project Managers (PMs), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory advises the client of a subcontract or work sharing arrangement in writing, and approval from the client (Figure 8-1) is retained in the project folder.

**Note:** In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work.

# 8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM (or Regional Account Executive (RAE) or Customer Service Manager (CSM)) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) are selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories and supporting documentation is available on the

#### **Company Confidential & Proprietary**

TestAmerica intranet site. Verify necessary accreditation for the requested tests prior to sending samples.

• Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;

The subcontracting firm must hold the appropriate certification necessary to perform the work required. Subcontractors (including other TestAmerica facilities) selected are NELAC, A2LA, or CALA accredited laboratories whenever possible and where NELAC, A2LA, or CALA accreditation is required. Where required by the scope of work, subcontractors are accredited to ISO/IEC 17025 for accredited tests to be subcontracted unless a subcontractor accredited for the tests to be subcontracted cannot be located.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client has approved sending the samples to that laboratory. The client must provide written acknowledgement that the samples can be sent to that facility. This written acknowledgement is to be provided using the form in Figure 8-1. The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to SOP No. CA-C-S-001, Work Sharing Process.

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide written acknowledgement that the samples can be sent to that facility. This written acknowledgement is to be provided using the form in Figure 8-1.

- 8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) by lab management and forwarded to Corporate Contracts for formal contracting with the laboratory. Corporate Contracts adds the lab to the approved subcontractor list on the intranet site along with the associate documentation and notifies the finance group for JD Edwards.
- 8.2.2 The client assumes responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use.
- 8.2.3 The laboratory is responsible to the client for the subcontractor's work, except where the customer or a regulatory authority specifies which subcontractor is to be used. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.
- 8.2.4 The status and performance of qualified subcontractors are monitored periodically by the Corporate Contracts and/or Quality Department. Any problems identified are brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.
- Complaints must be investigated. Documentation of the complaint, investigation and corrective action is maintained in the subcontractor's file on the intranet site. Complaints are

posted using the Vendor Performance Report (Form No. CW-F-WI-009).

- Information is updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing are retained on the intranet listing. The lab QA Manager notifies all TestAmerica laboratories and Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification is posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers and Sales Personnel.

### 8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which reflect the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE or CSM, etc) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it is current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-2) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company's Total Assess Database.

The Shipping department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within TestAmerica.

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

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

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

**Note:** The results submitted by a TestAmerica work sharing laboratory may be transferred electronically, and the results reported by the TestAmerica work sharing lab are identified on the

final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

# 8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, the PM must verify certifications and notify the laboratory QA Manager of this event.

The comprehensive subcontractor approval process, as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures must then be initiated within 30 calendar days of subcontracting.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 8-5 of 8-6

Figure 8-1.
Client-Approved Subcontractor Form
Client Information:
Client Name & Account Number:
Client Contact:
Client Address:
Project Information: (Please choose all applicable.)
♦ Certification required: □ State □ NELAC □ A2LA □ AIHA
□ Method□ Target compound□ Other
Required Turn around time (method provisional)
Subcontractor's Information:
Subcontractor's Name:
Subcontractor's Contact:
Subcontractor's Email:
Subcontractor's Address:
Subcontractor's Phone Number:
Analytical Test/Compound/Method to be subcontracted:
□ I, the client, have designated this subcontractor for my project(s).

□ TestAmerica – Nashville has designated this subcontractor for my project(s).

#### **Certification Statement:**

I hereby give **TestAmerica Nashville** permission to use the above noted subcontractor for the above noted testing procedures/methods. I realize that the above subcontractor will be held liable for the validity of the above mentioned testing procedures/methods. All subcontractors must meet the requirements as spelled out in project information and will follow all analytical holding times and turn around times for analytical reports. The subcontract laboratory, and not TestAmerica, will be held liable for liquidated damages for delays in subcontracted analytical reports and/or electronic data deliverables.

**Client Signature** 

Date

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 8-6 of 8-6

# Figure 8-2.

# Subcontracted Sample Form

Date/Time:		
Subcontracted Laboratory Information:		
Subcontractor's Name:		
Subcontractor Point of Contact:		
Subcontractor's Address:		
Subcontractor's Phone:		
Analyte/Method:		
Certified for State of Origin:		
NELAC Accredited:	Yes	_No
• A2LA (or ISO 17025) Accredited:	Yes	_No
AIHA Accredited:	Yes	_No
CLP-like Required: (Full doc required)	Yes	_No
<ul> <li>Requested Sample Due Date: (Must be put on COC)</li> </ul>		
Project Manager:		
Laboratory Sample # Range: (Only of Subcontracted Samples)		
Laboratory Project Number (Billing Control #):		

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

PM Signature\_\_\_\_\_Date\_\_\_\_\_

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 9-1 of 9-8

#### **SECTION 9**

# PURCHASING SERVICES AND SUPPLIES (NELAC 5.4.6)

# 9.1 <u>OVERVIEW</u>

Evaluation and selection of suppliers and vendors are performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, CW-F-S-007.

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

Non-capital expenditure items are purchased through the requisition and approval process in JD Edwards or through other TestAmerica authorized methods (approved web-sites, purchasing cards). Labs have the ability to select from the approved vendors in JD Edwards.

# 9.2 <u>GLASSWARE</u>

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

# 9.2.1 Glassware Cleaning

The proper technique for cleaning glassware depends upon the intended use of the glassware being cleaned. The goal is to remove all substances from the glassware that might interfere with the analysis. Water-soluble substances can be removed with tap water followed with multiple rinses with reagent water. In some instances, detergent may be required. Detergent washings are followed by rinses with analyte-free water. Specific guidelines can be found in Table 9-1.

#### 9.2.2 Glassware Storage

Once cleaned, glassware is capped, inverted or covered for storage in a designated cabinet, away from bulk chemicals or reagents.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 9-2 of 9-8

# 9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pretested in accordance with Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

#### 9.3.1 <u>Purchasing</u>

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

The analyst provides the item number, item description, package size, and the quantity needed to his/her department supervisor. Any substitution of items requires the approval of the Technical Manager, QA Manager, or Lab Director, using a Supply Item Substitution Approval Form as described in SOP Standards Control / NV08-214. Completed Supply Item Substitution forms are filed with the Purchasing Manager. The department supervisors and the Purchasing Manager place the orders. See SOP Purchasing / NV08-232 for further details.

# 9.3.2 <u>Receiving</u>

It is the responsibility of the purchasing manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to date the material when received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified in the SOP and meets applicable specifications described below. Material Safety Data Sheets (MSDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

#### 9.3.3 <u>Specifications</u>

All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP or the SOP Standards Control / NV08-214. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and pure organic chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals must not be used past the manufacturer's or SOP's expiration date unless 'verified'.

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

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

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 10 microohm-cm at 25°C. An in-line resistivity meter check is performed and recorded daily, and the conductivity is checked and recorded weekly. If the water's conductivity is greater than the specified limit, the Technical Manager must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

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

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean, and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable) and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with a previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager or Quality Assurance Manager.

# 9.3.4 <u>Storage</u>

Reagent and chemical storage is important from the aspects of both integrity and safety. Lightsensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions. Table 9-2 details general storage instructions for reagents and chemicals. Section 22 discusses conditions for standard storage.

# 9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

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

Upon receipt of a new or used piece of equipment, it is assigned a short name (such as HP-20) added to the equipment list (described in Section 21) that is maintained by the QA Department, and IT must be notified so that it can be linked to the server for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (see Section 20). For software, its operation must be deemed reliable. Evidence of instrument verification must be retained at the instrument and in the QA Department. Software certificates supplied by the vendors are filed with the IT department. The manufacturer's operation manual is retained at the bench.

# 9.5 <u>SERVICES</u>

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventive maintenance is discussed in Section 21. The need for service is determined by analysts and/or Department Managers/Supervisors. The service providers who perform the services are approved by the Department Managers/Supervisors/Technical Manager.

#### 9.6 <u>SUPPLIERS</u>

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated expenses and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services are subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 9-5 of 9-8

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

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).

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

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

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

### 9.6.1 <u>New Vendor Procedure</u>

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (CW-F-WI-007 – refer to Figure 9-1), available on the intranet site. New vendors to be used for technical purchases must also undergo vendor evaluation as described in laboratory SOP Purchasing, NV08-232.

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

#### Table 9-1.

#### Laboratory Glassware Cleaning Procedures

Analysis/Parameter Cleaning Procedure (in specified order)	
Extractable Organics (including Pesticides and Herbicides)	13, 1, 2, 3, 4, 5 or 7, (6 or 8 optional), 14, 15
Purgeable Organics	7, 11
Trace Metals	1, 2, 3, 4, 10, 4, 9, 3, 4
Teflon™ Microwave Vessels	3, 9, 4
Nutrients (except P) and other Wet Chemistry	1, 2, 4, 9, 4
Minerals, Demand, CN and Phenols	1, 2, 4
Residues	1, 2, 4, 12
Phosphorus	9, 1, 2 (Alconox™), 4

Key to laboratory glassware cleaning procedures:

1 Remove all labels.

2	Wash with hot tap water, a brush to scrub inside glassware and stopcocks and other small pieces, if possible, using a suitable laboratory-grade detergent:
	Organics – Liquinox™, Alconox™ or equivalent,
	Inorganic Anions – Liquinox™ or equivalent,
	Inorganic Cations – Liquinox™, Acationox™, Micro or equivalent.
•	Direct the second base with the statement of

- 3 Rinse thoroughly with hot tap water.
- 4 Rinse thoroughly with deionized water.
- 5 Rinse thoroughly with pesticide-grade acetone.
- 6 Rinse thoroughly with pesticide-grade methylene chloride.
- 7 Rinse thoroughly with pesticide-grade methanol.
- 8 Rinse thoroughly with pesticide-grade hexane.
- 9 Rinse or soak with 1:1 HCI.
- 10 Rinse or soak with 10% HNO<sub>3</sub>.
- 11 Bake at 105°C for 3-4 hours (Note: Class A volumetric glassware must not be baked.)
- 12 Bake crucibles at 105°C or 180°C for 1 hour (prior to use as per method).
- 13 After use, rinse with solvent used.
- 14 Store inverted or capped with suitable material or container stopper.
- 15 Rinse with solvent used in analysis as the last step prior to use.

Table 9-2.

# Storage of Reagents and Chemicals

Chemical	Storage Requirements
Concentrated Acids and Bases	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids must not be stored with bases.
Standards for metals analysis	Stored at room temperature in the standards cabinet or a drawer near the instrument in the metals department.
Standards for extractable organics	Stored in freezers at temperatures below 0°C in the department (except DRO department standards which are stored refrigerated at <4 $\pm$ 2°C.
Standards for volatile organics	Neat standards are stored at room temperature in the standard cabinet in the department. Stock solutions and working solutions are stored in the freezer (-10 to -20°C).
Bulk Dry Chemicals	Stored at room temperature in the reagent storage area of the primary analytical department.
Working Solutions Containing Organic Compounds	Stored in the departmentas per method recommendation/ requirement. They are generally stored refrigerated at 4± 2°C.
Working Solutions Containing Only Inorganics	Stored at room temperature in the department; refrigeration is optional.
Flammable Solvents	Stored in solvent cabinets at room temperature in the organic extraction laboratory.
Non-Flammable Solvents	Stored separately from the flammable solvents in cabinets at room temperature in the organic extraction laboratory.

Figure 9-1.

Example – JD Edwards Vendor Add Request Form



THE LEADER IN ENVIRONMENTAL TESTING

# JD Edwards Vendor Add Request Form

Vendor name:	Lab location and individual making request:
Vendor address (remit to):	Vendor phone:
Vendor address (remit to):	Vendor fax:
Contact name:	Product / service provided:

#### Reason for Vendor Addition: Check all reasons that apply

Cost Reduction	Estimated Annual Savings \$
Replace Current Vendor	Reason?
	Vendor being Replaced?
New Product / Service	Describe:
ISO Approved (Required for Aerotech / P&K only)	

#### Small Business:

Does this vendor help us to meet our small business objectives:

If yes, which category:

#### Personal and Ethical Considerations:

Is there any personal conflict of interest with a TestAmerica employee and the vendor listed above?

Have ethical considerations been taken into account in your evaluation of this vendor?

#### Can this product be sourced from another TestAmerica facility?

Please complete form and email to NCPurchasing@testamericainc.com or fax to (330) 966-9275.

I approve the addition of this vendor:

Purchasing Manager - Patrick Eckman Corporate Controller - Leslie Bowers

Form No. CW-F-WI-007

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 10-1 of 10-2

#### **SECTION 10**

# SERVICE TO THE CLIENT (NELAC 5.4.7)

### 10.1 <u>OVERVIEW</u>

TestAmerica Nashville cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements discussed in Section 5. The laboratory has procedures to ensure confidentiality to clients (Section 16 and 26).

**Note:** ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request." This topic is discussed in Section 7.

### 10.2 <u>SPECIAL SERVICES</u>

The laboratory's standard procedures for reporting data are described in Section 26. Special services are also available and provided upon request. These services include:

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

#### 10.3 CLIENT COMMUNICATION

Project managers are the primary communication link to the clients. The lab informs its clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management maintains ongoing client communication throughout the entire client project.

The Technical Manager is available to discuss any technical questions or concerns that the client may have.

#### 10.4 <u>REPORTING</u>

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

# 10.5 <u>CLIENT SURVEYS</u>

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica Nashville participates in the American Council of Independent Laboratories (ACIL) Seal of Excellence program. This program includes the submission of a survey to laboratory clients. The clients send their responses directly to ACIL.

TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 11-1 of 11-3

#### **SECTION 11**

# COMPLAINTS (NELAC 5.4.8)

### 11.1 <u>OVERVIEW</u>

TestAmerica Nashville believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client concerns captures "client knowledge" that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory stands behind its data, service obligations and products.

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

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

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

The process of complaint resolution and documentation utilizes the procedures outlined in Section 13 (Corrective Actions) and is documented in the Project Information and Problem Electronic (PIPE) non-conformance database or in a Corrective Action Report (CAR).

# 11.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process and the documentation of the complaint.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints are those where the immediate issue that resulted in the complaint cannot be altered, but reoccurrences may be addressed. Non-correctable complaints are reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery

# **Company Confidential & Proprietary**

• Process Improvement

Complaints related to analytical reports are generally investigated by a Project Manager. These types of complaints may include, but are not limited to: report content and/or format, potential errors, turnaround time, bottle order shipping errors, and compliance with project requirements. The investigation may include discussions with the analyst, QA Manager, Laboratory Director, or Technical Manager, and is documented in a Customer Non-conformance Report (Figure 11-1) in the PIPE database.

Complaints related to quality systems, accreditation issues, and audit findings are investigated by the QA Manager. These complaints are documented in the CAR database.

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

# 11.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance. The procedures that must be followed regarding corrective action generation are outlined in Section 13. In addition, corporate management, Sales and Marketing, and Information Technology (IT) may initiate a complaint by contacting the laboratory management staff or through the corrective action system described in Section 13.

# 11.4 MANAGEMENT REVIEW

The number and nature of client complaints are reported by the QA Manager to the laboratory and Corporate QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions are part of the Annual Management Review (Section 17)

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 11-3 of 11-3

FrmICPAR : Form × Customer NC NC ID: 2364 E-Mail List: Γ AD Initiated By: Minn Phone # dele Client Name: Sample Range: Client Contact: SDG: Client Account NO: Analyst No: Date: Γ 7/24/2003 Supervisor: Process • Add -Action Additional bottles were sent to client. Client notes are to be revised Corrected action not chosen Corrective Preventive Action Report (CPAR) has been requested / generated. Other action taken (Please explain in summary section below). Report was amended and resubmitted Report was not amended (no change) Verbal or written clarification was provided to client via phone, email, or letter. Assigned to: closed process Action Comments: Add Cancel Record: II 🔳 1 | | | | | | + | of 1 ▶

### Figure 11-1

### **Customer Non-Conformance Report**

Process Drop-down Menu
Add compounds to the report if possible
Add Facility ID Number to Analytical Report
Alter Reporting Limits
Bottle Order did not arrive or arrived late
Bottle Order received incomplete/incorrect
Change Project Name
Change Project Number
Change Sample Collection Date/Time
Change Sample ID
Change Sampler Name
Change Test Method Reported if possible
Client did not receive email version of data
Client did not receive the fax/hardcopy data
Client Notes/Special Instructions were not followed
COC requested analysis not reported
Compound missing for QC section of Analytical Report (See below)
Correct units not used in report
Detailed case narrative required
Hardcopy data vs. EDD does not agree
Incorrect pricing applied to invoice (test:)
Other ANALYTICAL (See Comments)
Other NON-ANALYTICAL (See Comments
Remove compounds not requested from report
Report analytical values to MDL
Report limits not achieved
Unprofessional attitude by Lab Personnel
Verify analysis (outside historical bounds)
Verify sample matrix type (solid vs. liquid)

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 12-1 of 12-3

#### **SECTION 12**

# CONTROL OF NON-CONFORMING WORK (NELAC 5.4.9)

### 12.1 <u>OVERVIEW</u>

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 13).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 13. This information can then be supplied to the client in the form of a footnote or a case narrative with the report. [Note: For Ohio VAP, the laboratory must not deviate from approved SOPs or the QA Manual.]

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 20. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Manager and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason. Data being reported to a non-NELAC state would need to note the change made to how the method is normally run.

On a monthly basis, the laboratory management team reviews the non-conformance corrective actions to determine if any trends are present. If trends are found, such as repeated occurrences, further corrective action is taken to eliminate the reoccurrences as outline in Section 13.

# 12.2 RESPONSIBILITIES AND AUTHORITIES

TestAmerica's Corporate SOP entitled Internal Investigation of Potential Data Discrepancies and Determination for Data Recall (SOP No. CA-L-S-001), outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director, the Technical Manager, a Department Supervisor, or the QA Manager may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client must be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures described in Section 13. This information may also need to be documented in logbooks and/or data review checklists and in LIMS, as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior management within 24 hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Manager. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures <u>must</u> be conveyed to an Ethics and Compliance Officer (ECO), the Director of Quality & Client Advocacy, and Corporate Quality Director within 24 hours of discovery.

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

The Laboratory Director, QA Manager, Ethics and Compliance Officers, Corporate Quality, Chief Operating Officer, General Managers and the Corporate Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

# 12.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CA-L-S-001) distinguishes between situations when it would be appropriate for the laboratory QA Manager and Laboratory Director (or his/her designee) to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECOs and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting (Section 13) in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CA-L-S-001.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 12-3 of 12-3

### 12.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system (Section 13).

On a monthly basis, the Senior Management team evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process is followed.

### 12.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 12.2, Paragraph 5 above.

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

The Laboratory Director must arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting must be held to confirm that there is a problem, that suspension/restriction of the method is required and must be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases a formal group meeting may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager also initiates a corrective action report as described in Section 13 if one has not already been started. A copy of any meeting notes and agreed upon steps are faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

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

Within 72 hours, the QA Manager determines if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Manager, QA Manager, Department Supervisor) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Director of Client Services and Sales and Marketing are notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The Laboratory Director, with the approval of the QA Manager, has the responsibility to authorize the resumption of work or the elimination of any restrictions after all corrective action is complete. Approval is given by final signature on the completed corrective action report as described in Section 13.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 13-1 of 13-12

#### **SECTION 13**

# CORRECTIVE ACTION (NELAC 5.4.10)

### 13.1 <u>OVERVIEW</u>

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

## 13.2 <u>GENERAL</u>

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

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigation.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution (see more on client complaints in Section 11).

**13.2.1** <u>Non-Conformance Report (NCR)</u> - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP (See Figure 13-1 for an example Analysis Non-conformance Report.)
- QC outside of limits (non matrix related)
- Isolated reporting / calculation errors
- Client complaints (See Figure 11-1 for an example Client Non-conformance Report.)
- Sample receipt non-conformances (See Figure 13-2 for an example Sample Non-conformance Report.)

**13.2.2** <u>Corrective Action Report (CAR)</u> - is used to document (see Figure 13-3 for an example) the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCRs.
- Issues found while reviewing NCRs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.

# **Company Confidential & Proprietary**

- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors.
- Health and safety violations.

### 13.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

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

### 13.3.1 Root Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCR or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for root cause. Table 13-1 provides some general guidelines on determining responsibility for assessment.
- Root Cause Analysis is an investigative process aimed at identifying the basic or causal factor(s) that underlies variation in performance or the occurrence of a documented non-conformance event. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, an immediate response is often directed at a symptom and not the cause.
- Systematically analyze and document the Root Cause(s) behind the issue. Attempt to identify Root Cause that, when corrected, potentially will lead to dramatic improvements in performance by eliminating entire classes of problems.
- Identify and review each issue associated with the event and ask why this event occurred. Brainstorm the root cause(s) of problems by asking why events occurred or conditions existed. Look at technique, processes, or other systems outside of the normal/obvious indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation. If there are sub issues, repeat the process for all issues associated with the non-conformance event.
- The root cause analysis step is the key to the corrective action process, as a long term corrective action cannot be determined until the root cause is determined.
- If the root cause is not readily obvious, the Department Supervisor, Technical Manager, Lab Director, or QA Manager (or QA designee) is consulted.

#### 13.3.2 <u>Selection and Implementation of Corrective Actions</u>

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

# 13.3.3 Monitoring of the Corrective Actions

- The Technical Manager, Department Manager/Supervisor and QA Manager are responsible to ensure that the corrective action taken was effective.
- All Corrective action follow up is documented in the CAR database and ineffective actions are re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCR and CAR is entered into a database for tracking purposes, and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCRs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 17). If a significant trend develops that adversely affects quality, an internal audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the laboratory QA Manager indicating the nature of the out-of-control situation and problems encountered in solving the situation.

# 13.3.4 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and are performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements. (Refer to Section 16.1.4 Special Audits.)
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

# 13.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 12 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCR or CAR.

Table 13-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs.

Table 13-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set must be treated if associated QC measurements are unacceptable. Specific procedures are included in method SOPs, QAM Sections 20 and 21, and SOP CA-L-S-001 (Internal Investigation of Potential Data Discrepancies and Determination for Data Recall. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 13-4 of 13-12

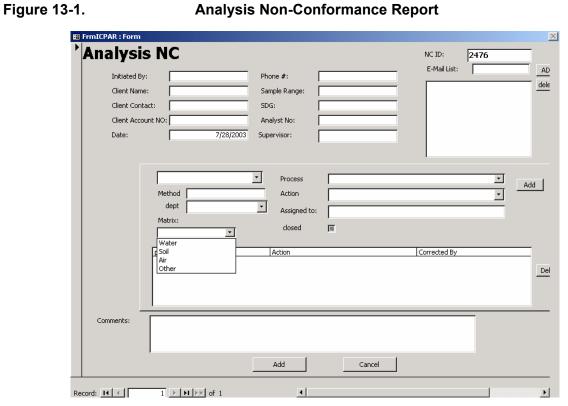
To the extent possible, samples are reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data must be reported with an appropriate data qualifier and/or the deficiency must be noted in the case narrative. Where sample results may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

# 13.5 BASIC CORRECTIONS

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

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

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



#### **Analysis Non-Conformance Report**

First Drop-down Menu	PEST	
CCV	HPLC	
Ext. Blank	QC HOLD	
Final Review Log-in	INORGANICS	
Final Review Sample	Process Drop-down Menu	
INST. Blank	(Describe in text)	
LCS	Action Drop-down Menu	
Log-in	Corrected action not chosen	
MS/MSD	Corrective and Preventive Action Report Generated	
Non Analysis	Incorrect Extraction/Extract Volumes Entered into LIMS	
Other	Metals MS/MSD out. Post-digestion Spike Analyzed.	
Sample Issues	Pesticide Breakdown Criteria Failed	
Surrogate	Re-analyze	
Dept. Drop-down Menu	Re-analyze – Out of Holding Time	
BNA	Re-analyze Insufficient Sample	
BTX	Re-analyze – NO (Comments needed in LIMS)	
DRO	Re-analyze – NO (Comments needed in LIMS)	
HALL	Re-extract – Insufficient Sample	
METALS	Re-extract – Out of Holding Time	
MISC	Re-extract	
PREP	Sample Dilutions Did Not Confirm Original Analysis	
TPH PREP	Worklist Taken Back to "Not Entered" Due to Data Entry, Unload or	
	Analysis Error	
VOA		

#### Figure 13-2.

#### Sample Non-conformance Report

nICPAR : Form			
Initiated By:	rmance/COC Revision	Form NC ID: 40568 E-Mal List:	ADD
Client Name:	Sample Range:		- delete
Client Account NO:	- SDG:	MLino	
ClientContact:	Analyst Name: 11/21/2005 Supervisor:		
NC on #	11/21/2005 Supervisor: NC Type:		
voject Name:	Project Number:		
COM Ferminal Manager	Project State:		
	Regulatory :	<u> </u>	
2	Process	- Add	
	Action	·	
	Assigned to:	*	
	Process closed		
process	Action	Corrected By	Def
Comments:			
h			
	Add	Cancel	

Record: 14 < 1 > 11 > 11 > 0f 1

Process Drop-down Menu	Action Drop-down Menu
Change Project Name	Account Number Provided in Comments
Change Project Number	Analysis cancelled per client request
Change Sample Collection Date/Time	Analytical Method Number provided in Comments
Change Sample ID	Cancelled Analysis
Change TAT to RUSH TAT per client request	Changed TAT per client request
Change TAT to Standard TAT per client request	Chromatograms have been requested
Change/Verify Method	Clarifications of special Instructions/COC provided by client
Changed Report Units – per client notes	Client Notified
Changed Report Units – per client request	Client Notified and Cancelled Analysis
Client cancelled sample analysis after analysis performed	COC sent in by client (via fax or e-mail)
Client reimbursement requested	Compounds removed as requested
COC is unclear-please clarify	Corrected action not chosen
Containers submitted-no analysis requested on COC	Data Qualifier added/removed as requested
Correct units not used in report/LIMS	Do Not Run
Dioxin/Furans List/Method?	Documentation Level: 1/2
Documentation Level?	Documentation Level: 3 (Full Doc minus Raw Data)
EDB Method?	Documentation Level: 4 (Full Doc plus Raw Data)
EnCore Prep Date Incorrectly Entered in LIMS	Documentation Level: CLP Methods
Fedex Delivery Failure	EDB Method: 504.1
HERB List?	EDB Method: 524/6210
Hold sample(s) per client/PM request	EDB Method: 601
Improper Bottle Used for Analysis	EDB Method: 624
Metals List?	EDB Method: 8011
Need List of Compounds?	EDB Method: 8021
Need MA MCP/CAM Checklist Provided	EDB Method: 8260
No Analysis Requested?	Herb List: Appendix IX
No COC-Please FAX	Herb List: Long
Oil & Grease Method?	Herb List: Other
Other NC/Process: See Comment Section Below	Herb List: Short

Out of Holding Time-List Analysis	Letter has been sent to NC per state cert. requirement
PO#	List provided by client
Provide Chromatograms per client request	MA MCP/CAM Checklist Provided by Project
	Management
Purchase Order Number Not provided	Metals List: 8 RCRA Metals
Remove compounds not requested	Metals List: CPL/TAL
Remove compounds requested on COC	Metals List: Other
Remove or add data qualifier flags	Metals List: Priority Pollutant List
Retag for additional analysis (Sample already completed)	Need List of Compounds in Comments
Sample Containers Broken in Shipment	New Analysis added per Client Request
Sample Containers Missing from Cooler-checked twice	No
Sample mislabeled	
Samples missing that are listed on the COC	
Samples submitted with Expired Holding Time	
Saturday Delivery Marked? Yes or No	Oil & Grease Method: 9070A/9070B/1664
Service Order Number not provided	Proceed with analysis and note in comment section of rpt
Special Instructions unclear-please clarify	Process Completed
Subcontract Samples?	Purchase Order Number provided in Comment Section
TCLP?	Resampling Request Form completed w/ documentation
Temperature outside Method Allowance-Run or Do Not	Run
Run?	
TPH Method?	Run Method 9071B HEM (soil)
Trip Blank not located in sample cooler	Sample held as requested
Update Demographics in Database-State, Matrix, Date,	Sample Placed on Hold
etc.	
Verify analysis/method/compound requested	Samples subcontracted to client approved subcontractor
Write letter to NC State for Nonconformance issue	Samples retagged; New SDB in Comment Section below
Wrong Preservation Used	Service Order Number provided in Comment Section
	Void NC
	Yes

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 13-8 of 13-12

## Figure 13-3.

## **Corrective Action Report**

😰 Corrective Action Report - Leslie VanExel 📃 🗐
Corrective Action Supervisor QA PM Print Exit
CAR No. <new> Status Open Client Complaint Commit Entered By Leslie VanExel Date Entered 10/28/2003 C</new>
Issue Batch/Work Order Information Supervisor Quality Assurance Project Manangement
Issue Information       C Employee       None Specified       Date of Occurrence       10/28/2003       Additional Issue Notes         © Department       Administrators       Instrument       Instrument
Issue Issue Cause
Description
Employee Oversight Internal Corrective Action
Description

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 13-9 of 13-12

CAR No. 14 Entered By Min	chael Bracken		Status Open Date Entered 10/17/2003	Client Complaint
Issue Batch/W	ork Order Inform	ation Supervisor	Quality Assurance Project	t Manangement
	Order Information K Orders Involved	I (Double Click to	Modify)	
Work Order	Sample	Matrix	Analysis	Analyte
Client Informatio	n			
Client Informatio	on Client		Project	Logged

CAR Nur	mber 🔼 Submitted By	Department	Date Entered	CAR Status	4
17	Gerardo Munoz	Pesticides	10/22/2003 2:22:02	Open	
6	Leslie VanExel	Administrators	10/08/2003 1:48:26	Open	
19	Leslie VanExel	Administrators	10/28/2003 9:54:33	Open	
16	Leslie VanExel	BTEX	10/17/2003 11:10:4	Open	
13	Leslie VanExel	Extractions	10/17/2003 10:22:0	Open	
12	Leslie VanExel	Extractions	10/17/2003 10:20:1	Open	
1	Leslie VanExel	Administrators	10/03/2003 4:01:08	Open	
11	Megan Tran	Extractions	10/17/2003 10:16:2	Open	
10	Megan Tran	GCMS-Volatiles	10/17/2003 10:04:3	Review	
9	Michael Bracken	GC-Volatiles	10/15/2003 8:42:50	PM Review	
8	Michael Bracken	GC-Volatiles	10/15/2003 8:39:29	Review	
7	Michael Bracken	GC-Volatiles	10/10/2003 12:57:5	Review	
4	Michael Bracken	GC-Volatiles	10/06/2003 3:34:15	Open	
°	Michael Prockers	GC Volstilos	10/06/2002 2/24/16	Douiou	

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 13-10 of 13-12

## Table 13-1.Example – General Corrective Action Procedures

(For Ohio VAP, see Ohio specific SOPs)

QC Activity (Individual Responsible	Acceptance Criteria	Recommended Corrective Action
for Initiation/Assessment)		Confective Action
Initial Instrument Blank (Analyst)	Instrument response < MDL.	Prepare another blank. If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards (Analyst, Department Supervisor)	Correlation coefficient $r \ge 0.995$ ( $r^2 \ge 0.99$ ) or standard concentration value. % Recovery within acceptance range. See details in method SOP.	Reanalyze standards. If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) (Analyst, Department Supervisor)	% Recovery within control limits.	Remake once and reanalyze standard. If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards	% Recovery within control limits.	Reanalyze standard once. If still unacceptable, then recalibrate and rerun affected samples.
(Analyst, Data Reviewer)		
Matrix Spike / Matrix Spike Duplicate (MS/MSD) <i>(Analyst, Data Reviewer)</i>	% Recovery within limits documented in the Control Limits Manual	If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. If the LCS is within acceptable limits, the batch is acceptable. The results of the duplicates, matrix spikes and the LCS are reported with the data set.
Laboratory Control Sample (LCS) (Analyst, Data Reviewer)	% Recovery within limits specified in the Control Limits Manual	Batch must be re-prepared and re- analyzed. If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Laboratory Control Sample Duplicate (LCSD) (Analyst, Data Reviewer)	% Recovery within limits specified in the Control Limits Manual	Batch must be re-prepared and re- analyzed. If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates in environmental samples (Analyst, Data Reviewer)	% Recovery within limits of method or within three standard deviations of the historical mean.	Individual sample must be repeated. Place comment in LIMS.

QC Activity	Acceptance Criteria	Recommended
(Individual Responsible for Initiation/Assessment)		Corrective Action
Surrogates in batch QC samples (Analyst, Data Reviewer)	Within limits of method or within three standard deviations of the historical mean.	Individual sample must be repeated. Place comment in LIMS. The batch may require repreparation and/or analysis if the QC sample calls into question the acceptability of the samples. If the surrogate % recovery exceeds the upper control limit AND any associated client samples in the batch are non-detect for target compounds, the data is acceptable to report. If the surrogate % recovery exceeds the upper control limit AND any associated client samples are greater than the RL, re-prep and re- analyze affected QC and client samples in the batch.
Method Blank (MB_ (Analyst, Data Reviewer)	< Reporting Limit <sup>1</sup>	Reanalyze blank. If still positive, determine source of contamination. If necessary, reprocess (i. e., digest or extract) entire sample batch. Report blank results.
Proficiency Testing (PT) Samples (QA Manager, Department Supervisor)	Criteria supplied by PT Supplier.	Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits (QA Manager, Department Supervisor, Operations Manager, Technical Manager, Laboratory Director)	Defined in Quality System documentation such as SOPs, QAM, etc.	Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Supervisor, QA <i>Manager</i> , Corporate QA, Corporate Management)	SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001.

#### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 13-12 of 13-12

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Client Complaints (Project Managers, Lab Director, Sales and Marketing)		Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report must result in the report being corrected and then follow- up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 17 for an example) (QA <i>Manager</i> , Lab Director, Department Supervisors/Managers)	QAM, SOPs.	Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Health & Safety Officer, Lab Director, Department Supervisor)	Environmental Health and Safety (EHS) Manual.	Non-conformance is investigated and corrected through CAR system.

#### Note:

1. Except as noted below for certain compounds, the method blank must be below the detection limit. Concentrations up to five times the reporting limit are allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction is not performed. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit. For Ohio VAP, the method blank must be below the reporting limit for all analytes that are a contaminant of concern for the project site.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 14-1 of 14-2

#### **SECTION 14**

#### PREVENTIVE ACTION (NELAC 5.4.11)

#### 14.1 <u>OVERVIEW</u>

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

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

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc.

The monthly QA Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process (Section 13) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

**14.1.1** The following elements are part of a preventive action system:

- <u>Identification</u> of an opportunity for preventive action.
- <u>Process</u> for the preventive action.
- <u>Define the measurements of the effectiveness of the process once undertaken.</u>
- <u>Execution</u> of the preventive action.
- <u>Evaluation</u> of the plan using the defined measurements.
- <u>Verification</u> of the effectiveness of the preventive action.
- <u>Close-Out</u> by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process, management reviews, and the Management of Change process (see below).

**Note:** There may be varying levels of formality and documentation during the preventive action process due to the simplicity/complexity of the action taken.

**14.1.2** Any Preventive Actions undertaken or attempted must be taken into account during the Annual Management Review (Section 17). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program provides management a measure for evaluation.

## 14.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, <u>Key</u> Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in SOP 08-161, Management of Change.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 15-1 of 15-7

#### **SECTION 15**

## CONTROL OF RECORDS (NELAC 5.4.12)

TestAmerica Nashville maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. See SOP Archiving / SA08-194 for the archiving procedures.

#### 15.1 <u>OVERVIEW</u>

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 15-1. Quality records are maintained by the Quality Assurance (QA) department in a database, which is backed up as part of the regular network backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the QA Department.

	Record Types <sup>1</sup> :	Retention Time:
Technical Records	- Raw Data - Logbooks <sup>2</sup> - Standards - Certificates - Analytical Records - Lab Reports	5 Years from analytical report issue*
Official Documents	<ul> <li>Quality Assurance Manual (QAM)</li> <li>Work Instructions</li> <li>Policies</li> <li>SOPs</li> <li>Policy Memorandums</li> <li>Manuals</li> </ul>	5 Years from document retirement date*
QA Records	<ul> <li>Internal &amp; External Audits/Respones</li> <li>Certifications</li> <li>Corrective/Preventive Actions</li> <li>Management Reviews</li> <li>Method &amp; Software Validation / Verification Data</li> <li>Data Investigation</li> </ul>	5 Years from archival* <u><b>Data Investigation:</b></u> 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)

#### Table 15-1. Record Index<sup>1</sup>

	Record Types <sup>1</sup> :	Retention Time:
Project Records	<ul> <li>Sample Receipt &amp; COC</li> <li>Documentation</li> <li>Contracts and Amendments</li> <li>Correspondence</li> <li>QAPP</li> <li>SAP</li> <li>Telephone Logbooks</li> <li>Lab Reports</li> </ul>	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits, Disposal Records	7 years
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

<sup>1</sup>Record Types encompass hardcopy and electronic records.

<sup>2</sup> Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

\* Exceptions listed in Table 15-2.

All records are legible and stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss at the Security Archives data storage facility. All records are protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration. Records are maintained on-site at the laboratory for at least one year after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention is calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 15-2 have lengthier retention requirements and are subject to the requirements in Section 15.1.3.

#### 15.1.1 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 15-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to whom to contact for authorization prior to destroying the data.

Program	<sup>1</sup> Retention Requirement
Drinking Water – All States	10 years (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
OH VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

## Table 15-2.Special Record Retention Requirements

<sup>1</sup>Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

**15.1.2** All records are held secure and in confidence. Records maintained at the laboratory are located in the Data Archive Room. Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Logs are maintained in each storage box to note removal and return of records.

**15.1.3** The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, see Section 20.12.1, "Computer and Electronic Data Related Requirements" for more information. Also reference SOP Electronic Lab Data Archiving / NV08-171.

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

• The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored with the project folder. An electronic copy is stored on the server. The chain of custody indicates the name of the

## **Company Confidential & Proprietary**

sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented. The LIMS maintains an audit trail of data verification steps.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e. g., set format for naming electronic files, set format for what is included with a given analytical data set). See SOP Electronic Lab Data Archiving / NV08-171. At a minimum, a data folder includes: benchsheet, prep sheets (where applicable), sample data, and associated QC data. Analytical data is stored by department and sequentially by date analyzed. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in reconstructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required. Each folder containing a batch of data is labeled, at a minimum, with the date analyzed and the benchsheet or sequence number.
- Changes to hardcopy records must follow the procedures outlined in Sections 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "Analyzed by."
- All generated data, except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage. Prior to the destruction of any of the hard copy information that was scanned, the scanning process must be verified in order to ensure that no data is lost, and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information.
- Also refer to Section 20.13.1, "Computer and Electronic Data Related Requirements."

## 15.2 TECHNICAL AND ANALYTICAL RECORDS

**15.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis must contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records must include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing of results.

**15.2.2** Observations, data and calculations are recorded at the time they are made and are identifiable to the specific task.

**15.2.3** Changes to hardcopy records must follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

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

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

## 15.3 LABORATORY SUPPORT ACTIVITIES

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

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a
  description of the specific computational steps used to translate parametric observations into
  a reportable analytical value;
- copies of final reports;

## **Company Confidential & Proprietary**

- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

## 15.3.1 <u>Sample Handling Records</u>

Sample handling and tracking is discussed in Section 24. Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

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

## 15.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. See Table 15-1.

## 15.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

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

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

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

**15.5.4** TestAmerica Nashville has a record management system (a.k.a document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis and are numbered sequentially within a given analysis. No analysis has more than one active notebook at a time, so all data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially in folders. Standards are maintained in the LIMS; no logbooks are used to record that data.

## **Company Confidential & Proprietary**

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 15-7 of 15-7

**15.5.5** Records are considered archived when moved off-site. Access to archived hardcopy information is documented with an access log and in/out records is used in archived boxes to note data that is removed and returned. Access to the data is limited to data storage facility and TestAmerica employees.

## 15.5.6 <u>Transfer of Ownership</u>

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

#### 15.5.7 <u>Records Disposal</u>

**15.5.7.1** Records are removed from the archive and destroyed after 10 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration.

**15.5.7.2** Electronic copies of records must be destroyed by erasure or physically damaging offline storage media so no records can be read.

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

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 16-1 of 16-4

#### **SECTION 16**

#### AUDITS (NELAC 5.4.13)

#### 16.1 INTERNAL AUDITS

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

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Audits, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are shown in Table 16-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Description	Performed by	Frequency
Quality Systems	QA Department or Designee	All areas of the laboratory annually
QA Technical Audits <ul> <li>Evaluate raw data</li> <li>versus final reports</li> <li>Analyst integrity</li> <li>Data authenticity</li> </ul>	QA Department or Designee	All methods within a 2-year period, with at least 15% of methods every quarter
SOP Method Compliance	Technical Manager and QA Department	<ul> <li>All SOPs within a 2-year period</li> <li>All new analysts or new analyst/methods within 3 months of IDOC</li> </ul>
Special	QA Department or Designee	Surveillance or spot checks performed as needed
Performance Testing	Analysts with QA oversight	Two successful per year for each NELAC field of testing or as dictated by regulatory requirements

#### Table 16-1. Types of Internal Audits and Frequency

## 16.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems, ISO 17025:2005, A2LA, AIHA, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 16-2 of 16-4

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

## 16.1.2 **QA Technical Audits**

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

## 16.1.3 <u>SOP Method Compliance</u>

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs must be assessed by the Technical Manager and QA department at least every two years. The work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products must be performed within 3 months of completing the documented training.

## 16.1.4 <u>Special Audits</u>

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

## 16.1.5 <u>Performance Testing</u>

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: drinking water, nonpotable water, soil and industrial hygiene.

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

• PTs generally do not have holding times associated with them. In the absence of any holding time requirement, it is recommended that the holding time begin when the PT sample is prepared according to the manufacturers instructions. Holding times apply to full volume PT samples only if the provider gives a meaningful "sampling date." If this is not provided, it is recommended that the date/time of opening of the full volume sample be considered the beginning of holding time.

- Login obtains the COC information from the documentation provided with the PTs, with review by QA or other designated staff.
- Vials are prepared as required in the instruction set provided with the samples. After preparation to full volume, the sample is spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis.
- PT samples do not undergo multiple preps, multiple runs, multiple methods (unless being used to evaluate multiple methods), multiple dilutions, unless this is what would be done to a normal client sample (e. g., if a client requests, as PT clients do, that we split VOA coeluters, then dual analysis is normal practice).
- The type, composition, concentration and frequency of quality control samples analyzed with the PT samples is the same as with routine environmental samples.
- Instructions are allowed to be included in the laboratory's SOPs for how low level samples are analyzed, including concentration of the sample or adjustment of the normality of titrant. When a PT sample falls below the range of the routine analytical method, the low-level procedure would be allowed where described in the SOP.
- No special reviews are performed by operations and QA, unless this is what would be done to a normal client sample. To the degree that special report forms or login procedures are required by the PT supplier, it is reasonable that the laboratory would apply special review procedures, as would be done for any client requesting unusual reporting or login processes.

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

## 16.2 EXTERNAL AUDITS

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

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

## 16.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 16-4 of 16-4

information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers must not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

## 16.3 <u>AUDIT FINDINGS</u>

Audit findings are documented using the corrective action process and database (refer to Section 13). The laboratory's corrective action responses for both types of audits (internal and external) are completed within a predetermined timeframe. When the corrective action has not yet been completed within that predefined response timeframe, a completion date must set and agreed to by operations management and the QA Manager.

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

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory must take timely corrective action, and must notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

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

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 17-1 of 17-2

#### **SECTION 17**

## MANAGEMENT REVIEWS (NELAC 5.4.14)

## 17.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report is prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director for review and comments. The final report is also submitted to the Technical Manager and lab management staff as well as the appropriate Corporate Quality Director. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

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

## 17.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Manager, QA Manager, Operations Managers) conducts an annual review of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It also provides a platform for defining quality goals & objectives.

This management systems review (Corporate SOP CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review keeps the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports (summarizing items, such as SOPs, CARs, MDLs, audits (internal and external), proficiency testing results, certifications, and training issues).
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior laboratory senior management meetings. Issues that may be raised from these meetings include:

- Adequacy of staff, equipment and facility resources.
- Adequacy of policies and procedures.
- Future plans for resources and testing capability and capacity.
- Review of the ACIL seal of excellence program performance.
- Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data integrity.

The annual review includes the previous 12 months. Based on the annual review, a report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and Corporate Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e. g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

The QA Manual is also reviewed at this time and revised to reflect any significant changes made to the quality systems.

#### 17.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The Corporate Data Investigation/ Recall SOP must be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's Chief Operating Officer, VP of Client and Technical Services, General Managers, and Corporate Quality Directors receive a monthly report compiled from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Managers are also made aware of progress on these issues for their specific labs.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 18-1 of 18-6

#### **SECTION 18**

#### PERSONNEL (NELAC 5.5.2)

#### 18.1 <u>OVERVIEW</u>

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Appendix 2.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff member that is undergoing training must have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff are qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training must be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

## 18.2 <u>EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL</u> <u>PERSONNEL</u>

The laboratory makes every effort to hire analytical staff that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Experience and specialized training are occasionally accepted in lieu of a college degree. (Basic lab skills such as using a balance, etc. are also considered). For any analysis, thorough training and working with another experienced staff member until proficiency has been demonstrated is required.

Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum

education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job descriptions are located on the TestAmerica intranet site's Human Resources web-page. (Also see Section 4 for position descriptions/responsibilities.)

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e. g., Fuels, BTEX- GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college applied sciences	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college applied sciences	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college applied sciences	And 2 years relevant experience Or 5 years of prior analytical experience
Operations Managers/Department Supervisors	Bachelor's Degree in an applied science or engineering An advanced (MS, PhD.) degree may substitute for one year of experience	And two years experience in environmental analysis of representative analytes for which they will oversee
Laboratory Director	Bachelor's Degree in science or a minor in chemistry	Or 10 years experience in the environmental laboratory industry
Quality Assurance Manager	Bachelor's degree in a basic or applied science and 1 year nonacademic analytical chemistry; Training in statistics or quality control procedures.	Or 4 years nonacademic analytical chemistry experience.
Technical Manager	Bachelor's degree in basic or applied science or engineering and at least 24 college semester credit hours in chemistry	At least two years experience in environmental analysis; master's or doctoral degree may substitute for one year experience.

Analysts-in-training perform tasks under the direct supervision of a qualified analyst or Department supervisor. The person supervising an analyst-in-training is accountable for the quality of the

## Company Confidential & Proprietary

analytical data. The trainer must review and approve the trainee's data and any associated corrective actions.

#### 18.3 <u>TRAINING</u>

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame*	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 20.

The training of technical staff is kept up to date by:

- Documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation of an annual assessment of continuing education and professional development needs/goals as part of an employee's performance evaluation.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics is maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e. g., ethics). This information is maintained in the employee's secured personnel file.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 18-4 of 18-6

• For each new method for which an analyst demonstrates capability, Figure 18-1, or an equivalent, is used to summarize that all components of the training have been completed and approved.

Further details of the laboratory's training program are described in the SOP Training / NV08-199.

#### 18.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times, TestAmerica has established an Ethics Policy No. CA-L-P-001 and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy are not tolerated. Employees who violate this policy are subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy.
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e. g., peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. investigations and data recalls.
- Consequences for infractions, including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 18-5 of 18-6

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 18-6 of 18-6

Figure	18-1.
--------	-------

#### TRAINING SUMMARY

Trainee:

\_\_\_\_\_ Date Training Began:\_\_\_\_\_

Topic(s) of Training (circle): Non-Analytical (General) Analytical Method Instrument

SOPs Reviewed:

SOPs Signed-off in QA Office: Y / N

- I. METHOD/PARAMETER
  - \_\_\_\_ Detailed Reference Method/SOP
  - Basic Method/Instrument Theory
  - Safety Precautions
  - Waste Handling
  - Instrument
  - \_\_\_\_ Routine Maintenance
  - Interferences
  - Extraction/Preparation

#### II. QUALITY CONTROL

- Calibration Curve, Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)
- Precision/Accuracy
- MDL Study
- Review of Chain-of-Custody
- Documentation (sequences, maintenance, logbooks/worksheets, observations, modifications, standards)
- QC Requirements (MS/MSD, LCS, Dups, Blanks, Surrogates, Internal Stds, Interference Checks, etc.)
- Miscellaneous QC (Retention Time Window Studies, IDL)
- Non-Conformance and Corrective Action Documentation

#### **III. DATA HANDLING AND REPORTING**

- \_\_\_\_ Review Equations and Calculations (concentrations, dry/wet weight)
- \_\_\_\_ Data Entry or Down-Loading
- Significant Figures
- Reporting Dilutions

#### **IV. GENERAL TRAINING**

Attach sheet to describe what was discussed. General Training Topics might include: Sample Receiving, Waste Disposal, Shipping, Safety.

#### **Results of Start-up QC :**

P&A Results Acceptable <u>Y / N</u>. Attach copy of Precision and Accuracy Results (Summary)

Comments (include any additional training requirements):

For AIHA methods, the training period must be a minimum of 20 business days.				
Trainee:	Date:			
Trainer:	Date:			
Group Manager:	Date:			
QA Manager:	Date:	· · · · · · · · · · · · · · · · · · ·		

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 19-1 of 19-3

#### **SECTION 19**

## ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)

#### 19.1 <u>OVERVIEW</u>

TestAmerica Nashville is a  $40,000 \text{ ft}^2$ , secure, laboratory facility with controlled access, designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment, including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

#### 19.2 <u>ENVIRONMENT</u>

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing is discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

## 19.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. For example, volatile organic chemical handling areas, including sample preparation and waste disposal, are separated from volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

#### 19.4 <u>FLOOR PLAN</u>

A floor plan can be found in Appendix 3.

#### 19.5 BUILDING SECURITY

Building keys are distributed to employees as necessary. All doors have automatic closures which lock when closed, except the two main entrances which are locked at 5:30 p.m. each business day.

Visitors to the laboratory sign in and out in a visitors' logbook. A visitor is defined as any person who visits the laboratory who is not an employee of TestAmerica Nashville. The visitors' logbook is used to ensure that everyone gets out of the building safely in case of emergency. The visitor is provided with a "Visitor" identification card to wear while in the laboratory. The Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

#### **Company Confidential & Proprietary**

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitors' logbook.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 20-1 of 20-22

#### **SECTION 20**

# TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)

#### 20.1 <u>OVERVIEW</u>

TestAmerica Nashville uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

#### 20.2 STANDARD OPERATING PROCEDURES (SOPS)

TestAmerica Nashville maintains SOPs that accurately reflect all phases of the laboratory, such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory (refer to Section 6 on Document Control):

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for SOP preparation, review, revision and control are incorporated by reference to SOPs: CW-Q-S-002 (Writing a Standard Operating Procedure (SOP)) and SOP Document Control / NV08-152.
- SOPs are reviewed at a minimum of every two years (annually for Drinking Water SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

## 20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory has available the published, referenced method as well as the laboratory developed SOP.

**Note:** If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory demonstrates that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 20-2 of 20-22

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

## 20.4 <u>SELECTION OF METHODS</u>

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information are summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e. g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology. The methods selected must be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

## 20.4.1 <u>Sources of Methods</u>

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods are used.

When clients do not specify the method to be used or methods are not required, the methods used must be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

**20.4.1.1** The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- HASL-300 28th Edition, Environmental Measurements Laboratory (EML), 1997.
- <u>Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air</u>, US EPA, January 1996.
- <u>Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act,</u> and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. <u>Revised as of July 1, 1995, Appendix</u> <u>A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)</u>
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- <u>Methods for the Determination of Inorganic Substances in Environmental Samples</u>, EPA-600/R-93/100, August 1993.
- <u>Methods for the Determination of Metals in Environmental Samples</u>, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- <u>Methods for the Determination of Organic Compounds in Drinking Water</u>, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. <u>Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series</u>) (EPA 500 Series methods)

- <u>Technical Notes on Drinking Water Methods</u>, EPA-600/R94-173, October 1994
- <u>NIOSH Manual of Analytical Methods</u>, 4<sup>th</sup> ed., August 1994.
- Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration
- <u>Standard Methods for the Examination of Water and Wastewater</u>, 18<sup>th</sup>/19<sup>th</sup> /20<sup>th</sup> edition /on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- <u>Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)</u>, Third Edition, September 1986; Final Update I, July 1992; Final Update IIA, August 1993; Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IIIB, June 2005; and Final Update IV, January 2008.
- <u>Annual Book of ASTM Standards</u>, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- <u>Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)</u>
- <u>Code of Federal Regulations (CFR) 40,</u> Parts 136, 141, 172, 173, 178, 179 and 261.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis determines the method utilized.

The laboratory must inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it must be documented.

#### 20.4.2 <u>Demonstration of Capability</u>

Before the laboratory may institute a new method and begin reporting results, the laboratory must confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

- **20.4.2.1** A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. A Demonstration of Capability is also performed when an instrument (new or used) is moved into a department and brought online for the first time in that analytical department.
- **20.4.2.2** The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the

laboratory's archiving procedures. Refer to Section 15, Control of Records and SOP Training / NV08-199.

**20.4.2.3** The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i. e., retention time window study). See SOP Method Startup / NV08-203.

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project specifications).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs (determined during Work Request Review, see Section 7) are client specified reporting limits which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 20.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).

## Note: For the purposes of Ohio VAP, the term reporting limit (RL) is used exclusively by the laboratory.

- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*
- Refer to Section 12 (Control of Non-Conforming Work).

## 20.4.3 Initial Demonstration of Capability (IDOC) Procedures

- **20.4.3.1** The spiking standard used must be prepared independently from those used in instrument calibration.
- **20.4.3.2** The analyte(s) are diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP or if unspecified to a concentration 1-4 times the laboratory RL.
- **20.4.3.3** At least four aliquots are prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days). Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

- **20.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.
- **20.4.3.5** When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory assesses performance against criteria described in the Method SOP.
- **20.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.
- **20.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:
  - Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 20.4.3.3 above.
  - Beginning with 20.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 20.4.3.1 above.

A certification statement (see Figure 20-1) is used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section must be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record replaces that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

# 20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a nonstandard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

# 20.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation is as extensive as necessary to meet

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 20-6 of 20-22

the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use. (See SOP Method Startup / NV08-203.)

### 20.6.1 <u>Method Validation and Verification Activities for All New Methods</u>

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

### 20.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

### 20.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these must be followed. The laboratory determinations of MDLs are described in Section 20.7.

# 20.6.1.3 <u>Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)</u>

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably detected. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

# Note: For the purposes of Ohio VAP, the terms MDL and reporting limit (RL) are used exclusively by the laboratory.

### 20.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

### 20.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

### 20.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

### 20.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

### 20.6.1.8 <u>Continued Demonstration of Method Performance</u>

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

# 20.7 <u>METHOD DETECTION LIMITS (MDL) / LIMITS OF DETECTION (LOD)</u>

Method detection limits (MDL) are initially determined in accordance with <u>40 CFR Part 136</u>, <u>Appendix B</u> or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 20.7.10). The analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve or ½ the lowest calibration curve standard) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates are analyzed over 2-4 days to provide a more realistic MDL.

### Note: For the purposes of Ohio VAP, the term MDL is used exclusively by the laboratory.

**20.7.1** MDLs are initially performed for each individual instrument and non-microbiological method analysis. Unless there are requirements to the contrary, the laboratory uses the highest calculated MDL for all instruments used for a given method as the MDL for reporting purposes. This MDL is not required for methods that are not readily spiked (e. g., pH, turbidity, etc.) or

# Company Confidential & Proprietary

where the lab does not report values to the MDL. For titration and gravimetric methods where there is no additional preparation involved, the MDL is based on the lowest discernable unit of measure that can be observed.

**20.7.2** MDLs must be run with acceptable instrument QC, including ICVs and Tunes. This is to insure that the instrument is in proper working condition and falsely high or low MDLs are not calculated.

**20.7.3** Use only clean matrix which is free of target analytes (e. g., Laboratory reagent water, Ottawa Sand, etc.) unless a project specific MDL is required in a field sample matrix.

**20.7.4** For Ohio VAP, the MDL spike concentration must not exceed the reporting limit.

**20.7.5** The Reporting Limit (also may be referred to as Limit of Quantitation or LOQ) is generally between 2 and 5 times the MDL. If the MDL is being performed during method development, use this guideline to determine the Reporting Limit for the analysis. If a sample is diluted or nominal prep volume/weight is vaired, the reported MDL and RL are adjusted according to the dilution factor.

**20.7.6** There are three criteria for success:

- 10 times the MDL must be the spike concentration; if not, the MDL is set at 1/2 the RL. (The RL cannot be less than the spike concentration.)
- Recovery should be between 50 and 150% of the true value (unless specified otherwise in the method SOP, and
- The ratio of the RL to the MDL should be greater than two (preferably three of more).

If the MDL must be repeated to achieve these criteria, extraction or digestion must also be repeated. A higher spike level may be necessary. The test concentration used for the MDL calculation becomes the new RL.

**20.7.7** The calculated MDL cannot be greater than the spike amount. If this occurs, repeat or increase the spike amount.

**20.7.8** If the most recently calculated MDL does not permit qualitative identification of the analyte, then the laboratory may use technical judgment for establishing the MDL (e. g., calculate what level would give a qualitative ID, compare with IDL (Section 20.8), spike at a level where qualitative ID is determined and assign that value as MDL, minimum sensitivity requirements, Standard deviation of method blanks over time, etc.). These alternate verification procedures must be documented in the laboratory SOP on MDLs (MDL / NV08-202).

**20.7.9** Each of the seven spikes must be qualitatively identifiable (e. g., appear in both columns for dual column methods, characteristic ions for GCMS mass spectra, etc.). Manual integrations to force the baseline for detection are not allowed.

**20.7.10** The initial MDL is calculated as follows:

MDL =  $t_{(n-1, 1-a = 0.99)} x$  (Standard Deviation of replicates)

where  $t_{(n-1, 1-a=0.99)} = 3.143$  for seven replicates.

# Company Confidential & Proprietary

**20.7.11** Subsequent to the initial MDL determination, periodic MDL verification, confirmation or determinations may be performed by the procedure in <u>40 CFR Part 136</u>, <u>Appendix B</u> or alternatively by other technically acceptable practices (e. g., method blanks over time, single standard spikes that have been subjected to applicable sample prep processes, etc.).

**20.7.12** Because of the inherent variability in results outside of the calibration range, TestAmerica does not recommend the reporting of results below the lowest calibration point in a curve; however, it is recognized that some projects and agencies require the reporting of results below the RL. Any result that falls between the MDL and the Reporting limit, when reported, must be qualified as an estimated value.

**20.7.13** Detections reported down to the MDL must be qualitatively identified.

**20.7.14** MDLs and reporting limits are adjusted in LIMs based on moisture content and sample aliquot size.

**20.7.15** Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP MDL/NV08-202 for details on the laboratory's MDL process.

# 20.8 INSTRUMENT DETECTION LIMITS (IDL)

**20.8.1** The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

**20.8.2** IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using seven to ten replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

**20.8.3** If IDL is > than the MDL, it may be used as the reported MDL.

# 20.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

**20.9.1** Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL for single analyte analyses (e. g., most wet chemistry methods, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e. g., GC, GCMS, ICP, etc.). The analytes must be qualitatively identified or see Sections 20.7.8 and 20.7.11 for other options. This verification does not apply to methods that are not readily spiked (e. g., pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab must not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established (see 20.7). MDLs must be verified at least annually (Quarterly for Texas).

**20.9.2** When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory must comply with any regulatory requirements.

### 20.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis, each analyte has a specific time of elution from injection on column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes.

For GC, HPLC and IC methods, there must be sufficient separation between analyte peaks so as to not misidentify analytes. In the mid-level standard, the distance between the valley and peak height cannot be any less than 25% of the sum of the peak heights of the analytes. This also applies to GCMS in the case where the two compounds share the same quantitation ion.

**Note:** Some analytes do not separate sufficiently to be able to identify or quantitate them as separate analytes (e. g., m-xylene and p-xylene) and are quantitated and reported as a single analyte (e. g., m, p-xylenes).

Once the analyst has determined that the instrument is in optimum working condition through calibration and calibration verification procedures, he or she uses a mid-range calibration or calibration verification standard to establish the retention times for each of the individual analytes in a method. The analyst makes three injections of the same standard over a 72-hour (24 hr-period for 300.0) period, tabulating the retention times for each analyte for each of the three injections. The width of retention time window is normally the average absolute retention time  $\pm$  3 Standard Deviations. A peak outside the retention time window is not identified by the computer as a positive match of the analyte of interest.

Record the retention time for each single component analyte and surrogate to three decimal places (e. g., 0.007). Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks (see the determinative methods for more details) and calculate the mean and standard deviation of those peaks.

If the standard deviation of the retention times for a target compound is between 0.000 and 0.010, then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as  $\pm 3$  times the standard deviation of the mean absolute retention time established during the 72 hour period. If the default standard deviation in (c) is employed, the width of the window is set at 0.03 minutes.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

The laboratory must calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument, except GC/MS.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 20-11 of 20-22

If the instrument data system is not capable of employing compound-specific retention time windows, then the analyst may choose a window that minimizes false negatives and positives and apply it to all compounds. As noted above, other approaches may also be employed, but must be documented by the analyst. In general, you do not use a window greater than 0.2 to 0.3 minutes. If windows larger than this have been determined, a cause must be looked for and the windows should be re-determined.

It is possible for the statistically calculated RT window to be too tight and need to be adjusted based on analyst experience. In these instances method default retention time windows may be used (e. g., for 8000 series methods a default of 0.03 minutes may be used). The same concept is applied when any peak outside of that window is not identified by the computer as a positive match.

The calibration verification standard at the beginning of a run may be used to adjust the RT for an analyte. This is essentially re-centering the window but the size of the window remains the same. The RTs are verified when all analytes are within their RT windows and are properly identified.

# 20.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

# 20.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

**20.12.1** Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

**20.12.2** Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

**20.12.3** The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 20-12 of 20-22

into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

**20.12.4** To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 + -0.5 mg/l.

**20.12.5** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e. g., 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required. Note: This section does not apply to AIHA.

**20.12.6** For the CALA accreditation program, there are a series of required steps for estimation of measurement uncertainty:

- Inventory all components of uncertainty in the test (e.g., sampling, subsampling, calibration, etc);
- Determine the significance of each component, eliminating any component that is insignificant;
- Identifying all available data that can be used in the uncertainty estimate and identifying the component that it applies to (e.g., duplicate data, spike recovery data, etc.);
- Identify any gaps in data; and,
- Use the available data, and logically derived estimates where gaps exist, to calculate the expanded uncertainty.

These criteria are met when using EPA methods, Standard Methods, ASTM, etc. Specific criteria are represented by the use of LCS, MS/MSD, or Duplicates and participation in PT programs. Control charting is used to trend and update uncertainty as percent acceptance.

Where test methods generate multi-analyte data of 10 analytes or more, the lab selects 3 analytes that represent each of three levels of uncertainty of the results – small, medium, and large levels of uncertainty – when estimating the measurement uncertainty.

Where the analyte is expected to occur over a wide concentration range (more than a factor of 10), the estimation of uncertainty is done at low, medium, and high concentrations within that range.

# 20.13 <u>CONTROL OF DATA</u>

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

### 20.13.1 <u>Computer and Electronic Data Related Requirements</u>

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOP LIMS / NV09-12 and Electronic Lab Data Archiving / NV09-171. The laboratory is currently running Element Datasystem which is a third party LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Microsoft SQL Server 2005 which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

- **20.13.1.1** <u>Maintain the Database Integrity:</u> Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.
  - LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
  - Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

**Note:** "Commercial off-the-shelf software (e.g. word processing, database, and statistical programmes) in general use within the designed application range may be considered to be sufficiently validated." *From TNI Standard EL-V1M2-ISO-2009.* However, laboratory specific configurations or modifications are validated prior to use.

- In order to assure accuracy, all data entered or transferred into the LIMS data system goes through a minimum of two levels of review.
- The QA department performs random data audits to ensure the correct information has been reported.
- Analytical data file security is provided through three policies.
  - The first policy forbids unauthorized personnel from using laboratory data acquisition computers.
  - The second policy is the implementation of network passwords and login names that restrict directory access.
  - The third layer is maintained through the LIMS and includes the use of username/password combinations to gain access to the LIMS system, the fact that all data in the LIMS is associated with the user to added/reviewed the data, and the restriction of review authority of data.
- All software installations must be in accordance with any relevant copyright licensing regulations.
- All software installed on any computer within the laboratory must be approved by the Information Technology department regional support technician assigned to the laboratory. Shrink-wrapped or otherwise sealed OEM software that is directly related to instrument usage does not need approval, but the Information Technology department must be notified of the installation.
- Anti-virus software is installed on all servers and workstations. The anti-virus software is configured to check for virus signature file and program updates on a daily basis, and these updates are pushed to all servers and workstations. The anti-virus software is configured to clean any virus-infected file, if possible; otherwise, the file is deleted.

Disks and CDs brought from any outside source that are not OEM software must be scanned for viruses before being accessed.

• The clocks on data acquisition systems for instruments may only be changed by IT, a department supervisor, a manager, or a director, and the change must be documented in the maintenance log. The Element clock can only be changed by the IT department.

### Interlab LIMS Permissions Policy

- <u>PURPOSE</u> The purpose of this policy is to provide a mechanism for maintaining the integrity of information contained in each laboratory's LIMS while providing the necessary access for information sharing to staff at other laboratory facilities.
- <u>DEFINITIONS</u> Host Laboratory: The laboratory facility that 'owns' the LIMS system or 'hosts' a project/job.
- POLICIES

(a) All permissions for the laboratory's LIMS system must only be granted by a representative of that laboratory.

- If someone outside of the host lab needs permissions for Project Management or other uses, they must go through the Lab Director or his/her designated representative.
- Permissions must never be granted without the knowledge of the host laboratory.

(b) Only laboratory analytical, Senior PMs, or QA staff from the home laboratory may have edit permissions for laboratory analysis data.

(c) Any changes made in laboratory's LIMS system:

- Must be documented and traceable.
- If made by staff of an affiliate lab, written permission from the home lab to make the changes (email approval is sufficient) is required.
- No corrections may be made in another laboratory's system without their knowledge.

(d) Data qualifiers in laboratory reports must only be corrected, edited, etc. by the staff at the host laboratory.

(e) Full analytical data "View" only permissions may be granted to outside Project Management and Sales staff. Search permissions may also be granted so status may be checked.

(f) All qualifiers must be approved by the lab QA Manager before adding to standard reference tables (Static tables).

(g) Please contact Corporate QA or IT staff if you have any questions regarding implementation or interpretation of the Interlab LIMS Permissions policy.

- **20.13.1.2** <u>Ensure Information Availability:</u> Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.
  - Insured by timely backup procedures on reliable backup media, stable file server network architecture, and UPS protection
  - UPS Protection:
    - Each fileserver is protected by an appropriate power protection/backup unit. In the event of a power outage, there is approximately 15-30 minutes of up-time for the

servers prior to shutdown. This allows for proper shutdown procedures to be followed with the fileservers.

- File Server Architecture
  - All files are maintained on multiple Windows 2000 or newer servers which are secured physically in the Information Technology office. Access to these servers is limited to members of the Information Technology staff.
  - All supporting software is maintained for at least 10 years from the last raw data generated using that software. (Length of time is dependent on local regulations or client requirements (e. g., OH VAP requires 10 years).)
- System Back-up Overview and Procedures
  - Data from both servers and instrument attached PCs are backed up and purged in compliance with the corporate back-up policy.
  - A Maintenance Plan has been defined to create a daily archive of all data within the LIMS database to a backup location. This backup is initiated automatically by either the database or back-up system.
  - Backup tapes are stored in compliance with the corporate Data Backup Policy. Backup verifications are carried out in accordance with the corporate Data Backup Policy.
  - Instrument data back-ups are verified on a periodic basis by the QA department when performing electronic data audits. The audit takes place on data that has been moved to a back-up location ensuring that it has been moved.
- **20.13.1.3** <u>Maintain Confidentiality:</u> Ensure data confidentiality through physical access controls of when electronically transmitting data.
  - All servers are located in a secure area of the IT department offices. Access to the servers is limited to IT staff members and senior lab management.
  - The company website contains SSL (Secure Socket Layer) encryption for secure website sessions and data transfers.
  - The reporting portion of the LIMS system requires a project manager to enter their unique password anytime they create a report that displays a signature on it (.PDF).
  - Electronic documents such as PDF files and electronic data deliverables are made available to clients via the secure web site. The logon page for this web site contains an agreement which states that the customer agrees not to alter any electronic data made available to them. The customer must accept this agreement before they are granted access the web site.

# 20.13.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e. g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

Manual integration of peaks must be documented and reviewed, and the raw data must be flagged in accordance with Section 20.13.5 and the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction must be applied only when required by the method or per manufacturer's indication; otherwise, it is not performed. Calculations are independently verified by appropriate laboratory staff.

- **20.13.2.1** All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/<u>year</u>). It must be easily identifiable who performed which tasks if multiple people were involved.
- 20.13.2.2 In general, concentration results are reported in milligrams per liter (mg/L) or micrograms per liter (μg/L) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (μg/kg) for solids. The units "mg/L" and "mg/kg" are the same as "parts per million (ppm)". The units "μg/L" and "μg/kg" are the same as "parts per billion (ppb)." For values greater than 10,000 mg/L, results can be reported in percent, i. e., 10,000 mg/L = 1%.
  - Several environmental methods, such as color, turbidity, conductivity, use very specific, non-concentration units to report results (e. g., NTU, umhos/cm etc).
  - Occasionally, the client requests that results be reported in units which take into account the measured flow of water or air during the collection of the sample. When they provide this information, the calculations can be performed and reported.
- **20.13.2.3** The rounding rule is: round up if the digit to be discarded is larger than 5; round down if the digit to be discarded is less than 5. If the digit is exactly 5, round down if the preceding digit is even; round up if the preceding digit is odd.
- **20.13.2.4** In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be reported to three significant figures. In general, results are reported to three significant figures on the final report.
- **20.13.2.5** For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte. Manually entered results are typed into LIMS in mg/L or mg/kg, except for low-level phenolics (ug/L) and parameters without mass-specific concentration measuring units (such as, NTU, micromhos, etc.).
- **20.13.2.6** The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst then checks the data in Element against the raw data, and the instrument printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The

data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

# 20.13.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out "real time" and have enough information on them to trace the events of the applicable analysis/task (e. g., calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- The following information is required to be documented in Analytical Run Logs:
  - Method reference
  - o Instrument ID
  - Sequence of standards, blanks, and samples including: dilutions, explanation of anomalies, standard ID numbers
  - Date of analytical batch
  - Analyst initials and date (Note: Initials across the pages if sheet taped in)
  - QC recoveries and RPDs (required for single target analyte methods only)
  - Any empty space crossed out with a "Z", dated and initialed
  - Reference to ID numbers of standards used
  - Evidence of department supervisor review on a monthly basis
  - pH/residual chlorine checks (volatiles and volatiles UST)
  - Reference to Maintenance log (when maintenance required)
- The following information is required to be documented in Prep Logs and/or Prep Sheets:
  - o Method reference
  - o Balance ID
  - Sequence of standards, blanks, and samples including: dilutions, explanation of anomalies, standard ID numbers, solvent ID numbers
  - Date and time of preparation
  - Analyst initials and date
  - How QC is prepared
  - Any empty space is crossed out with a "Z", dated and initialed.
  - Evidence of department supervisor review on a monthly basis
  - pH/residual chlorine checks
- Corrections are made following the procedures outlined in Section 13.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.

- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Manager or QA Manager at the facility. The QA Department controls all worksheets following the procedures in Section 6.

### 20.13.4 <u>Review / Verification Procedures</u>

Review procedures are outlined in several SOPs (e. g., Sample Control / NV02-01, Project Management / SA02-174, Data Package Procedure / NV08-139) to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (CA-Q-S-002). The general review concepts are discussed below, more specific information can be found in the SOPs.

- **20.13.4.1** The data review process at TestAmerica Nashville starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.
- **20.13.4.2** The next level of data review occurs with the analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analysts transfer the data into the LIMS and add data qualifiers if applicable (see Appendix 7 for list of common data qualifiers). To ensure data compliance, a department supervisor or group leader (or other designee) performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. Data review checklists are used to document both analyst and second level reviews. Issues that deem further review include the following:
  - QC data are outside the specified control limits for accuracy and precision
  - Reviewed sample data does not match with reported results
  - Unusual detection limit changes are observed
  - Raw data indicating some type of contamination or poor technique
  - Inconsistent peak integration
  - Transcription errors
  - Results outside of calibration range
- **20.13.4.3** Unacceptable analytical results may require reanalysis of the samples. Corrective action for analytical non-conformances is documented in the PIPE database. Problems may also be brought to the attention of the Laboratory Director, Project

### **Company Confidential & Proprietary**

Manager, Quality Assurance Manager, Technical Manager, Customer Service Manager, or Department Supervisor for further investigation.

- **20.13.4.4** As a final (third level) review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The following are some examples of chemical relationships that are reviewed (if data is available):
  - Total Results are > Dissolved results (e.g. metals)
  - Total Solids (TS) > TDS or TSS
  - TKN <u>></u> Ammonia
  - Total Phosphorus <u>></u> Orthophosphate
  - COD <u>></u> TOC
  - Total cyanide <u>></u> Amenable Cyanide
  - TDS <u>></u> individual anions
  - Total Chromium > Hexavalent Chromium
  - $BOD \ge cBOD$
- **20.13.4.5** Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. (*Also see section 26 on Reporting Results*). The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.
- **20.13.4.6** A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 20-3.

### 20.13.5 <u>Manual Integrations</u>

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines.

**20.13.5.1** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 20-20 of 20-22

needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

- 20.13.5.2 Analysts must not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.
- **20.13.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- **20.13.5.4 All** manual integrations are documented by printing **before and after** chromatograms/spectra with a brief explanation of why the integration was performed. The chromatograms/spectra must be scaled in such a way that the reviewer can easily identify the changes. All manual integrations receive a second level review and secondary review is documented.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 20-21 of 20-22

### Figure 20-1. Example - Demonstration of Capability Documentation

Laboratory Name:			
Laboratory Address:			
Method:		Matrix:	
Date:	Analyst(s):		
Source of Analyte(s):			

Analytical Results

Analyte	Conc. (units)	Rep 1	Rep 2	Rep 3	Rep 4	Average % Recovery	%RSD

% RSD = Percent relative standard deviation = standard deviation divided by average % Recovery

#### **Certification Statement:**

We, the undersigned, certify that:

- 1. The cited test method has met Demonstration of Capability requirements.
- 2. The test method was performed by the analyst(s) identified on this certification.
- 3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on site.
- 4. The data associated with the method demonstration of capability are true, accurate, complete, and self-explanatory.
- 5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility, and the associated information is well organized and available for review.

Analyst Signature

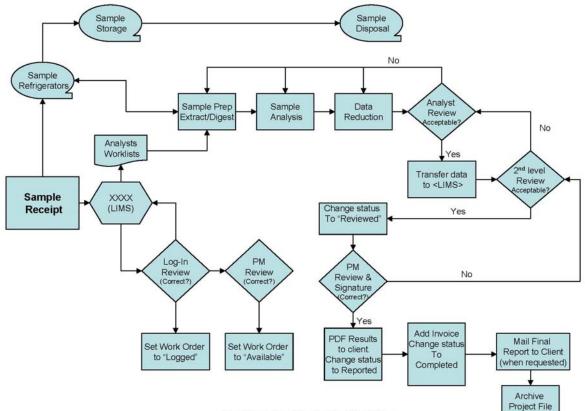
Date

Technical Manager Signature

Date

### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 20-22 of 20-22

Work Flow



TestAmerica Nashville Workflow

# Figure 20-2.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-1 of 21-33

### **SECTION 21**

# EQUIPMENT (AND CALIBRATIONS) (NELAC 5.5.5)

# 21.1 <u>OVERVIEW</u>

TestAmerica purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 21-1.

Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel.

# 21.2 PREVENTIVE MAINTENANCE

**21.2.1** TestAmerica Nashville follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

**21.2.2** Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

- **21.2.2.1** Calibrations, routine maintenance, and adjustments are part of the analysts' and Department supervisors' responsibilities. However, service contracts may be in place for some instruments to cover any major repairs.
- **21.2.2.2** High purity gases, reagents, and spare parts are kept on hand to minimize repair time and optimize instrument performance.

**21.2.3** Table 21-2 lists examples of schedules for routine maintenance. It is the responsibility of each Department supervisor to ensure that instrument maintenance logs, containing records of both preventive and corrective maintenance, are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals.

**21.2.4** Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs must be kept for all

# Company Confidential & Proprietary

major pieces of equipment. Instrument maintenance logs are also used to specify instrument parameters.

- **21.2.4.1** Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- **21.2.4.2** Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e. g., CCV run on *"date"* was acceptable, or instrument recalibrated on "date" with acceptable verification, etc.).
- **21.2.4.3** When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.
- **21.2.5** In addition, the maintenance records contain:
- The identification of the instrument/equipment (instrument's Serial Number and Model Number).
- Any maintenance procedures and frequency or a reference to their location.
- Initials of supervisor review.

**21.2.6** If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it must be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory must examine the effect of this defect on previous analyses.

**21.2.7** In the event of equipment malfunction that cannot be resolved, service must be obtained from the instrument vendor manufacturer or a qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements are made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved for the analysis, perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples are subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

### 21.3 <u>SUPPORT EQUIPMENT</u>

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens,

### Company Confidential & Proprietary

refrigerators, freezers, incubators, water baths, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. A summary of support equipment requiring calibration checks can be found in Table 21-3. All raw data records associated with the support equipment are retained to document instrument performance; these include:

- instrument model number or a unique lab identification.
- identification of standards used for the calibration check.
- performance tolerances.
- results of the calibration checks, the initials of the individual making the check, and the date of the check.

# 21.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF or CF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration must be reported with appropriate data qualifiers (refer to Section 13).

**Note:** Instruments are calibrated initially and as needed after that and at least annually.

### 21.4.1 CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP; however, the general procedures are described below.

**21.4.1.1** For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods. If a reference or mandated method does not specify the number of calibration standards, the minimum number is three, not including blanks or a zero standard. All of the standard solutions are prepared using Class A volumetric

glassware, calibrated pipettes, and/or microsyringes and appropriate laboratory quality solvents and stock standards.

- **21.4.1.2** Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials, whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard log is maintained in LIMS by each department, containing concentration, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.
- **21.4.1.3** The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).
- **21.4.1.4** The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The lowest calibration standard must be at or below the reporting limit. The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.
- **21.4.1.5** Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration then involves the analysis of each of these sets of the appropriate number of standards.
- **21.4.1.6** All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.
- **21.4.1.7** A new calibration curve must be made yearly, at minimum. All curves must document the date created.

# 21.4.2 CALIBRATION FOR ORGANIC METHODS (GC, HPLC, GC/MS)

**21.4.2.1** Many of the organic analytical methods utilize an internal standard calibration (GCMS and some GC). Because of the complex nature of the multipeak chromatograms produced by the method, some instruments necessitate the use of external standard calibration (most GC and HPLC). Surrogate compounds are included in the calibration processes for all appropriate organic analyses. However,

varying surrogate concentration is not required. For more details on the calibration types listed below, refer to SOP No. CA-Q-S-005, Calibration Curves.

- **21.4.2.2** Once the operating parameters have been established according to the method, each instrument is calibrated for the appropriate method. The analyst prepares five or more standard solutions at various concentrations containing all of the analytes of interest, internal standards, and surrogates that are appropriate for the method. Note: There are a several EPA methods that have different requirements and are exceptions where a minimum of three calibration standards are prepared and analyzed.
- **21.4.2.3** The standard solutions are introduced into the instrument in the same manner as samples are; whether it is by direct injection, by headspace analysis, or by purge and trap. The calibration factor (CF) for methods that use external standards, and the response factor (RF) for methods that use internal standards are calculated for the standards.
  - External standard calibration involves 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 standards. The ratio of the response to the amount of analyte in the calibration standard is defined as the Calibration factor (CF). Note: The calibration models in section 21.4.6.7 or 21.4.6.8 are used for Ion Chromatography (Average CF is not used.)
  - Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC and HPLC methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes vary. The internal standard solution contains

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-6 of 21-33

one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution (e. g., not all internal standards need to be at the same concentration in this solution). The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. Calculate the volume of the solution spiked into sample extracts such that minimal dilution of the extract occurs (e. g., 10 uL of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations).

An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard produces an instrument response (e. g., area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This results in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP No. CA-Q-S-005, Calibration Curves, for specific calculations.

### Calibration Factors and Response Factors for each analyte are calculated as follows:

Calibration Factor (CF) =	<u>A(s)</u>
	C(s)

<b>Response Factor</b>	(RF) =	<u>A(s) x C(is)</u>
		A(is) x C(s)

where

A(s) = Peak area (or height) of the analyte or surrogate.

A(is) = Peak area (or height) of the internal standard.

C(s) = Concentration (or mass) of the analyte or surrogate, in ug/L.

C(is) = Concentration (or mass) of the internal standard, in ug/L.

**Note:** In the equation above, RF is unit-less, i. e., the units from the two area terms and the two concentration terms cancel out. Therefore, units other than ug/L may be used for the concentrations of the analyte, surrogate, and internal standard, provided that both C(s) and C(is) are expressed in the same units. The concentration of the analyte and internal standard may also be used in calculating the RF value.

The CF or RF for each analyte at each concentration is tabulated to determine the graphical linearity of concentration versus response factor or calibration factor. The five (or more) CFs or RFs for each analyte in the initial calibration must have an acceptable Percent Relative Standard Deviation (% RSD) that is determined by each analytical method. If the RSD of the calibration or response factors is less than or equal to the acceptance limit stated in the published method over the calibration range, then linearity through the origin may be assumed, and the average calibration response factor may be used to determine sample concentrations. The CFs or RFs for each compound are calculated and kept in the calibration files.

The % Relative Standard Deviation is calculated as follows:

$$%$$
RSD = (SD /  $x_i$ ) X 100

where SD = Standard Deviation of initial 5 CFs or RFs for each compound calculated as follows:

$$SD = \sqrt{\sum_{i=1}^{n} \frac{\left(x_i - \overline{x_i}\right)^2}{n-1}}$$

where

 $x_i$  = Mean (Average) of initial 5 CFs or RFs for each compound.

n = number of standards

 $x_i$  = individual CF or RF

- **21.4.2.4** Policies regarding the use of calibration standard results for creating the calibration curve are as follows:
  - A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting limit must be elevated to be the lowest calibration standard used for calibration.
  - The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
  - Mid-calibration standards may not be excluded unless an obvious reason is found, i.e., cracked vial, incorrectly made, etc. The failed standard may be re-run once immediately (within 24 hours of the calibration run and prior to the processing of samples against the curve) and inserted into the initial calibration. If not useful, recalibration is required.

### 21.4.2.5 Percent RSD Corrective Action

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

**21.4.2.5.1** The first step is generally to check the instrument operating conditions. This option applies in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-8 of 21-33

- **21.4.2.5.2** If the RSD for any analyte is greater than the applicable acceptance criteria in the applicable analytical method SOP, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.
- **21.4.2.5.3** A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. Changes to the upper end of the calibration range affect the need to dilute samples above the range, while changes to the lower end affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

**Note:** When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

- 21.4.2.6 Alternatively, the least squares regression may be used to determine linearity. A five point line must result in a correlation coefficient (r) of 0.995 or better ( $r^2 > r^2$ 0.99) using the least squares method to be considered acceptable. In some cases it may be allowable that the curves be forced through zero (not to be confused with including the origin as an additional data point, which is not allowed). **Note:** EPA Method 8000B does not allow forcing through zero; however, the agency has revaluated this position and has since changed this stance to allow forcing through zero. In addition, from EPA Method 8000C: "However, the use of a linear regression or forcing the regression through zero may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards." OH VAP does not allow forcing through zero on any samples taken for the purpose of their program. South Carolina does not allow forcing through zero for the linear calibration curve.
- 21.4.2.7 Instead of a linear curve model (either Average RF or least squares regression), a second order curve (quadratic) may be used (and preferred) as long as it contains at least six data points. It is preferred that curve weighing be used in lieu of higher level polynomials. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (COD or r<sup>2</sup>) for the quadratic curve must be at least 0.99 for it to be considered acceptable. For more details on the calculations, see Calibration Curve SOP CA-Q-S-005. Some limitations on the use of quadratic curve fits:
- **21.4.2.7.1** Care MUST be exercised to assure that the results from this equation are real, positive, and fit the range of the initial calibration.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-9 of 21-33

- **21.4.2.7.2** They **must not** be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).
- **21.4.2.7.3** They **must not** be used to compensate for detector saturation. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1<sup>st</sup> order fit or average RF.

### **Coefficient of Determination**

### **Correlation Coefficient**



where:

y = Response or Response ratio (see below)

x = Concentration

**21.4.2.7.4** South Carolina does not allow the use of second order regression. Second order (quadratic) curve fits may not be used when analyzing any South Carolina samples.

### 21.4.3 Calibration for Inorganic Analyses

EPA Method 7000 from EPA SW-846 is a general introduction to the quality control requirements for metals analysis. For inorganic methods, quality control measures set out in the individual methods and in the *Standard Methods for the Examination of Water and Wastewater* (20th Edition) may also be included. Standard Operating Procedures for the analysis and the quality control documentation measures are kept in the departments' SOP reference binders and the Control Limits Manual.

In general, inorganic instrumentation is calibrated with external standards. Some exceptions would be Inductively Coupled Plasma (ICP), Inductively Coupled Plasma Mass Spec (ICPMS), and Ion Chromatography Mass Spec (ICMS). These analyses may use an internal standard to compensate for viscosity or other matrix effects. While the calibration procedures are much the same for inorganics as they are for organics, CF's or RF's are not used as often. The calibration model in 21.4.2.6 is generally used for most methods, however in some instances the model from section 21.4.2.7 may be used. A correlation coefficient (r) of 0.995 or greater must be used to accept a calibration curve generated for an inorganic procedure. Correlation coefficients are determined by hand-held scientific calculators or by computer programs and documented as part of the calibration raw data. Coefficients of calibration curves used for quantitation must be documented as part of the raw data. Curves are not allowed to be stored in calculator memories and must be written on the raw data for the purposes of data validation.

- **21.4.3.1** "Calibrations" for titrimetric analyses are performed by standardizing the titrants against a primary standard solution or certified solutions, as applicable. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (18 and 20th Edition) for more information.
- **21.4.3.2** Spreadsheets that are used for general chemistry calculations must have all cells containing calculations locked to prevent accidental changes to the calculations.
- **21.4.3.3** Instrument technologies (e. g., ICP) with validated techniques from the instrument manufacturer or other methods using a zero point and single point calibration require the following:
  - **21.4.3.3.1** The instrument is calibrated using a zero point and a single point calibration standard.
  - **21.4.3.3.2** The linear range is established by analyzing a series of standards, one at the reporting limit (RL).
  - **21.4.3.3.3** Sample results within the established linear range do not need to be qualified.
  - **21.4.3.3.4** The zero point and single standard is run daily with each analytical batch.
  - **21.4.3.3.5** A standard at the RL is analyzed daily with each analytical batch and must meet established acceptance criteria.
  - **21.4.3.3.6** The linearity is verified at a frequency established by the manufacturer or method.

# 21.4.4 <u>Calibration Verification</u>

The calibration relationship established during the initial calibration must be verified at periodic intervals as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

**Note:** The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration, and is not appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e. g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

**Note:** If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only initial daily verifications are needed. The results from these

verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

- **21.4.4.1** Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.
- **21.4.4.2** A continuing instrument calibration verification (CCV) must be repeated at the beginning and at the end of each analytical batch for non-GC/MS methods. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples.
- **21.4.4.3** The acceptance limits for calibration verifications can be found in each method SOP. As a general rule: GCMS <u>+</u> 20%, GC and HPLC <u>+</u> 15%, Inorganics: <u>+</u> 10 %. Actual methods may have wider or tighter limits; see the method SOP for specifics.
- **21.4.4.4** If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the initial calibration to quantitate sample results.
- **21.4.4.5** If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second, consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two, consecutive, successful, calibration verifications, or a new, initial, instrument calibration must be performed. However, sample data associated with an unacceptable, calibration verification may be reported as qualified data under the following special conditions:
  - **21.4.4.5.1** When the acceptance criteria for the calibration verification are exceeded high, i. e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification must be reanalyzed after a new calibration curve has been established, evaluated and accepted.
  - **21.4.4.5.2** When the acceptance criteria for the calibration verification are exceeded low, i. e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification must be reanalyzed after a new calibration curve has been established, evaluated and accepted.

# 21.4.4.6 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

The Percent Difference is calculated as follows:

% Difference = 
$$(CF(v) \text{ or } RF(v)) - (Avg. CF \text{ or } RF) X$$
 100  
(Avg. CF or RF)

where:

CF(v) or RF(v) = CF or RF from verification standard Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

% Drift = <u>Result - True Value</u> X 100 True Value

The Percent Recovery is calculated as follows:

% Recovery = <u>Result</u> X 100 True Value

# 21.4.4.7 <u>Verification of a Non-Linear Calibration</u>

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations described in 21.4.4.6 above.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

# 21.5 <u>TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS</u>

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification is determined by the purpose of the analyses being conducted. Data system library search routines do not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it must not be reported as a TIC. If the compound is reported on the same form as true TICs, it must be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

- **21.5.1** Use the following guidelines for making tentative identifications
- **21.5.1.1** Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) are present in the sample spectrum.
- **21.5.1.2** The relative intensities of the major ions must agree within  $\pm$  20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
- **21.5.1.3** Molecular ions present in the reference spectrum should be present in the sample spectrum.
- **21.5.1.4** lons present in the sample spectrum but not in the reference spectrum are reviewed for possible background contamination or presence of co-eluting compounds.
- **21.5.1.5** lons present in the reference spectrum but not in the sample spectrum are reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

The concentration of any non-target analytes identified in the sample (see above) are estimated. The same formulae as calibrated analytes is used with the following modifications: The areas  $A_s$  and  $A_{is}$  are from the total ion chromatograms, and the RF for the compound is assumed to be 1.

The resulting concentration is reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

Note: The above guidelines above are from EPA SW846 III edition, method 8260B.

For general reporting if TICs are requested, the 10, largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard are termed "Tentatively Identified Compounds" (TICs). More or fewer TICs may be identified based on client requirements.

# 21.5.2 <u>TIC Reporting Limits (RLs)</u>

In general RLs cannot be specified because of the unknown nature of the TIC. Any reporting limit that is reported can only be evaluated as an estimate as the quantitation is based on the

assumption that the TIC responds exactly as the IS responds which is most likely not the case. In general, it is not recommended to set a Reporting limit at too low of a concentration as it gives a false impression.

TICs that meet the above identification criteria (Section 21.5.1) at 10% area of the IS: The RL would be 10% of the concentration of the internal standard used for quantitation (e. g., 2.5 ug/L for 8260B, 4.0 ug/L for 8270C). In general, if the 10% area criterion is not met, the TIC RLs are set at a level approximately five times the level of the poorest performer in the analysis.

If a compound meets the TIC criteria, the reporting limit must reflect the ratio between the TIC and the IS or five times the level of the poorest performer whichever is lower.

# 21.6 <u>GC/MS TUNING</u>

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

**21.6.1** The concentration of the BFB or DFTPP must be at or below the concentrations that are referenced in the analytical methods. Part of the purpose of the tune is to demonstrate sensitivity and analyzing solutions at higher concentrations does not support this purpose. Tune failures may be due to saturation and a lower BFB/DFTPP concentration may be warranted.

**21.6.2** Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex  $\pm 1$  scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

- **21.6.3** Other Options or if Auto Tune Fails:
- **21.6.3.1** Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. In this case, manually identify and average the apex peak ±1 scan and background correct as in Section 21.6.2 above. This is consistent with EPA 8260 and 8270.
- **21.6.3.2** Or the scan across the peak at one half peak height may be averaged and background corrected. This is consistent with Standard Methods 6200, EPA 624 and EPA 625.
- **21.6.3.3** Adjustments such as adjustments to the repeller and ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as <u>all</u> of the subsequent injections in the 12 hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than 2 tries) without clear

documentation is not allowed. Necessary maintenance is performed and documented in instrument log.

- **21.6.3.4** A single scan at the Apex (only) may also be used for the evaluation of the tune. For SW 846 and EPA 600 series methods, background correction is still required.
- **21.6.3.5** Cleaning the source or other maintenance may be performed and then follow steps for tune evaluation above. The maintenance must be documented in the maintenance log and should be noted in the sequence log. Note: If significant maintenance was performed, see methods 8000B or 8000C then the instrument may require recalibration prior to proceeding.

**21.6.4** Tune evaluation printouts must include the chromatogram and spectra as well as the tune evaluation information. In addition, the verifications must be sent directly to the printer or pdf file (no screen prints for DFTPP or BFB tunes). This ability is typically built into the instrument software.

**21.6.5** Since the limits are expressed in whole percentages, the results may be rounded to whole percentage before comparing to criteria when assessing the tune verification against the tune requirements. However, the comparison to the criteria is usually done automatically by the software and if the printout says "Fail" then there would have to be documentation on the raw data and comparison to the criteria if the lab intends to still accept the tune. In most cases the analyst is better off performing an adjustment and rerunning the tune standard.

**21.6.6** All MS tune settings must remain constant between running the tune check and all other samples. It is recommended that a separate tune method not be used, however a separate method may be used as long as the MS conditions between the methods are the same as the sample analysis method and tracked so any changes that are made to the analysis method are also made to the tune method.

### Table 21-1.

# Laboratory Equipment and Instrumentation

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
ABSORPTION UNIT	MITSUBISHI	Sigma 10	7520591	1996	USED
ACCELERATED SOLVENT EXTRACTOR	DIONEX	ASE200	02060437	2002	NEW
ACCELERATED SOLVENT EXTRACTOR	DIONEX	ASE200	02060440	2002	NEW
AUTO BOAT	MITSUBISHI	ABC	75A10673	1996	USED
AUTOSAMPLER	CENTAC TECHNOLOGIES	ASX-510	120347ASX	2005	NEW
AUTOSAMPLER	AMERICAN PRECISION	3i9A	9033	1992	1
AUTOSAMPLER	AMERICAN PRECISION	32E-6108AY	9205	1992	1
AUTOSAMPLER	DIONEX	ASI-100	4290509	2005	NEW
AUTOSAMPLER	DIONEX	ASI-100	4440407	2005	NEW
AUTOSAMPLER	EST	CENTURION	CENT123020504	1	1
AUTOSAMPLER	EST	CENTURION	CENT153112904	1	1
AUTOSAMPLER	EST	CENTURION	CENT124030104	2004	1
AUTOSAMPLER	EST	CENTURION	CENT126030104	1	1
AUTOSAMPLER	EST	CENTURION	CENT197042106	1	1
AUTOSAMPLER	EST	CENTURION	CENT196041706	1	1
AUTOSAMPLER	EST	CENTURION	CENT171060705	1	1
AUTOSAMPLER	EST	CENTURION	CENT172060705	1	1
AUTOSAMPLER	EST	CENTURION	CENT176102105	1	1
AUTOSAMPLER	EST	CENTURION	CENT174060705	1	1
AUTOSAMPLER	EST	CENTURION	CENT198050106	1	1
AUTOSAMPLER	EST	ENCON	215081902	1	1
AUTOSAMPLER	EST	ENCON	219082202	1	1
AUTOSAMPLER	HEWLETT PACKARD	718593B	3114A25542	1	1
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3247A30576	2005	1
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3514A42318	1	1
AUTOSAMPLER	HEWLETT PACKARD	HP7673A	2843A1152	1	1
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3248A33147	1	1
AUTOSAMPLER	HEWLETT PACKARD	HP7673	2546A01716	1	1
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3514A42318	2007	USED
AUTOSAMPLER	SHIMADZU	SIL-10A	60341F	1997	NEW
AUTOSAMPLER	SHIMADZU	SIL-10A	C20363506886	1999	NEW
AUTOSAMPLER	SHIMADZU	SIL-10A	C20363506585	1999	NĘW
AUTOSAMPLER	SHIMADZU	AOC20i	C11143602793		1
AUTOSAMPLER	SHIMADZU	AOC20i	C11144208289	2004	1
AUTOSAMPLER	SHIMADZU	AOC20i	C11143703828	1	1
AUTOSAMPLER	SHIMADZU	AOC20i	C11143703823	'	'

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-17 of 21-33

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
AUTOSAMPLER	SHIMADZU	AOC20i	C11143602454	1	1
AUTOSAMPLER	SUPERIOR ELECTRIC	MO61-LF-517	A218524	1992 OR 93	1
AUTOSAMPLER	SUPERIOR ELECTRIC	MO61-LF-517	033473653	2003	1
AUTOSAMPLER	SUPERIOR ELECTRIC	MO61-LF-517	040939389	2004	1
AUTOSAMPLER	THERMO JARRELL ASH	AS300	12677003	1994	NEW
AUTOSAMPLER	THERMO JARRELL ASH	AS300	0492	1997	NEW
AUTOSAMPLER	THERMO JARRELL ASH	AS300	67247	1998	USED
AUTOSAMPLER	OI ANALYTICAL	4552	LR92489	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	11798-895	1	1
AUTOSAMPLER	VARIAN	ARCHON	12314	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	13799	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	13795	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	13735	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	13790	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	13800	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	12201	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12194	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12307	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12952	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12761	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12974	1999	NEW
AUTOSAMPLER	VARIAN	ARCHON	13161	1	1
AUTOSAMPLER	VARIAN	ARCHON	13812	1	1
AUTOSAMPLER	VARIAN	ARCHON	13765	1	1
AUTOSAMPLER	VARIAN	ARCHON	12945	1	1
AUTOSAMPLER	VARIAN	ARCHON	13722	1	1
AUTOSAMPLER	VARIAN	ARCHON	12920	1	1
AUTOSAMPLER	VARIAN	ARCHON	13600	1	1
AUTOSAMPLER	VARIAN	ARCHON	13641	1	1
AUTOSAMPLER	VARIAN	ARCHON	13087	1997	NEW
AUTOSAMPLER	VARIAN	ARCHON	12958	1996	USED
AUTOSAMPLER	VARIAN	ARCHON	13748	1995	NEW
AUTOSAMPLER	VARIAN	ARCHON	13291	1994	NEW
AUTOSAMPLER	VARIAN	ARCHON	13077	1	1
AUTOSAMPLER	VARIAN	ARCHON	13723	2006	USED
AUTOSAMPLER	VARIAN	ARCHON	13165	1994	NEW
AUTOSAMPLER TOWER	HEWLETT PACKARD	18596C	3530A43392	1	1
AUTOSAMPLER	HEWLETT PACKARD	18596C	3120A26333	1	1
AUTOSAMPLER	HEWLETT PACKARD	18596C	3120A27415	1	1
TOWER AUTOSAMPLER TOWER	HEWLETT PACKARD	18593A	2843A1162	1	1

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-18 of 21-33

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
AUTOSAMPLER TOWER	HEWLETT PACKARD	18593B	3114A25542	1	1
AUTOSAMPLER TOWER	HEWLETT PACKARD	18593B	3447A40940	1	1
AUTOSAMPLER TOWER	AGILENT	7683	US03815233	1	1
AUTOSAMPLER	AGILENT	7683	US91807060	1	1
AUTOSAMPLER TOWER	AGILENT	7683	US94209739	1	1
AUTOSAMPLER TOWER	AGILENT	7683	US00411367	1	1
AUTOSAMPLER TOWER	AGILENT	7683	CN30729442	2010	USED
AUTOSAMPLER TOWER	AGILENT	1200	DE64775625	2010	NEW
AUTOSAMPLER TOWER	HEWLETT PACKARD	7673	3351A37407	2010	AUTOSA MPLER
AUTOSAMPLER TRAY	HEWLETT PACKARD	18593B	3106A21456	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD	18593B	3415A35076	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD	18593B	3339A33311	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD		3348A31236	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD		(unreadable)	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD		3131A25799	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD		3304A31194	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD	6890	3514A42318	2010	USED
AUTOSAMPLER TRAY	HEWLETT PACKARD	6890	3415A35076	2010	USED
AUTOSAMPLER TRAY	AGILENT	7683	US82601220	1	1
AUTOSAMPLER TRAY	AGILENT	7683	US81100420	1	1
AUTOSAMPLER TRAY	AGILENT	7683	US24314927	1	1
AUTOSAMPLER TRAY	AGILENT	7683	US00407128	1	1
AUTOSAMPLER TRAY	AGILENT	7683	CN91653019	2010	USED
AUTOSAMPLER	AGILENT	1200	DE64775625	2010	NEW
AUTOSAMPLER TRAY	EST	CENTURION WS	CENTS1570526 10	2010 USED	

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-19 of 21-33

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
AUTOSAMPLER	MARKELOV	HS9000	HS121053007	2007	NEW
WITH HEADSPACE		1139000	113121033007	2007	
AUTOSAMPLER	MARKELOV	HS9000	HS120053007	2007	NEW
WITH HEADSPACE		1139000	113120033007	2007	
BALANCE	DENVER INSTR.	XL610	NO113767	1	1
BALANCE	DENVER INSTR.	XE010 XE310	NO109645	1	1
BALANCE	METTLER	AE240	N28161	1993	NEW
BALANCE	METTLER	PB602-S	1118381704	1995	
BALANCE	METTLER	PB602-3	1117421936	1	1
BALANCE	METTLER	PB602-S	1126143319	2005	NEW
BALANCE	METTLER	PB002-5 PG802	1115352767	2005	
				1	
BALANCE	METTLER	PB602-S	1120430697	1	NEW
BALANCE	METTLER	AE200	F15224	1	1
BALANCE	METTLER	PM600T	H19877	1	1
BALANCE	METTLER	PB302	1116383230	1	1
BALANCE	METTLER	AE200	L94571	1	
BALANCE	METTLER	PG203-S	119331062	1	NEW
BALANCE	METTLER	AE200	J63202	1	NEW
BALANCE	METTLER	PL601-S	1202140224	1	NEW
BALANCE	METTLER	AG204	1122173241	2003	NEW
BLOCK DIGESTOR	LACHAT	BE – 46	10040000973	2010	NEW
BOD INCUBATOR	FISHER	307	WB05105311	1990	NEW
BOD INCUBATOR	FISHER	FU199ERWI	FGL52777	1989	USED
BOD INCUBATOR	KENMORE	253.65802508	BA73420257	2008	NEW
BOD INCUBATOR	EQUATHERM	HA757376	TA14SLB	1992	USED
CALORIMETER	PARR	1341	5427	1986	NEW
CENTRIFUGE	BECKMAN	ALLEGRA 6	ALS08D16	2009	NEW
CENTRIFUGE	CLAY ADAMS	DYNAC III 420104	3820031	1	NEW
CENTRIFUGE	CLAY ADAMS	DYNAC III 420104	4150009	1	NEW
CHILLER	NESLAB	CFT-33	293223	1993	NEW
COD REACTOR	HACH		901103566	1990	NEW
CONDUCTIVITY METER	WTW	330i	0148004	2002	NEW
DISCRETE ANALYZER	KONELAB	AQUA 20	E0719526	2004	NEW
DISCRETE	KONELAB	AQUA 20	S2419226	2009	USED
DISSOLVED OXYGEN METER	YSI	5100	00B0314	2000	NEW
DISSOLVED OXYGEN METER	YSI	5100	00D0552	2000	NEW
FLASHPOINT TESTER, AUTO	PETROLAB	PMA-4	741	1997	NEW
FLASHPOINT TESTER, AUTO	PETROLAB	PMA-4	741	1997	NEW
FLASHPOINT, OPEN CUP	FISHER		201N0005	2002	NEW
FLASHPOINT, PENSKY-MARTENS	FISHER	13-497-5	1787	1986	NEW

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-20 of 21-33

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
FLOW INJECTION ANALYZER	LACHAT	8000	A83000234	1995	NEW
FLOW INJECTION ANALYZER	LACHAT	FIA+	A83000-1323	1999	NEW
FLOW INJECTION ANALYZER	LACHAT	8500 FIA	050600000167	2008	USED
FLOW INJECTION ANALYZER	LACHAT	8500	090400001098	2009	NEW
FLUOROMETER	SEQUOIA - TURNER	450	B002262TV	1992	NEW
FURNACE	THERMOLYN	30400	10599810039263	1984	NEW
GC w/ECD/ECD	PERKIN ELMER	AUTOSYS	610N9122009	2000	NEW
GC w/ECD/ECD	PERKIN ELMER	AUTOSYS	610N9122008	2000	NEW
GC w/ECD/ECD	PERKIN ELMER	AUTOSYS XL	610N9122010	2000	NEW
GC w/ECD/ECD	SHIMADZU	17A	C11123681357	1999	NEW
GC w/ECD/ECD	HEWLETT PACKARD	6890	US00011427	2000	NEW
GC w/ECD/ECD	HEWLETT PACKARD	5890	3336A61865	2006	USED
GC w/ECD/ECD	HEWLETT PACKARD	5890	3336A51644	2007	USED
GC w/ECD/ECD	HEWLETT PACKARD	7890A	CN10734082	2009	NEW
GC w/FID	HEWLETT PACKARD	5890	2950A27678	1	1
GC w/FID	HEWLETT PACKARD	5890	3108A34413	1999	USED
GC w/FID	HEWLETT PACKARD	5890	3235A45233	2006	USED
GC w/FID	HEWLETT PACKARD	5890	2950A26161	2006	USED
GC w/FID	HEWLETT PACKARD	5890	3203A42028	2006	USED
GC w/FID	HEWLETT PACKARD	5890/7673	3235A45233	2006	USED
GC w/FID	HEWLETT PACKARD	5890	3336A51997	2006	USED
GC w/FID	HEWLETT PACKARD	5890	3235A46759	2009	USED
GC w/FID	HEWLETT PACKARD	6890/7683	US00029922	2010	USED
GC w/FID	HEWLETT PACKARD	6890	US00010165	2010	USED
GC w/FID	HEWLETT PACKARD	5890	3336A61864	2010	USED
GC w/FID	HEWLETT PACKARD	6890	CN10727030	2010	NEW
GC w/FID	PERKIN ELMER	AUTOSYS	610N1040206	1992	NEW
GC w/FID	PERKIN ELMER	CLARUS 500	650N3100901	2003	NEW
GC w/FID	PERKIN ELMER	CLARUS 500	650N4031504	2004	NEW
GC w/FID	SHIMADZU	17A	C11123580493	1998	NEW
GC w/FID	SHIMADZU	17A	C11123681362	1999	NEW
GC w/FID	SHIMADZU	17A	C11123781625	1999	NEW
GC w/FID	SHIMADZU	17A	C11123781731	1999	NEW

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-21 of 21-33

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
GC w/NPD/FPD	PERKIN ELMER	CLARUS 500	650N4061602	2004	NEW
GC w/PID/FID	HEWLETT PACKARD	5890	3027A29703	2001	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3126A36232	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	2908A91269	1990	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3133A37656	1996	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3203A42091	1996	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3336A58503	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	2938A24989	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3336A50264	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	2938A25493	1998	USED
GC w/PID/FID	HEWLETT PACKARD	6890	US00041322	2002	NEW
GC w/PID/FID	HEWLETT PACKARD	6890	US00041324	2002	NEW
GC w/PID/FID	HEWLETT	6890	US00027429	2002	NEW
GC w/PID/FID	HEWLETT	5890	3336A60618	2003	USED
GC w/PID/FID	HEWLETT PACKARD	5890	DE00003878	2003	NEW
GC w/PID/FID	HEWLETT	6890N	US10350005	2004	NEW
GC w/PID/FID	HEWLETT PACKARD	6890N	US10350002	2004	NEW
GC w/PID/FID	HEWLETT PACKARD	5890	2950A27370	2005	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3223A43282	2005	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3019A28662	2005	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3203A41711	2006	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3203A41407	2007	USED
GC w/PID/FID	SHIMADZU	17A	C11123580601	1998	NEW
GC w/PID/FID	SHIMADZU	17A	01123681361	1999	NEW
GC w/PID/FID	TREMETRICS	9001	100035	1996	NEW
GC w/PID/FID	TREMETRICS	9001	100036	1996	NEW
GC w/PID/FID	VARIAN	3400	4035	1999	USED
GC w/TCD	SHIMADZU	8A	C10493313148	1998	
GC/MS	HEWLETT	589011/5972	3307A00396	1998	USED
GC/MS	HEWLETT PACKARD	5890II/5971	3234A04319	1994	NEW

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-22 of 21-33

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
GC/MS	HEWLETT PACKARD	5890II/5971	2749A00081	1997	NEW
GC/MS	HEWLETT PACKARD	589011/5972	3329A00693	1994	NEW
GC/MS	HEWLETT	5890115972	3501A02393	1995	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US82311321	1999	USED
GC/MS	HEWLETT PACKARD	6890/5973	US80210937	1999	USED
GC/MS	HEWLETT PACKARD	6890/5973	US94222434	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US01140208s	2000	NEW
GC/MS	HEWLETT PACKARD	G1530A/ G1099A	US91421544	1999	NEW
GC/MS	HEWLETT PACKARD	G1530A/ G1099A	3435A01921	1998	1
GC/MS	HEWLETT PACKARD	6890/5973	US81221486	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US91921735	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US94212231	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US94211203	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US94260109	2001	USED
GC/MS	HEWLETT PACKARD	6890/5973N	US03340475	2001	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US03340479	2002	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US21844021	2002	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US21844025	2002	NEW
GC/MS	HEWLETT PACKARD	5890/5972	3336A57928	2004	USED
GC/MS	HEWLETT PACKARD	5890/5972	3235A44162	2005	USED
GC/MS	HEWLETT PACKARD	5890/5972	2423A01966	2005	USED
GC/MS	HEWLETT PACKARD	5890/5973	US00027481	2005	USED
GC/MS	HEWLETT PACKARD	6890/5973	4510250045	2005	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00037997	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00037995	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00027177	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00033436	2006	USED

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-23 of 21-33

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
GC/MS	HEWLETT PACKARD	6890/5973	US00008026	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00022681	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00022412	2008	USED
GC/MS	HEWLETT PACKARD	6890/5973	US10462174	2009	NEW
GC/MS	HEWLETT PACKARD	6890/5973	Us10204013	2010	USED
GC/MS w/ENTEC CONCENTRATOR	HEWLETT PACKARD	6890/5973N	US10450528	2001	NEW
GEL PERMEATION CLEANUP	O. I. ANALYTICAL	AUTOPREP 2000	E427330254	2004	NEW
HEADSPACE ANALYZER	TEKMAR	7000	91242001	2001	USED
HOTPLATES	THERMODYNE	CIMAREC 3		1991 -	NEW
HPLC	SHIMADZU	SCL-10AVP	C21013650343	1999	NEW
HPLC	DIONEX	TCC-100MSV	1280301	2005	NEW
HPLC	DIONEX	P680A LPG-4	5550503	2005	NEW
HPLC SYSTEM (ISOCRATIC)	SHIMADZU	SCL-10AVP	C21013650380	1999	NEW
HPLC	AGILENT	1200	DE64255220 DE60557139	2010	NEW
ICP - TRACE	JARRELL - ASH	61 E	472790	1997	USED
ICP – TRACE	THERMO- FISHER	6500	20100905	2010	NEW
ICP – TRACE	THERMO	ICAP 6000 SERIES	20084103	2009	NEW
ICP/MS	HEWLETT PACKARD	7500	JP51201460	2005	NEW
ION CHROMATO- GRAPH	METROHM	732	01631	1998	NEW
ION CHROMATO- GRAPH	METROHM	732	08140	1998	NEW
ION CHROMATO- GRAPH	METROHM	819	10149	2010	NEW
ION CHROMATO- GRAPH	METROHM	861	04157	2010	NEW
ION CHROMATO- GRAPH	METROHM	861	04168	2010	NEW
ION CHROMATO- GRAPH	METROHM	861	07200	2010	NEW
ION CHROMATO- GRAPH	METROHM	861	03135	2010	NEW
KARL – FISCHER TITRATOR	BRINKMAN	684	16840013	1993	NEW
MECHANICAL SHAKER	GLAS-COL	VS50012	399665	2005	NEW
MECHANICAL SHAKER	GLAS-COL	VS50012	399872	2005	NEW
MERCURY ANALYZER	LEEMAN	Hydra II	0031	2010	NEW

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-24 of 21-33

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
MERCURY ANALYZER	LEEMAN	HYDRA AA	6008	2008	USED
MERCURY	LEEMAN	HYDRA II	0002	2010	NEW
ANALYZER		in Broth	0002	2010	
MICROWAVE	CEM	MARS XPRESS 907501	MD2979	2009	NEW
MICROWAVE	CEM	MARS XPRESS 907501	MD8469	2006	NEW
MIDI-DISTILLATION	ANDREWS GLASS		MCVA1290374	1994	NEW
MIDI-DISTILLATION	WESTCO	EASY-DIST	EA-1006	1996	NEW
OVEN	BLUE M	SW11TA1	SW3034	1989	NEW
OVEN	BLUE M	SW17TA1	SW7252	1993	NEW
OVEN	BLUE M	SWIITA-1	SW3990	1	1
OVEN	FISHER	ISOTEMP 650G	803N0038	1	1
OVEN	FISHER	ISOTEMP 516G	903N0083	1	1
OVEN	FISHER	500	51100409	1	1
OVEN	NAPCO	620	(ILLEGIBLE)	1	1
OVEN	FISHER	ISOTEMP 650G	202N0036	2002	NEW
OVEN	FISHER	6926	608516-30	2010	NEW
OVEN	FISHER	6926	275812-45	2011	NEW
OXYGEN PROBE	ORION	97-08-00		1991	NEW
pH METER	ORION	330	005043	2000	NEW
pH/mv METER	ACCUMET	10	C9002414	1994	NEW
PH/mv METER	ACCUMET	XL15	5186494	2007	NEW
pH/mv METER	CORNING	220	12351	1991	NEW
pH/mvMETER	ACCUMET	AB15	AB92331859	2009	NEW
pH/mvMETER	ACCUMET	AB15	642122	2011	NEW
PURGE/TRAP	EST	ENCON	217082202 E	1997	NEW
PURGE/TRAP	EST	ENCON	126112900 E	1997	NEW
PURGE/TRAP	EST	ENCON	216081902 E	1994	NEW
PURGE/TRAP	EST	ENCON	218082202 E	1994	NEW
PURGE/TRAP	EST	ENCON	215081902E/P	1	1
PURGE/TRAP	EST	ENCON	219082202E/P	1	1
PURGE/TRAP	EST	ENCON	237082022	2006	USED
PURGE/TRAP	EST	ENCON	126112900	2006	USED
PURGE/TRAP	HEWLETT PACKARD	G1900-60500	3651A10630	1995	NEW
PURGE/TRAP	HEWLETT	3000	3636A10579	1	1
PURGE/TRAP	O.I. ANALYTICAL	4560	K811460272	1998	NEW
PURGE/TRAP	O.I. ANALYTICAL	4560	D308371	1993	NEW
PURGE/TRAP	O.I. ANALYTICAL	4560	H421460320	1994	NEW
PURGE/TRAP	TEKMAR	3000	93251007	1993	NEW
PURGE/TRAP	TEKMAR	3000	95093006	1995	NEW
PURGE/TRAP	TEKMAR	3000	99053016	2003	1
PURGE/TRAP	TEKMAR	3000	94057008	1	1
PURGE/TRAP	TEKMAR	3000	98236009	1	1
PURGE/TRAP	TEKMAR	3000	98139001	1	1
PURGE/TRAP	TEKMAR	3000	98098001	1	1

#### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-25 of 21-33

Table 21-1:				Year	Condition
Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Put into Service	When Received
PURGE/TRAP	TEKMAR	3000	98364008		1
PURGE/TRAP	TEKMAR	3000	93316011	1	1
PURGE/TRAP	TEKMAR	3000	94252002	1	1
PURGE/TRAP	TEKMAR	3000	94252002	1	1
PURGE/TRAP	TEKMAR	3000	93334005	1	1
PURGE/TRAP	TEKMAR	3000	95144001	1	1
PURGE/TRAP	TEKMAR	3000	95166007	1	1
PURGE/TRAP	TEKMAR	3000	94266005	1	1
PURGE/TRAP	TEKMAR	3000	99196020	1	1
PURGE/TRAP	TEKMAR	3000	97009013	1	1
PURGE/TRAP	TEKMAR	3000	98092002	1	1
PURGE/TRAP	TEKMAR	3000	995207010	1	1
PURGE/TRAP	TEKMAR	3000	98008002	1	1
PURGE/TRAP	TEKMAR	3000	3449A10215	1	1
PURGE/TRAP	TEKMAR	3000	95153007	1	1
PURGE/TRAP	TEKMAR	3000	98229017	1	1
PURGE/TRAP	TEKMAR	3000	94005035	1	1
PURGE/TRAP	TEKMAR	3000	94005015	1	1
PURGE/TRAP	TEKMAR	3000	96325013	1	1
PURGE/TRAP	TEKMAR	3000	93170002	1	1
PURGE/TRAP	TEKMAR	3000	99076004	1	1
PURGE/TRAP	TEKMAR	LCS-3000	94067010	2002	NEW
PURGE/TRAP	TEKMAR	3100	02115005	1	1
PURGE/TRAP	TEKMAR	3100	98152010	1	1
PURGE/TRAP	TEKMAR	3100	00104011	1	1
PURGE/TRAP	TEKMAR	3100	00104012	1996	USED
PURGE/TRAP	TEKMAR	3100	99305025	1	1
PURGE/TRAP	TEKMAR	3100	US01354002	2004	1
PURGE/TRAP	TEKMAR	3100	US02249003	2004	1
PURGE/TRAP	TEKMAR	3100	US02155008	1	1
PURGE/TRAP	TEKMAR	3100	US02326003	1	1
PURGE/TRAP	TEKMAR	3100	95129003	1	1
PURGE/TRAP	TEKMAR	3100	95039008	1	1
PURGE/TRAP	TEKMAR	3100	94104002	1	1
PURGE/TRAP	TEKMAR	3100	97273002	1	1
PURGE/TRAP	TEKMAR	LSC 2000	91309006	1997	NEW
PURGE/TRAP	TEKMAR	LSC2000	177003	1997	NEW
PURGE/TRAP	TEKMAR	LSC2000	90100003	1998	NEW
PURGE/TRAP	TEKMAR	LSC2000	90115009	1998	NEW
PURGE/TRAP	TEKMAR	LSC2000	88274012	1998	NEW
PURGE/TRAP	TEKMAR	LSC-2000	8826010	1988	NEW
PURGE/TRAP	TEKMAR	LSC-2000	91092004	1991	NEW
PURGE/TRAP	TEKMAR	LSC-2000	90043025	1991	NEW
PURGE/TRAP	TEKMAR	LSC-2000	92008004	1992	NEW
RECIPROCAL WATER	NEW	R 76	400364888	2003	NEW
ВАТН	BRUNSWICK SCIENTIFIC				
ROTATOR	ASSOC. DESIGN	3740-48BRE		1994	NEW

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-26 of 21-33

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
SOLID PHASE EXTRACTOR	HORIZON TECHNOLOGY	SPE-3000XL	01-1200	2004	NEW
SOLVENT CONCENTRATOR	LABCORCO	NVAP	020998064F	2002	NEW
SOLVENT CONCENTRATOR	LABCORCO	NVAP	020897646F	2002	NEW
SOLVENT CONCENTRATOR	LABCORCO	NVAP	020998063F	2002	NEW
SONICATOR	SONICS & MATERIALS	VCX600	28785F	1999	NEW
SONICATOR	SONICS & MATERIALS	VCX600	19574D	1998	NEW
SONICATOR	SONICS & MATERIALS	VCX600	18683	2001	NEW
SONICATOR	SONICS & MATERIALS	QVC502	26925	1998	NEW
SONICATOR	SONICS & MATERIALS	VC501	23840F	1997	NEW
SONICATOR	SONICS & MATERIALS	VC750	31469F	2000	NEW
SONICATOR	SONICS & MATERIALS	VC750	37121G	2003	NEW
SONICATOR	SONICS & MATERIALS	VC750	31321G	2000	NEW
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
THERMOMETER (NIST)	ERTCO	ASTM 62C	4240	2004	NEW
THERMOMETER (NIST)	ERTCO	ASTM 62C	4254	2004	NEW
THERMOMETER (NIST)	ERTCO	1007	3161	2006	NEW
THERMOMETER (NIST)	ERTCO	1007	3437	2009	NEW
TITRATOR, AUTO	MAN-TECH	PCM-1104-00	MS-OC7-752	2008	NEW
TITRATOR, AUTO	MAN-TECH	PC-1000.688	MS-1A1-603	2011	NEW
TOC	SHIMADZU	5050	29118894	1993	NEW
TOC	SHIMADZU	TOC-VCSN	H51204635241	2009	NEW
TOC	O.I. ANALYTICAL	1030W	F923730891 AUTOSAMPLER: 612788821	2009	NEW
TOC W/SOLIDS OPTION	SHIMADZU	5050A	36001341	1999	NEW
ТОХ	MITSUBISHI	SIGMA 10	75R00110	1998	USED
TOX	MITSUBISHI	TOX-100	A7M41934	2009	USED

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-27 of 21-33

Table 21-1: Equipment/				Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
TOX	MITSUBISHI	TOX100	A7M20849	1999	NEW
TURBIDIMETER	HF	DRT – 100B	602164	2000	NEW
TURBIDIMETER	HF	MICRO 100	201008129	2010	NEW
UV/VIS	SHIMADZU	160U	28D06563	1990	NEW
UV/VIS	SHIMADZU	1601	A10753782132	2001	NEW
UV/VIS	HACH	DR/2500	030500005514	2003	NEW
ZHE PRESSURE	MILLIPORE	T316	22361028	1993	NEW
ZHE ROTATOR (4)	MILLIPORE	Z3	455TR4042	1990	NEW

<sup>1</sup>Although equipment is operational and calibration maintained, this information is not available.

Table 21-2. Pr	eventive Maintenance Procedure	es for Laboratory Equipment
Table 21-2:	Activity	Frequency
Instrument/Equipment		
Туре		
Gas Chromatograph	Change septum	As needed – record
	Check gases	Daily
	Replace or clip column	As needed – record. Rerun
	Clean detector	calibration/RT study As needed – record
	Check autosampler seals	Daily
	Clean injectors; replace liners	As needed – record
	Clean or replace PID lamp	As needed – record
HPLC or IC	Vendor repair	As needed – record work order
HPLC OF IC	Check seals for leakage	Each use
	Replace seals/valves/lamps	As needed – record
	Replace suppressor (IC only)	As needed – record
	Replace column	As needed – record. Rerun
		calibration/RT study
	Clean source/analyzer	As needed – record
00/040	Vendor repair	As needed – record work order
GC/MS	Change Merlin (SVOC only)	As needed – record
	Bake trap (VOC only)	Daily
	Clean source	As needed – record
	Change vacuum pump oil	Biannually – record
	Clean injector; replace liner (SVOC only)	Daily
	Replace column	As needed – record. Rerun
		calibration/RT study
	Vendor repair	As needed – record work order
ICP	Torch inspection	Each use
	Clean torch and nebulizer	As needed – record
	Inspect filters	Daily
	Change filters	As needed – record
	Inspect pump tubing	Daily
	Change pump tubing	As needed – record
	Vendor repair	As needed – record work order
	Inspect contact rings	Each use
	Clean windows	Each use
	Align lamp	Each use
	Vendor repair	As needed – record work order
Mercury Analyzer	Inspect tubes and reagents	Daily
	Vendor repair	As needed – record work order
Microwave	Check power output	Weekly
	Check cleanliness and rotation	Each use
	Vendor repair	As needed – record work order
InfraRed spectrometer	Change dessicant	As needed – record
	Vendor repair	As needed – record work order

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-29 of 21-33

Table 21-2: Instrument/Equipment Type	Activity	Frequency
pH Meter	Clean electrode	Each use
	Inspect electrode	Each use
	Vendor repair	As needed – record work order
TCLP Extractors	Verify rotation	Each use – record
And Zero Headspace	Check for leakage	Each use
Extractors	Vendor repair	As needed – record work order
Turbidimeter	Check lamp	Each use
	Clean sample holder	Each use
	Vendor repair	As needed – record work order
UV/VIS Spectrometer	Check paper	Daily
-	Clean sample compartment	As needed
	Auto-check calibration	Daily at start-up
	Wavelength calibration	Six months – record
	Vendor repair	As needed – record work order
Total Organic Carbon	Check gas flow	Daily
Analyzer	Check fluid level (IC reservoirs)	Daily
-	Replace "O" rings	As needed – record
	Check needle	Each use
	Replace scrubbers (halogen and $CO_2$ )	Yearly – record
	Replace catalyst	As needed – record
	Vendor repair	As needed – record work order
Total Organic Halogen	Clean inlet tube	As needed
Analyzer	Clean cell	Each use
	Check electrode levels	Each use
	Vendor repair	As needed – record work order
Weighing balances	Clean pan	Each use
0 0	Check calibration	Daily – record
	Vendor repair	As needed – record work order
Temperature devices:	Monitor temperature	Daily or when used (refrigerators
refrigerators, incuba-	•	2 times per day) - record
tors, evaporators, flash point tester, COD re- actor, water circulator, drying ovens	Vendor repair	As needed – record work order
Ultrasonic Disruptors	Clean, Tune by depressing tuning button while adjusting % output knob until lowest reading is obtained. For dual head, disconnect one horn at a time. Vendor repair	Each use record As needed – record work order

Table 21-2	Activity	Frequency
Instrument/Equipment Type		
Discrete Analyzer	Perform "start-up"	Daily
	Wash procedure	Daily
	Run water blank	Daily
	Clear daily files	Daily
	Check water and waste	Weekly
	container; empty if needed	
	Check cuvette bin	Weekly
	Check syringe plunger tip;	Weekly; as needed
	replace syringe	
	Run dichromate at 480 ηm, if	As needed
	problems with water blanks	
	are noted.	
	Save database to CD	Monthly
	Delete messages	Monthly
	Clean/lubricate incubator rod	Monthly
	and fetcher arm	
	Replace lamp	Every 6 months

Table 21-3: Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Analytical Balance	Accuracy determined using A2LA-accredited NIST weights. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person semi-annually.	Calibrated semi-annually. Daily calibration verification prior to use.	± 0.2%	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service.
Top Loading Balance	Accuracy determined using A2LA-accredited NIST weights. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person semi-annually.	Calibrated semi-annually. Daily calibration verification prior to use.	± 0.5%	Clean. Replace.
A2LA- accredited NIST Weights	Accuracy determined by accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
NIST- Traceable Thermo- meter	Accuracy determined by A2LA-accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
Thermo- meter (liquid)	Against NIST-traceable thermometer	Yearly at appropriate temperature range for intended use	± 2.0°C	Replace
Minimum- Maximum Thermo- meters	Against NIST-traceable thermometer	Yearly	± 2.0°C	Replace

# Table 21-3. Periodic Calibration

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-32 of 21-33

Table 21-3:	Type of Calibration/		Acceptance	Corrective
Instrument	Number of Standards	Frequency	Limits	Action
InfraRed Temperature Guns	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 1.5°C	Repair/replace
Dial-type Therm- ometers	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 2.0°C	Replace
Digital Therm- ometers	Against NIST-traceable thermometer	Quarterly	±2.0°C	Replace
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, check again in two hours.	2.7 ± 1.7°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Freezer	Temperature checked using NIST-traceable thermometer	Daily. If out of range, check again in two hours.	(-10)-(-20)°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Oven	Temperature checked using NIST-traceable thermometer.	When in use.	104 ± 1°C (drying) 180 ± 2°C (TDS)	Adjust. Replace.
Incubator	Temperature checked using NIST-traceable thermometer.	When in use.	BOD: 20 ± 1.0°C	Adjust. Replace.
Water Bath	Temperature checked using NIST-traceable thermometer.	When in use.	± 2°C	Adjust. Replace.
Volumetric Dispensing Devices (Eppendorf ® pipette, automatic dilutor or dispensing devices)	One delivery by weight. Using DI water or Methanol, dispense into tared vessel. Record weight with device ID number. Note: For dispensers not used for analytical measurements, a label is applied to the device stating that it is not calibrated.	Monthly	± 2% Calculate accuracy by dividing weight by stated volume times 100 for percent.	Adjust. Replace.
Glass Microliter Syringes	Syringes greater than 25 ul require calibration	Semi-annually	± 2%	Replace.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 21-33 of 21-33

Table 21-3: Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Conductivity Meter	Cell impedance calibrated with three KCI standards.	Each use.	r ≥ 0.99	Recalibrate.
Deionized Water	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Weekly	<10 µmhos/cm <sup>2</sup>	Record on log. Report discrepancies to Technical Manager.
Barometer	Barometric pressure checked against 17025 standards	Yearly	±5 mBars	Replace.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 22-1 of 22-4

#### **SECTION 22**

#### MEASUREMENT TRACEABILITY (NELAC 5.5.6)

## 22.1 <u>OVERVIEW</u>

Traceability of measurements must be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard are subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware should be routinely inspected for chips, acid etching or deformity. If the Class A glassware is suspect, the accuracy of the glassware must be assessed prior to use.

## 22.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement are used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

Calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty.

The uncertainty calculations of the calibration laboratory are supported by uncertainty budgets and are represented by <u>expanded</u> uncertainties typically using a coverage factor of k=2 to approximate the 95% confidence level. This explanation accompanies the measurement result and the associated uncertainty.

The tolerance uncertainty ratio (TUR) is calculated using the expanded uncertainty of the measurement, not the collective uncertainty of the measurement standards. A statement to this effect accompanies the TUR along with the coverage factor and confidence level.

The calibration report or certificate submitted to TestAmerica Nashville contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis

#### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 22-2 of 22-4

upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

The calibration laboratory supports in-house calibration systems: documented procedures for in-house calibrations, evidence by a report, certificate, or sticker, for an appropriate amount of time; training records of calibration personnel; certificates from accreditation services demonstrating traceability to national or international standards of measurement; procedures for evaluating measurement uncertainty; timely and documented recalibration of reference standards. When subcontracting to a calibration laboratory, TestAmerica Nashville does not use a firm who subcontracts the work.

## 22.3 **REFERENCE STANDARDS / MATERIALS**

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, NIST with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a description of the standard, a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to Table 9-1 in Section 9 for general storage requirements, and to method SOPs "Standards and Reagents" section and SOP Standards Control / NV08-214 for additional storage information. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

## 22.4 <u>DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND</u> <u>REFERENCE MATERIALS</u>

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in three-ring binders in the analytical departments of the laboratory. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and SOP Standard Control / NV08-214.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction must be made to concentrations applied to solutions prepared from the stock commercial material.

**22.4.1** All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte

• Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date, and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**22.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from LIMS)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Special Health/Safety warnings if applicable. See EH&S manual for further information.

**22.4.3** In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

To maintain traceability, standard ID numbers must be noted on all associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as outlined in lab Method SOPs; and 3) according to SOP Standard Control / SA08-214 and Table 9-2.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 23-1 of 23-4

#### **SECTION 23**

#### SAMPLING (NELAC 5.5.7)

# 23.1 <u>OVERVIEW</u>

TestAmerica Nashville does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

# 23.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

# 23.2.1 <u>Preservatives</u>

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid Reagent ACS (Certified VOA Free) or equivalent
- Methanol Purge and Trap grade
- Nitric Acid Instra-Analyzed or equivalent
- Sodium Bisulfate ACS Grade or equivalent
- Sodium Hydroxide Instra-Analyzed or equivalent
- Sulfuric Acid Instra-Analyzed or equivalent
- Sodium Thiosulfate ACS Grade or equivalent

## 23.2.2 Preparing Container Orders

When new containers arrive at the laboratory, the date of receipt is recorded on the packing list received with them for retained documentation. Periodically, containers are evaluated for cleanliness based upon their intended parameter sample analysis. Upon request, the containers are then sent to clients for use in collecting samples. The shipping date, type and number of containers are maintained on file by the lab. Shipping personnel insure that container stock is rotated so that "first in" is "first out." When a client requests containers, a client services representative creates a container request in LIMS; it is then stored permanently in LIMS with a unique container order number. Copies of the container request are printed for the shipping department.

The laboratory also provides EnCore, TerraCore or other soil sampling devices when requested.

If containers are provided directly to the client from the manufacturer or from other sources, the laboratory will not be responsible for any of the above records.

# 23.3 FIELD QUALITY CONTROL (QC)

Common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the site specific Quality Assurance Project Plan (QAPP) or by the client. TestAmerica provides trip blanks for VOC analysis with the sample containers for all volatile organic analyses. Blanks generated in the field are analyzed along with the field samples.

**23.3.1** Equipment Blank / Rinsate Blank - The equipment blank, sometimes referred to as a rinsate blank, is a sample of the water used to decontaminate sampling equipment. The source water must be as free of target analytes as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and are also affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank begins when the equipment is rinsed and the water is collected.

**23.3.2** <u>Field Blank</u> - The field blank is water that is as free of target analytes as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination. The sampling time for the field blank is when the blank is prepared in the field.

**23.3.3** <u>Trip Blank</u> - The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers. They are filled with organic-free water (the source of the organic free water is the same source of water used to prepare volatile standards, method blanks, LCS and sample dilutions), sealed and taken into the field with the empty containers which are to be used for sampling. Alternatively, they may be purchased as certified organic-free for targets analyzed. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method. Unless otherwise specified, the sampling time for the trip blank is the time of receipt at the laboratory (When the "Trip" ends).

**23.3.4** <u>Field Duplicates</u> - Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

# 23.4 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e. g., 14 days, 28 days), the holding time is based on calendar day measured. Holding

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 23-3 of 23-4

times expressed in "hours" (e. g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine all holding times based on the date and specific time of sampling or to the hour, versus "days" approach.

**23.4.1** <u>Semi-Volatile</u> - Holding times for sample preparation for semi-volatile organics are measured from the sampling date (and time where applicable) until the day (and time where applicable) solvent contacts the sample. If a sample is to be extracted on the day of expiration, the actual time of extraction must be recorded on the sample preparation worksheet. Holding times for analysis are measured from the date (and time where applicable) of initiation of extraction to the time of injection into the gas chromatograph.

**23.4.2** <u>Volatiles</u> - Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date and time of injection into the gas chromatograph. The time of initiation of purging is considered the injection time, but data systems record the start of the chromatographic run rather than the start of purging. Hence, if a sample is so near expiration that the start-of-purging time rather than the chromatographic run time is needed to document the integrity of the sample; the analyst must observe and record the start-of-purging time in the instrument log. Extractions, e. g., for high level soils, must be completed in time to allow for analysis to be initiated within the maximum allowable holding time.

**23.4.3** <u>Inorganics</u> - For inorganic and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date (and time where applicable).

# 23.5 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports must be qualified using a flag, footnote or case narrative. "Analyze immediately" is an EPA designation reserved for tests which, for compliance monitoring projects, are performed by field instrumentation or a laboratory "generally within 15 minutes" of sampling (Federal Register, Vol. 48, No. 209, p. 11). TestAmerica qualifies data for these parameters if analysis cannot be performed within 15 minutes of sampling. "As Soon As Possible" or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

# 23.6 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Each sample is handled by analysts as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 23-4 of 23-4

Refer to SOP Sample Subsampling, Homogenization and Compositing / NV-08-229 for specific details on taking sample aliquots and subsampling.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 24-1 of 24-11

#### **SECTION 24**

# HANDLING OF SAMPLES (NELAC 5.5.8)

Sample management procedures at TestAmerica Nashville ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

# 24.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 24-1.

## 24.1.1 <u>Field Documentation</u>

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 24-1). This form includes information such as:

- Client name, address, phone number
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g., quote number) if available
- The date and time that each person relinquished or received the sample(s), including their signed name.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 24-2 of 24-11

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

**Note:** Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in Sample Control by date; it lists all receipts each date.

# 24.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, Sample Control completes the custody seal (Figure 24-2), retains the shipping record with the COC, and initiates an internal COC (Figure 24-3) for laboratory use by analysts and a sample disposal record (Figure 24-4).

# 24.2 <u>SAMPLE RECEIPT</u>

Samples are received at the laboratory by designated sample receiving personnel, and a unique laboratory project identification number is assigned. Each sample container is assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections and in full detail in SOP Sample Control / NV02-01.

# 24.2.1 <u>Laboratory Receipt</u>

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Cooler Receipt Form, Figure 24-5.

## 24.2.1.1 Inspection of samples include a check for:

- Complete documentation to include sample identification, location, date and time of collection, collector's name, preservation type, sample type and any additional comments concerning the samples.
- Complete sample labels to include unique identification in indelible ink.
- Use of appropriate sample containers (see Section 23)
- Adherence to holding times as specified in the test method and/or summarized in Section 23.
- Adequate sample volume for required analyses (see Section 23).

- Damage or signs of contamination to sample container. Volatile vials are also inspected for headspace
- **24.2.1.2** Check and record the temperature of the samples that require thermal preservation.
  - Samples are deemed acceptable if upon arrival they are not frozen (excludes voas) and are less than or equal to 6.0°C. Samples that are hand-delivered immediately after collection may not be at the required temperatures; however, if there is evidence that the chilling process has begun, such as the arrival on ice, the samples are considered acceptable. This must be documented on the COC.
  - If the samples were shipped in ice and solid ice is still present and in direct contact with samples, report the samples as "received on ice." Direct contact means samples must be surrounded by ice cubes or crushed ice. Ice present in a plastic bottle or other container does not constitute direct contact. Samples shipped with only "blue ice" may not be reported as "received on ice."
- **24.2.1.3** Verify sample preservation as specified in the test method. Check for correct pH as specified in the test method. The results are documented on the Cooler Receipt Form and in LIMS. In the case of volatiles, it is recorded after analysis on the run log and benchsheet. Chlorine is checked on samples requiring extractable organics, BOD, TOX, cyanide, fluoride, ammonia, TKN, CBOD and Nitrate; presence or absence is recorded.
- **24.2.1.4** After inspecting the samples, the sample control receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions on the Cooler Receipt Form and store them in appropriate refrigerators or storage locations.
- **24.2.1.5** If samples are received without a COC, TestAmerica provides a generic COC form to be completed by the client when the samples are brought to the laboratory. The client is always provided with a copy of the completed COC form for their records.
- **24.2.1.6** If analyses with short holding times are requested, the dates and times are inspected to ensure that holding times have not already expired.
- **24.2.1.7** Samples received after normal working hours are left in their coolers and placed in the walk-in cooler. The person receiving the samples must record the date and time received, the presence or absence of ice and custody seals, the temperature of samples, presence and type of packing material, and initials.
- **24.2.1.8** Any deviations from the checks described in Section 24.2.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required are documented using the PIPE non-conformance database (see Section 13) and are resolved by consultation with the client. If the sample acceptance criteria (Section 24.3) are not met, the laboratory must either:
  - Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

• Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

**24.2.1.9** The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

## 24.2.2 <u>Sample Control</u>

All samples that are received by the laboratory are logged into the LIMS to allow the laboratory to track and evaluate sample progress. Each group of samples that are logged in together (typically one project from a given client/sampling event) is assigned a unique job number. Within each job, each sampling point (or sample) receives a unique number. Sample numbers are generated sequentially over time, and are not re-assigned. A sample may be composed of more than one bottle since different preservatives may be required to perform all analyses requested. Even if multiple containers are received for a single sample, each container is uniquely identified with an alphabetic letter added to the sample number. The LIMS generates sample labels that are attached to each bottle for a given sample.

Each job/set of samples is logged into LIMS with a minimum of the following information:

- Client Name, Project Name, Address, Phone, Fax, Report to information, invoice to information (most of this information is "default information" that is stored in the LIMS).
- Date and time sampled;
- Date and time received;
- Job and/or project description, sample description;
- State or country of origin of sample. (Also, see Appendix 13, A13.7.1, for AIHA guidelines.)
- Sample matrix, special sample remarks;
- Reporting requirements (i.e., QC level, report format, invoicing format);
- Turn-around-time requirements;
- Parameters (methods and reporting limits or MDLs are default information for a given parameter)

Complete details are described in SOP Sample Control / NV02-01.

#### 24.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy that clearly outlines the circumstances under which samples are accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis and necessary QC;

- samples must be preserved according to the requirements of the requested analytical method;
- sample holding times must be adhered to;
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- sample container must be received in good condition;
- the project manager is notified if any sample fails to meet the criteria listed in the sample acceptance policy.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided with all laboratory-supplied container shipments.

# 24.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators or freezers, as required. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed, at minimum, every two weeks. Samples are not stored in refrigeration units containing standards or reagents. Samples are also stored away from food and other potentially contaminating sources. Samples must not be stored in the refrigerator compartment of a unit that has standards stored in the freezer compartment.

After all analyses are complete, samples are placed into sample storage for at least 60 days. This area is at room temperature. At the end of approximately 60 days, they are disposed of in accordance with SOP Sample Disposal / NV10-83.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

# 24.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in designated, isolated areas. Any sample that is known to be hazardous at the time of receipt or after completion of analysis is placed in one of two refrigerators designated for highly contaminated samples. Foreign and U. S. soils, requiring separate storage (as specified in SOP USDA Disposal / NV10-162) due to potential contamination with foreign organisms, are tagged with an orange sticker, and placed in specified locations. Foreign soils are heat-treated prior to disposal.

## 24.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 24-6 of 24-11

transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses, a trip blank is enclosed when required by method specifications or state or regulatory programs. The chain-of-custody form is signed by the laboratory and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

#### 24.7 <u>SAMPLE DISPOSAL</u>

Samples are retained for a minimum of 30 days after the project report is sent; however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e. g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement.

Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP Waste Disposal / NV10-83). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested.

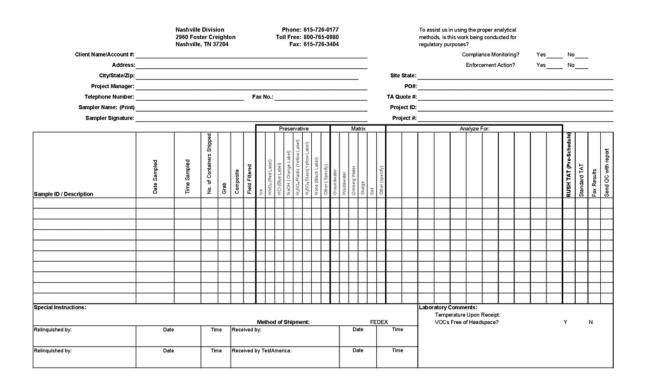
If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory removes or defaces sample labels prior to disposal, unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record (Figure 24-4) must be completed.

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to this document and SOP Waste Disposal / NV10-83.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 24-7 of 24-11

#### Figure 24-1.

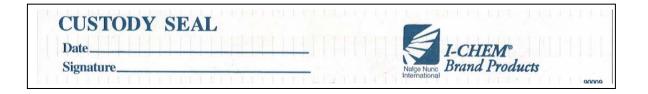
#### Example: Chain of Custody (COC)



Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 24-8 of 24-11

Figure 24-2.

Example: Custody Seal



Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 24-9 of 24-11

Figure 24-3.

Example: Internal Chain of Custody (COC)

#### VOLATILES

Client ID	Lab Number
Influent	03-A114050
Midfluent	03-A114051
MID-BAC 1	03-A114052
MID-GAC 2	03-A114053
MID-GAC3	03-A114054
Effluent	03-A114055

Relinguished by	Received by	<u>Reason</u>	<u>Date</u>	<u>Time</u>

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 24-10 of 24-11

Sample #	Shelf	Bottle	Day In	Day Out	Name	Dispose
						-

# Figure 24-4. Example: Sample Disposal Record

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 24-11 of 24-11

Figure 24-5. Cooler Receipt Form	
Cooler Received/Opened On	
1. Tracking #(last 4 digits, FedEx)	
Courier: IR Gun ID	
2. Temperature of rep. sample or temp blank when opened: Degrees Celsius	
3. If Item #2 temperature is $0^{\circ}$ C or less, was the representative sample or temp blank frozen?	YES NONA
4. Were custody seals on outside of cooler?	YESNONA
If yes, how many and where:	
5. Were the seals intact, signed, and dated correctly?	YESNONA
6. Were custody papers inside cooler?	YESNONA
I certify that I opened the cooler and answered questions 1-6 (intial)	
7. Were custody seals on containers: YES NO and Intac	t YESNONA
Were these signed and dated correctly?	YESNONA
8. Packing mat'l used? Bubblewrap Plastic bag Peanuts Vermiculite Foam Insert Paper	Other None
9. Cooling process: Ice Ice-pack Ice (direct contact) Dry ice Other	None
10. Did all containers arrive in good condition (unbroken)?	YESNONA
11. Were all container labels complete (#, date, signed, pres., etc)?	YESNONA
12. Did all container labels and tags agree with custody papers?	YESNONA
13a. Were VOA vials received?	YESNONA
b. Was there any observable headspace present in any VOA vial?	YESNONA
14. Was there a Trip Blank in this cooler? YESNONA If multiple coolers, sequence	e #
I certify that I unloaded the cooler and answered questions 7-14 (intial)	
15a. On pres'd bottles, did pH test strips suggest preservation reached the correct pH level?	YESNONA
b. Did the bottle labels indicate that the correct preservatives were used	YESNONA
16. Was residual chlorine present?	YESNONA
I certify that I checked for chlorine and pH as per SOP and answered questions 15-16 (intial)	
17. Were custody papers properly filled out (ink, signed, etc)?	YESNONA
18. Did you sign the custody papers in the appropriate place?	YESNONA
19. Were correct containers used for the analysis requested?	YESNONA
20. Was sufficient amount of sample sent in each container?	YESNONA
I certify that I entered this project into LIMS and answered questions 17-20 (intial)	
I certify that I attached a label with the unique LIMS number to each container (intial)	
21. Were there Non-Conformance issues at login? YESNO Was a PIPE generated? YESN	lO#

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 25-1 of 25-8

#### **SECTION 25**

# ASSURING THE QUALITY OF TEST RESULTS (NELAC 5.5.9)

## 25.1 <u>OVERVIEW</u>

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 21, but also by routine process quality control measurements (e. g., Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

# 25.2 <u>CONTROLS</u>

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

## 25.3 <u>NEGATIVE CONTROLS</u>

**25.3.1** <u>Method Blanks</u> are used to assess preparation and analysis for possible contamination during the preparation and processing steps.

- **25.3.1.1** The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e. g., Reagent water, Ottawa sand, glass beads, Teflon chips, etc.) and is processed along with and under the same conditions as the associated samples.
- **25.3.1.2** The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
- **25.3.1.3** The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
  - Evaluation criteria and corrective action for method blanks is defined in the specific standard operating procedure for each analysis.

**25.3.2** <u>Calibration Blanks</u> are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

These blanks may be termed Initial Calibration Blanks (ICB) or Continuing Calibration Blanks (CCB).

**25.3.3 Instrument Blanks** are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content (i.e. CCB).

**25.3.4 <u>Trip Blanks</u>** are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.

**25.3.5** <u>Field Blanks</u> are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

**25.3.6** <u>Equipment Blanks</u> are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

**25.3.7** <u>Holding Blanks</u>, also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory. (Refer to section 24.4 and SOP Refrigerator and Freezer Holding Blanks for Volatile Sample Storage Units / NV08-224).

**25.3.8** Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. Blanks are not knowingly selected for matrix QC, as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID provides information to identify the different blanks with labels such as "FB", "EB", or "TB".

# 25.4 **POSITIVE CONTROLS**

Control samples (e. g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluate field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

## 25.4.1 <u>Method Performance Control - Laboratory Control Sample (LCS)</u>

- **25.4.1.1** The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.
- **25.4.1.2** The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, Teflon Chips, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), an initial calibration verification standard is reported as the LCS, as long as the more stringent criteria is met.
- **25.4.1.3** Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e. g., solid matrix LCS for metals, TDS, etc.).
- **25.4.1.4** The LCS is made from a different source from the calibration standard source.
- **25.4.1.5** As stated in the opening of this section, the LCS goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
- **25.4.1.6** The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.
- **25.4.1.7** If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory spikes all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in certain cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components of each spiking mix must represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory must also ensure that all reported components are used in the spike mixture within a two-year time period.
  - **25.4.1.7.1** For methods that have 1-10 target analytes, spike all components.

- **25.4.1.7.2** For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- **25.4.1.7.3** For methods with more than 20 target analytes, spike at least 16 components.
- **25.4.1.7.4** Exception: Due to analyte incompatibility in pesticides, toxaphene and chlordane are only spiked at client request based on specific project needs.
- **25.4.1.7.5** Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.
- **25.4.1.8** <u>Accuracy Calculation</u>: Percent Recovery (%R) calculation (applies to LCS, CCV, Surrogates, and Matrix Spikes.

$$\% R = \frac{AV}{TV} \times 100$$

where: AV = Analyzed Value TV = True Value

## 25.5 SAMPLE MATRIX CONTROLS

## 25.5.1 <u>Matrix Spikes (MS)</u>

- **25.5.1.1** The Matrix Spike is used to assess the effect that sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used.
- **25.5.1.2** An MS is essentially a sample fortified with a known amount of the test analyte(s). At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.
- **25.5.1.3** If the mandated or requested test method does not specify the spiking components, the laboratory spikes all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in certain cases where the components interfere with accurate assessment (such as, simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number of the listed components (see LCS analytes 25.4.1.6 above) are used to control the test method. The selected components of each spiking mix must represent all chemistries, elution patterns and masses, permit-specified analytes and other client requested components. However, the laboratory must also ensure that all reported components are used in the spike mixture within a two-year time period.

**25.5.1.4** The percent recovery calculation for matrix spikes is essentially the same as the calculation shown in 25.4.1.8 except that:

AV = Sp - Sa

where: Sp = Spike result Sa = Sample result

## 25.5.2 <u>Surrogate Spikes</u>

- **25.5.2.1** Surrogate Spikes are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
- **25.5.2.2** Surrogate compounds are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method (also refer to Section 25.5). Poor surrogate recovery may indicate a problem with sample composition and must be reported, with data qualifiers, to the client whose sample produced poor recovery. See specific method SOPs and Section 13 for the discussion of corrective actions.

## 25.5.3 <u>Duplicates</u>

- **25.5.3.1** For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure. Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed, both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.
- 25.5.3.2 <u>Precision Calculation</u> (Relative Percent Difference RPD)

$$RPD = \frac{|S-D|}{(S+D)} \times 100$$

where: S=Sample Concentration D=Duplicate Concentration

**25.5.3.2.1** When calculating precision for NIOSH methods, use the approach referenced in Appendix A13.8 of the QA manual.

## 25.5.4 Internal Standards

**25.5.4.1** When required in organic methods, internal standards are spiked into all environmental and quality control samples (including the initial calibration standards). An internal standard is also used with some metals analyses. It is added to sample extracts after the extraction (post-prep). Consult the method-specific SOPs for details on the internal standard compounds, calculations and acceptance criteria.

## 25.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

**25.6.1** As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits or regulatory mandated control limits. When this occurs, the regulatory or project limits supersede the laboratory's in-house limits.

**Note:** For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

**25.6.2** Once control limits have been established, they are verified, reviewed, and updated, if necessary, on an annual basis unless the method requires more frequent updating (e. g., EPA SW846 8000 series methods). Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

- **25.6.2.1** The lab is to consider the effects of the spiking concentration control limits, and to avoid censoring of data. The acceptance criteria for recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling data to generate control limits.
- **25.6.2.2** Not only are the results to all be from a similar matrix, but the spiking levels are also approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results must all be generated using the same set of extraction, cleanup and analysis techniques. For example, results from solid samples extracted by ultrasonic extraction are not mixed with those extracted by Soxhlet.
- **25.6.2.3** The laboratory avoids discarding data that do not meet a preconceived notion of acceptable performance. This results in a censored data set, which, when used to develop acceptance criteria, leads to unrealistically narrow criteria. For a 99% confidence interval, 1 out of every 100 observations likely will still fall outside the limits. For methods with long analyte lists this may mean occasional failures every batch or two. While professional judgment is important in evaluating data to be used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. However, data points are discarded if they were the result of human or mechanical error or sample concentration exceeded spike level by > 4x. (Right clicking on the control chart and selecting View Data from the drop down menu allows viewing a table of all the charted points with any

qualifiers. This assists in determining if any points are to be discarded prior to limit generation.)

**25.6.3** Laboratory generated % Recovery acceptance (control) limits are generally established by taking  $\pm$  3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred). The system defaults to collecting the previous three months' data. This time frame is shortened if there are more than 200 points since the system slows down tremendously. The time frame is extended if there are not 20-30 points].

- **25.6.3.1** Regardless of the calculated limit, the limit is no tighter than the Calibration Verification (ICV/CCV) (unless the analytical method specifies a tighter limit).
- **25.6.3.2** The lowest acceptable recovery limit is 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- **25.6.3.3** The maximum acceptable recovery limit is 150%.
- **25.6.3.4** The maximum acceptable RPD limit is 50%. The minimum RPD limit is 10%.
- **25.6.3.5** If either the high or low end of the control limit changes by  $\leq$  5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.
- **25.6.3.6** The QA department generates a Control Limits Manual that contains tables summarizing the precision and accuracy acceptability limits for analyses performed at TestAmerica Nashville. The Control Limits Manual is considered to be part of the QA Manual and therefore carries the same revision number as the current QA Manual. Letter suffixes are added to the revision number if it becomes necessary to update the Control Limits Manual during the course of the year prior to a QA manual update. This manual includes an effective date, is updated each time new limits are generated and is located in each analytical department and the QA office. Unless otherwise noted, limits within these tables are laboratory generated. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Manager and QA Manager) and entered into the LIMS. The Quality Assurance department maintains an archive of all limits used within the laboratory, as described in SOP Archiving / NV08-194. Archived limits are available upon request.
- **25.6.3.7** Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

**25.6.4** A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and are

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 25-8 of 25-8

reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if the analyte results are below the reporting limit and the LCS is above the upper control limit. See specific method SOPs for complete discussion of acceptance criteria and corrective action.

**25.6.5** If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 13.

**25.6.6** If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. Dilutions or cleanup procedures may be required when obvious chromatographic interference is present. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). See specific method SOPs for a more detailed discussion of acceptance criteria and corrective action.

# 25.7 <u>METHOD DETECTION LIMITS (MDLS)</u>

MDLs, calculated as described in Section 20.7, are updated or verified annually, or more often if required by the method. Once values are approved, they are distributed to the analysts, entered in LIMS analyte by analyte, and tabulated in the Control Limits Manual.

# 25.8 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

**25.8.1** The laboratory has written and approved method SOPs procedures to assure the accuracy of the test method including calibration (see Section 21), use of certified reference materials (see Section 22) and use of PT samples (see Section 16).

**25.8.2** A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 20.

**25.8.3** Use of formulae to reduce data is discussed in the method standard operating procedures and in Section 21.

- **25.8.4** Selection of appropriate reagents and standards is included in Section 9 and 22.
- **25.8.5** A discussion on selectivity of the test is included in Section 5.
- **25.8.6** Constant and consistent test conditions are discussed in Section 19.
- **25.8.7** The laboratories sample acceptance policy is included in Section 24.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 26-1 of 26-8

#### **SECTION 26**

## REPORTING RESULTS (NELAC 5.5.10)

## 26.1 <u>OVERVIEW</u>

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory must work with the client during project set up to develop an acceptable solution. A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there must be a reference to a full report that is made available to the client.

Review of reported data is included in Section 20.

## 26.2 <u>TEST REPORTS</u>

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report must contain the following information:

**26.2.1** A report title (e. g., Analytical Report for Samples) with a "sample results" column header.

**26.2.2** Each report page printed on company letterhead which includes the laboratory name, address and telephone number.

**26.2.3** A unique identification of the report (e. g., work order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of report are represented as page # of ##, where the first number is the page number and the second is the total number of pages. Pages are automatically paginated by LIMS when the report is created.

**26.2.4** A copy of the chain of custody (COC).

• Any COCs involved with subcontracting are included.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 26-2 of 26-8

- There is a statement on the front of the report that says, "The Chain of Custody, X page(s), is included and is an integral part of this report." The number of pages of the CoC (X) is calculated automatically by the number of pages that are scanned in the CoC folder. Any pages scanned with the CoC (i. e., non-conformances, emails, etc.) are included in the page count.
- Any additional addenda to the report must be treated in a similar fashion, so it is a recognizable part of the report and cannot accidentally get separated from the report (e. g., sampling information).

26.2.5 The name and address of client and a project name/number, if applicable.

**26.2.6** Client project manager or other contact

**26.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.

**26.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours. The analysis time is always included on the report. The preparation date and time are included for those methods that require a separate preparation.

- **26.2.9** Date reported or date of revision, if applicable.
- **26.2.10** Method of analysis including method code (EPA, Standard Methods, etc).
- **26.2.11** Reporting limit.
- 26.2.12 Method detection limits (if requested)
- **26.2.13** Definition of data qualifiers and reporting acronyms (e. g., ND).

26.2.14 Sample results.

**26.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

**26.2.16** Condition of samples at receipt including temperature (included on the Cooler Receipt Form, which is part of the CoC).

**26.2.17** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

**26.2.18** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory.

**26.2.19** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director. For applying an

electronic signature, refer to the Electronic Signature Policy (Section 26.4) and the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002).

**26.2.20** When NELAC accreditation is required, the lab certifies that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not. Accreditations/certifications are included near the end of the report, listed by method.

**26.2.21** The laboratory includes a cover letter.

**26.2.22** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

**26.2.23** When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

**26.2.24** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**26.2.25** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e. g., partial report, draft report). A complete report must be sent once all of the work has been completed.

**26.2.26** Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

**26.2.27** Reports for Ohio VAP work require a VAP affidavit be completed and included with the report.

## 26.3 <u>REPORTING LEVEL OR REPORT TYPE</u>

TestAmerica Nashville offers three levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level II is a report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Procedures used to ensure client confidentiality are outlined in Section 26.7.

## 26.3.1 <u>Electronic Data Deliverables (EDDs)</u>

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Nashville offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), EXCEL, dBASE, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs are subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

## 26.4 ELECTRONIC REPORTING AND SIGNATURE POLICY

Following the lead of the Federal Paperwork Reduction Act, TestAmerica has implemented policies and procedures to help reduce paper usage. One of these procedures is to generate final reports and provide them to clients in pdf format.

Laboratory Director appointed representatives may approve final reports using an electronic signature that is applied to the report at the time of generation. This policy is prepared to state that the electronically applied signatures on TestAmerica Nashville reports are as legally binding as a handwritten "wet signature." This policy is intended to prevent the possibility of non-repudiation (denial that an individual signed the document) and to insure authenticity and security. In order to insure the electronic signatures are valid and unequivocally represent the identity of the signer, TestAmerica uses 21 CFR Part 11 "Electronic Records; Electronic Signatures" from the FDA as well as EPA's procurement policy (EPS 00-01) as guidance documents for this policy.

In order to ensure authenticity of the reports, the following conditions must be met:

## 26.4.1 <u>Report Content</u>

- State that the report was electronically signed.
- The printed name and title of the signer must be underneath the signature.
- The date and time when the signature was executed is represented in the "Report Issued" entry on the cover page of the report.
- The meaning of the signature: (e. g., reviewed and approved).

In order to insure the integrity of the signatures, the following security features have been implemented.

## 26.4.2 <u>General Requirements</u>

- The identity of the signatory must be verified before an electronic signature can be created for that person.
- Each electronic signature must be unique to a single individual and must not be reused by or assigned to another individual
- Persons using an electronic signature must certify that the electronic signatures in the system are intended to be the legally binding equivalent to their traditional handwritten signature. On this certification, the signatory states that their passwords are to remain completely confidential and can only be used by the genuine owner of the password and the sign-off does not take place until each page has been viewed.

## 26.4.3 <u>Components and Controls</u>

Two distinct identification components are utilized for each individual. The components are a) user name b) password. Each signing requires that the entry of the username and the password be reentered. The signatures are not copied, excised or transferred from the report by ordinary means.

The report is not changed once the signature has been applied and the pdf files are stored on the file server with security as well as password protected to ensure no changes are made to the file.

"pdf" reports must be backed up on a magnetic tape or other durable storage media (e. g., DVD) and maintained secure for up to 10 years.

## 26.5 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report. Refer to Appendix 7 for a list of the laboratory's standard footnotes and qualifiers.

**26.5.1** Numeric results with values outside of the calibration range, either high or low are qualified as "estimated."

**26.5.2** Where quality system requirements are not met, a statement of compliance/noncompliance with requirements and/or specifications, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

**26.5.3** Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

**26.5.4** Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director must determine if a response can be prepared. If so, the Laboratory Director designates the appropriate member of the management team to prepare a response. The response must be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, and a comment must be added suggesting that the client verify the opinion or interpretation with their regulator.

## 26.6 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If TestAmerica Nashville is not able to provide the client the requested analysis, the samples are subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

# 26.7 <u>CLIENT CONFIDENTIALITY</u>

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica must not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information <u>known</u> to be potentially endangering to national security or an entity's proprietary rights must not be released.

**Note:** This does not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica does, to the extent feasible, provide reasonable notice to the client before disclosing the information.

**Note:** Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

**26.7.1** Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Page 26-7 of 26-8

recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

## 26.8 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

## 26.9 <u>AMENDMENTS TO TEST REPORTS</u>

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 13).

The revised report is retained on the Archive data server, as is the original report. When the pdf of a report is created, the pdf name has the work order number and the date and time that the pdf was created. The original report has the earliest date, and any revisions have later dates and times.

When the report is re-issued, a notation that the report has been revised is placed on the cover/signature page of the report along with a brief explanation of reason for the re-issue and a reference back to the last final report generated. For Example: *Report was revised on 11/3/07 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/07 at 10:47am.* 

## 26.10 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

## 26.10.1 <u>Sample Reanalysis Policy</u>

Because there is a certain level of uncertainty with any analytical measurement a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g. sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory reanalyzes samples at a client's request with the following caveats.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ±1 reporting limit for samples < 5x the reporting limit, the original analysis is reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory investigates the discrepancy and reanalyzes the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client is typically charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to nonhomogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Supervisor, Technical Manager, QA Manager, or Laboratory Director if unsure.

## 26.10.2 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e. g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely <u>no possible</u> impact on the interpretation of the analytical results and there is <u>no possibility</u> of the change being interpreted as misrepresentation by anyone inside or outside of our company.

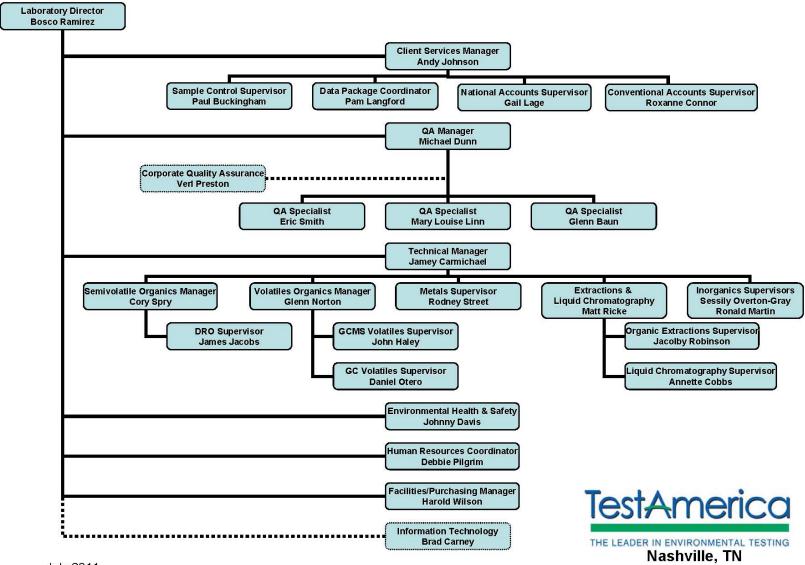
## 26.10.3 <u>Multiple Reports</u>

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by the lab QA Manager.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 1 Page 1 of 1

(Reserved)

## Laboratory Organization Chart



July 2011

## Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 3 Page 1 of 1

## Laboratory Floor Plan



Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 4 Page 1 of 1

(Reserved)

## Glossary/Acronyms

## Glossary:

Acceptance Criteria:

Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

## Accreditation:

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

## Accrediting Authority:

The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

## Accuracy:

The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

## Aliquot

A measured portion of a sample used for analysis.

## Analyst:

The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

## Batch:

Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

## Blank:

A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample:

A 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.

#### Calibration:

To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard must bracket the range of planned or expected sample measurements. (NELAC)

#### Calibration Curve:

The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method:

A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:

A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):

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. (ISO Guide 30–2.2)

## Chain of Custody:

An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:

The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):

The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

Compromised Samples:

Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

### Confidential Business Information (CBI):

Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its

representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:

Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation Alternate wavelength Derivatization Mass spectral interpretation Alternative detectors or Additional Cleanup procedures

(NELAC)

Conformance:

An affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Continuing Calibration Blank: solvent blank following CCV.

Continuing Calibration Verification: A standard from the calibration curve that is used to verify that the instrument remains properly calibrated throughout the analytical run.

Corrective Action:

The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

<u>Correction</u>: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst is most frequently the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

Data Audit:

A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction:

The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency:

An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

#### Detection Limit:

The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

#### Document Control:

The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

#### Duplicate Analyses:

The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

#### Equipment Blank:

Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

#### External Standard Calibration:

Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Federal Water Pollution Control Act (Clean Water Act, CWA):

The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

#### Field Blank:

Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

#### Field of Testing:

NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

#### Finding:

An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)

Holding Times (Maximum Allowable Holding Times):

The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Initial Calibration Blank: Solvent blank immediately following ICV.

Initial Calibration Verification:

A standard from a second source that is analyzed to verify the calibration curve.

Internal Standard:

A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration:

Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:

A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Response:

Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

Laboratory:

A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):

A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS must be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples are used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:

Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1<sup>st</sup> Order Curve):

The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value

of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):

An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Matrix:

The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be are to be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples must be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: 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. (NELAC)

Matrix Spike (spiked sample or fortified 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 spikes are performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) are to be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and is reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):

A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates are analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory documents their procedure to select the use of an appropriate type of duplicate. The selected sample(s) are to be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and are reported to the client whose sample was used for the duplicate. (QAMS)

#### Method Blank:

A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

#### Method Detection Limit:

The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Method Reporting Limit:

The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD.

#### Negative Control:

Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

#### Performance Audit:

The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

#### Performance Based Measurement System (PBMS):

A set of processes 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. (NELAC)

#### Positive Control:

Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision:

The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

#### Preservation:

Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

#### Proficiency Testing:

A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

## Proficiency Testing Program:

The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

#### Proficiency Test Sample (PT):

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. (QAMS)

#### Quality Assurance:

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. (QAMS)

### Quality Assurance [Project] Plan (QAPP):

A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

#### Quality Control:

The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

## Quality Control Sample:

An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

#### Quality Manual:

A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

#### Quality System:

A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

## Quantitation Limits:

The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

#### Range:

The difference between the minimum and the maximum of a set of values. (EPA-QAD)

#### Reagent Blank (method reagent blank):

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. (QAMS)

#### Reference Material:

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. (ISO Guide 30-2.1)

#### Reference Method:

A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

#### Reference Standard:

A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

#### Replicate Analyses:

The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

#### Report Limit (RL):

The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD.

Requirement:

Denotes a mandatory specification; often designated by the term "shall". (NELAC)

#### Resource Conservation and Recovery Act (RCRA):

The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

#### Safe Drinking Water Act (SDWA):

The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

#### Sample Duplicate:

Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic): The  $2^{nd}$  order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The  $2^{nd}$  order regression generates a coefficient of determination (COD or  $r^2$ ) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes,  $r^2$  must be greater than or equal to 0.99.

### Selectivity:

(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

#### Sensitivity:

The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

#### Spike:

A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory spikes all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in certain cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components are used to control the test method. The selected components of each spiking mix must represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory must also ensure that all reported components are used in the spike mixture within a two-year time period. (NELAC)

## Standard:

The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

## Standard Operating Procedures (SOPs):

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. (QAMS)

#### Standardized Reference Material (SRM):

A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

#### Supervisor (however named):

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. (NELAC)

### Surrogate:

A substance with properties that mimic the analyte of interest, but is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and is reported to the client whose sample produced poor recovery. (QAMS)

#### Systems Audit (also Technical Systems Audit):

A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

## Technical Manager:

Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

#### Test:

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. (ISO/IEC Guide 2-12.1, amended)

#### Test Method:

An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

#### Toxic Substances Control Act (TSCA):

The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

#### Traceability:

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. (VIM-6.12)

#### Uncertainty:

A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

## Acronyms:

BS – Blank Spike BSD – Blank Spike Duplicate CAR – Corrective Action Report CCB – Continuing Calibration Blank

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 5 Page 12 of 13

CCV – Continuing Calibration Verification CF – Calibration Factor CFR – Code of Federal Regulations COC – Chain of Custody CQMP - Corporate Quality Managemetn Plan DOC – Demonstration of Capability DQO – Data Quality Objectives **DUP** - Duplicate EHS – Environment, Health and Safety EPA – Environmental Protection Agency GC - Gas Chromatography GC/MS - Gas Chromatography/Mass Spectrometry HPLC - High Performance Liquid Chromatography ICB – Initial Calibration Blank ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy ICP/MS – ICP/Mass Spectroscopy ICV – Initial Calibration Verification IDL – Instrument Detection Limit IH – Industrial Hygiene IS – Internal Standard LCS – Laboratory Control Sample LCSD – Laboratory Control Sample Duplicate LIMS – Laboratory Information Management System LOD – Limit of Detection LOQ - Limit of Quantitation MB – Method Blank MDL – Method Detection Limit MDLV – MDL Verification Check Standard MRL – Method Reporting Limit MS – Matrix Spike MSD – Matrix Spike Duplicate MSDS - Material Safety Data Sheet NELAC - National Environmental Laboratory Accreditation Conference NELAP - National Environmental Laboratory Accreditation Program PT – Performance Testing QAM – Quality Assurance Manual QA/QC – Quality Assurance / Quality Control QAPP – Quality Assurance Project Plan QL - Quantitation Limit RF – Response Factor RL – Report Limit **RPD** – Relative Percent Difference RSD – Relative Standard Deviation SD – Standard Deviation SOP: Standard Operating Procedure TAT – Turn-Around-Time **TNI - The NELAC Institute** 

VOA – Volatiles VOC – Volatile Organic Compound

## See Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and

Definitions.

#### Laboratory Certifications, Accreditations, Validations

TestAmerica Nashville maintains accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Lab Number	Organization	Lab Number
A2LA	0453-07	Mississippi	none
AIHA	100790	Montana	none
Alabama		Nevada	TN00032
Alaska	UST-087	New Hampshire (2 <sup>nd</sup> NELAP)	2963
Arkansas	88-0737	New Jersey (2 <sup>nd</sup> NELAP)	TN965
Arizona	AZ0473	New York (2 <sup>nd</sup> NELAP)	11342
CALA	3744	North Carolina	387
California	01168CA	North Dakota	R-146
Colorado	none	Oklahoma	9412
Connecticut	PH-0220	Oregon (2 <sup>nd</sup> NELAP)	TN200001
USACE	none	Pennsylvania (2 <sup>nd</sup> NELAP)	68-00585
Florida (1° NELAP)	E87358	Rhode Island	LAO00268
Illinois (2 <sup>nd</sup> NELAP)	200010	South Carolina	84009001, 002
Iowa	131	Tennessee	02008
Kansas (2 <sup>nd</sup> NELAP)	E-10229	Texas (2 <sup>nd</sup> NELAP)	T104704077
Kentucky	90038	Utah (2 <sup>nd</sup> NELAP)	6157260177
Kentucky UST	19	Virginia (2 <sup>nd</sup> NELAP)	460152
Louisiana Drinking H <sub>2</sub> O	LA080021	Virginia Drinking H <sub>2</sub> O	00323
Louisiana (2 <sup>nd</sup> NELAP)	01945	Washington	C1712
Maryland	316	West Virginia	219
Massachusetts	M-TN032	Wisconsin	998020430
Minnesota	047-999-345	Wyoming UST	A2LA 0453-07

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.

## **Claims of Accreditation Status**

TestAmerica Nashville has agreed to make only valid claims as to its accreditation/certification status by any authority by ensuring that the expiration dates are not exceeded and the method-specific scope or parameter lists are supportable, as required by each. Any false claims would be reported to that authority. The agreement covers the use of the authority's name, such as "Authority-Accredited," logo, or certificate number. The only valid proof of accreditation/certification is the current certificate and scope of the authority. It is the responsibility of the laboratory to make these documents available to all staff, and it is the staff's duty to reference only the current documents.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 6 Page 2 of 2

A report with scope and non-scope analytes may only be presented on the same report if the non-accredited results are clearly and unambiguously identified. No report with non-scope analytes may be associated with the logo, "Authority accredited" phrase, or the certificate number. Only the analytes specified by a unique method are valid within the scope. There must be no intentional misleading of the users of the laboratory's services in this regard.

No opinions and/or interpretations based on results outside the laboratory's scope may be presented on a document referenced by "Authority-accredited, the logo, or the certificate number. If these are made, they must be written in a separate letter which is not endorsed by the authority.

The "Authority-accredited" logo may only be affixed to equipment calibrated by a laboratory that is accredited by the authority. If calibration labels contain the logo, they must also show the calibration laboratory's name or its certificate number, the instrument's unique identification, the date of the last calibration, and a cross-reference to the last calibration certificate.

If the company decides to use the "Authority-accredited" logo in marketing activities, no misrepresentation may occur. Only reference to the accredited scope at a specific laboratory site is allowed. If any "Authority-accredited" language is used in proposals or quotations, any non-scope analytes must be clearly denoted as not accredited by that authority. The same is true for any use of laboratory letterhead with the "Authority-accredited" wording or logo. The logo may not be affixed to any material, item, product, part, or packaging, thereby implying accreditation status to that piece. In literature, any use of the logo must be positioned adjacent to the accredited laboratory's name and clearly state that the presence of the logo does not imply certification/approval of the products tested. At no time may the logo appear to suggest that a person is accredited. Misrepresentation of accreditation status is never allowed and must be reported if it occurs. If in doubt, the idea of the logo's use may be presented to the authority for approval.

If accreditation is terminated or suspended, the laboratory must immediately cease to use the "Authority-accredited" wording, the logo, or the certificate number reference in any way and inform clients impacted by the change.

# Data Qualifiers

<	< [Custom Value]
>	< [Custom Value]
A-01	[Custom Value]
B	Analyte was detected in the associated Method Blank.
D	
B1	Analyte was detected in the associated method blank. Analyte concentration in the sample is greater than 10x the concentration found in the method blank.
ы	Analyte detected in refrigerator/freezer holding blank. Reported value for this
B7	sample within 10x detected concentration in holding blank.
	The contamination reported in the HPLC method blank for one detector was not
BCD	confirmed by the other detector.
	Calibration Verification recovery was above the method control limit for this analyte.
С	Analyte not detected, data not impacted.
	Calibration Verification recovery was above the method control limit for this analyte,
C1	however the average % difference for all analytes met method criteria.
	Calibration Verification recovery was below the method control limit for this analyte,
C2	however the average % difference for all analytes met method criteria.
C4	Calibration Verification recovery was below the method control limit for this analyte.
	Calibration Verification recovery was above the method control limit for this analyte.
C8	A high bias may be indicated.
CF2	Confirmatory analysis was past holding time.
	The sample was originally analyzed with a positive result, however the reanalysis did
CF5	not confirm the presence of the analyte.
CF6	Results confirmed by reanalysis.
CF7	Result may be elevated due to carry over from previously analyzed sample.
CISP	The concentration indicated for this analyte is derived from a single point calibration. Recovery for this analye was within Laboratory historical limits but outside contract
CL1	required limits of 70-130 %.
CN4	Amenable cyanide results reported from total determination method.
CSTM	[Custom Value]
	Concentration exceeds the calibration range and therefore result is semi-
Е	quantitative.
	Concentration estimated. Analyte exceeded calibration range. Reanalysis not
E1	possible due to insufficient sample remaining.
	Concentration estimated. Analyte exceeded calibration range. Reanalysis not
E3	peformed due to holding time requirements.
EPH%	[Custom Value] %RPD
Н	Sample analysis performed past method-specified holding time.
	Sample analysis performed past the method-specified holding time per client's
H1	approval.
	The holding time calculation is based on a sampling time of 00:00 on the sampling
	date noted on the Chain of Custody. No sampling time was provided to the
140	laboratory.
H10	
ЦЭ	Initial analysis within holding time. Reanalysis for the required dilution or
H2	confirmation was past holding time.
H3	Sample was received and analyzed past holding time.

## Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 7 Page 2 of 6

me. Trmed Dre, S ased The nit. The mit.
ore, ased The nit. The
ore, ased The nit. The
ore, ased The nit. The
s nsed The nit. The
nit.
The nit. The
nit. The
The
nit.
mit.
ove
r
tion y.
n is
y
y
-) -)

M2	The MS and/or MSD were below the acceptance limits due to sample matrix interference. See Blank Spike (LCS).		
М3	Results exceeded the linear range in the MS/MSD and therefore are not available for reporting. The batch was accepted based on acceptable recovery in the Blank Spike (LCS).		
M4	The MS/MSD required a dilution due to matrix interference. Because of this dilution, the matrix spike concentrations in the sample were reduced to a level where the recovery calculation does not provide useful information. See Blank Spike (LCS).		
M6	Any analyte not run due to matrix		
M7	The MS and/or MSD were above the acceptance limits. See Blank Spike (LCS).		
M8	The MS and/or MSD were below the acceptance limits. See Blank Spike (LCS).		
M9	Matrix Spike recovery was high. Data Reported per ADEQ policy 0154.000		
MCP	No results were reported for the MS and/or MSD due to a clogged autosampler port. Batch was accepted based on Blank Spike (LCS) recoveries.		
MHA	Due to high levels of analyte in the sample, the MS/MSD calculation does not provide useful spike recovery information. See Blank Spike (LCS).		
MNR	No results were reported for the MS/MSD. The sample used for the MS/MSD required dilution due to the sample matrix. Because of this, the spike compounds were diluted below the detection limit.		
MNR1	There was no MS/MSD analyzed with this batch due to insufficient sample volume. See Blank Spike.		
MNR3	Insufficient sample received to meet method QC requirements.		
MNR4	Insufficient sample received to meet method QC requirements. Batch QC requirements satisfy ADEQ policies 0154.000 and 0155.000		
N1	See case narrative.		
N2	See corrective action report.		
NO	No		
NoRES	>100,000		
Р	The sample, as received, was not preserved in accordance to the referenced analytical method.		
P1	Sample received and analyzed without chemical preservation.		
P12	The method required trip blank was not provided along with this sample.		
P13	Sample volume, as received, was inappropriate to meet method specifications.		
P2	Sample received without chemical preservation, but preserved by the laboratory.		
P3	Sample was received above recommended temperature.		
P4	Sample received in inappropriate sample container.		
P6	Sample received unpreserved, however the sample was analyzed within 7 days per EPA recommendation.		
P7	Sample filtered in lab.		
P8	Sample unable to be adjusted to correct pH due to matrix.		
P9	This analyte has been shown to degrade upon preservation with HCI and cannot accurately be quantitated.		
рН	pH [Custom Value]		
pH<1	pH<1		
pH<2	pH<2		
pH>12	pH>12		
pri~iz			

pH>2	pH>2			
P-HS	Sample container contained headspace.			
PV	Acid preservation was indicated on the sample vial. However, a pH of <2 was not obtained.			
PX	Sample for VOA analysis not received in preserved VOA vials or Encore or similar sampling device.			
Q2	The chromatographic pattern is consistent with diesel fuel.			
Q3	The chromatographic pattern is not consistent with diesel fuel.			
Q5	Results in the diesel organics range are primarily due to overlap from a gasoline range product.			
QFL	Florisil clean-up (EPA 3620) performed on extract.			
QGP	Gel Permeation (EPA 3640) clean-up performed on extract.			
QP	Hydrocarbon result partly due to individual peak(s) in quantitation range.			
QP1	The primary contamination elutes between [Custom Value], which is in the motor oil range.			
QP2	The primary contamination elutes between [Custom Value], which is in the diesel fuel range.			
QP3	The primary contamination elutes between [Custom Value], which is in the kerosene range.			
QP4	The primary contamination elutes between [Custom Value], which is in the mineral spirits range.			
QP5	There was insufficient contamination present to perform a pattern match.			
QP6	The contamination did not match any standards in our library.			
QP7	The contamination is similar to our [Custom Value] standard.			
QSG	Silica Gel clean-up performed on extracts.			
QSP	Sulfuric Acid / Permanganate (EPA3665) clean-up performed on extract.			
QSU	Sulfur (EPA 3660) clean-up performed on extract.			
QU	Unquantitated hydrocarbons present in the sample outside of the reported carbon range.			
R	The RPD exceeded the method control limit. The individual analyte QA/QC recoveries, however, were within acceptance limits.			
R1	The RPD between the primary and confirmatory analysis exceeded 40%. Per method 8000B, the higher value was reported.			
R10	The RPD between the primary and confirmatory analysis exceeded 40%. Per method 8000B, the lower value was reported due to apparent chromatographic problems. The RPD between the primary and confirmatory analysis exceeded 40%. Per			
R12	method 8000C, the lower value was reported.			
R13	The RPD calculation is not applicable for results expressed as less than (<) or greater than (>).			
R2	The RPD exceeded the acceptance limit.			
R3	The RPD exceeded the acceptance limit due to sample matrix effects.			

R4	Due to the low levels of analyte in the sample, the duplicate RPD calculation does not provide useful information.
R7	LCS/LCSD RPD exceeded the acceptance limit. Recovery met acceptance criteria.
R9	Sample RPD exceeded the laboratory control limit.
RL1	Reporting limit raised due to sample matrix effects.
RL4	Reporting limit raised due to insufficient sample volume.
S10	Insufficient sample available for reanalysis.
S12	Sample highly flammable and ignited upon contact.
S6	Sediment present.
S7	Sample breakthrough to 2nd section is > 10%. Results may be biased low.
SB	Sustained burning when exposed to open flame.
SPS	Percent solids result provided by the client.
SSV	Solvent volume not present on sample vial. The lab has assumed a 1:1 ratio.
STW	No tare weight present on sample vial. Result should be considered an estimated value.
T1	Method approved by EPA, but not yet licensed by ADHS.
T14	The PAH compounds were analyzed and reported by method 8270.
T15	The method used is for screening purposes only; the result reported for this analyte is an estimate.
T16	Trace <2 mg/L.
T2	Cited ADHS licensed method does not contain this analyte as part of method compound list.
T4	The cited licensed method does not contain this analyte as part of the method compound list.
Т5	Less than the prescribed sample amount was available to perform the leachate extraction. The volume of extraction fluid was adjusted proportionately based on the method prescribed ratio of extraction fluid to sample weight.
Т6	The temperature during the 18 hour TCLP extraction exceeded the 21-25 degrees C range stated in EPA Method 1311. The temperature range during the extraction was [Custom Value] degrees C.
T7	Tentatively identified compound. Concentration is estimated based on the closest internal standard.
T8	The reported result cannot be used for compliance purposes.
TND	Compound not detected using TIC procedure. Quantitation is estimated based on closest Internal Standard. An assumption is made that the compound will purge or extract and respond chromatographically consistent with target compounds analyzed by this method.
YES	Yes
Z	Due to sample matrix effects, the surrogate recovery was below the acceptance limits.
Z1	Surrogate recovery was above acceptance limits.
<u> </u>	
Z10	Surrogate outside laboratory historical limits but within method guidelines. No effect on data.
210	Surrogate low but all targets within method criteria. No effect
Z11	on data.
Z2	Surrogate recovery was above the acceptance limits. Data not impacted.

Z3	The sample required a dilution due to the nature of the sample matrix. Because of this dilution, the surrogate spike concentration in the sample was reduced to a level where the recovery calculation does not provide useful information.
Z5	Due to sample matrix effects, the surrogate recovery was outside acceptance limits. Secondary surrogate recovery was within the acceptance limits.
Z6	Surrogate recovery was below acceptance limits.
Z7	Surrogate recovery was high. Data reported per ADEQ policy 0154.000.
Z8	Surrogate recovery was low. Data reported per ADEQ policy 0154.000.
ZE	The EPH surrogate recovery was outside QC limits due to poor fractionation. Both fractions were evaluated for all EPH carbon ranges. The sample was non-detect at the reporting limit for all carbon ranges, so the data was accepted without further analysis.
zx	Due to sample matrix effects, the surrogate recovery was outside the acceptance limits.

For Ohio VAP: The laboratory must implement corrective action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale is presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

Appendix I Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
VOLATILES			
Acetone	67-64-1		8260
Acrylonitrile	107-13-1		8260
Benzene	71-43-2	0.005	8260
Bromochloromethane; Chlorobromomethane	74-97-5	0.000	8260
Bromodichloromethane; Dichlorobromomethane	75-27-4	**	8260
Bromoform; Tribromomethane	75-25-5	**	8260
Carbon disulfide	75-15-0		8260
Carbon tetrachloride	56-23-5	0.005	8260
Chlorobenzene	108-90-7	0.1	8260
Chloroethane; Ethyl chloride	75-00-3		8260
Chloroform; Trichloro-methane	67-66-3	**	8260
Dibromochloromethane; Chlorodibromomethane	124-48-1	**	8260
1,2-Dibromo-3-chloropro-pane; DBCP	96-12-8	0.0002	8011
1,2-Dibromoethane; Ethylene dibromide; EDB	106-93-4	0.00005	8011
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	0.6	8260
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	0.075	8260
trans-1,4-Dichloro-2-butene	110-57-6		8260
1,1-Dichloroethane; Ethylidene chloride	75-34-3		8260
1,2-Dichloroethane; Ethylene dichloride	107-06-2	0.005	8260
1,1-Dichloroethylene; Vinylidene chloride; 1,1- Dichloroethene	75-35-4	0.007	8260
cis-1,2-Dichloroethylene; cis-1,2-Dichloroethene	156-59-2	0.07	8260
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	0.1	8260
1,2-Dichloropropane; Propylene dichloride	78-87-5	0.005	8260
cis-1,3-Dichloropropene	10061-01-5		8260
trans-1,3-Dichloropropene	10061-02-6		8260
Ethylbenzene	100-41-4	0.7	8260
2-Hexanone; Methyl butyl ketone	591-78-6		8260
Methyl bromide; Bromomethane	74-83-9		8260
Methyl chloride; Chloromethane	74-87-3		8260
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3		8260
Methyl iodide; lodomethane	74-88-4		8260
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1		8260
Methylene chloride; Dichloromethane	75-09-2	0.005	8260
Styrene	100-42-5	0.1	8260
1,1,1,2-Tetrachloroethane	630-20-6		8260
1,1,2,2-Tetrachloroethane	79-34-5		8260
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	0.005	8260
Toluene	108-88-3	1	8260
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	0.2	8260
1,1,2-Trichloroethane	79-00-5	0.005	8260
Trichloroethylene; Trichloroethene	79-01-6	0.005	8260

## Federal Appendix I - Constituents for Assessment Monitoring (40 CFR Part 258)

### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 8 Page 2 of 2

Appendix I Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
Trichlorofluoromethane	75-69-4		8260
1,2,3-Trichloropropane	96-18-4		8260
Vinyl acetate	108-05-4		8260
Vinyl chloride; Chloroethene	75-01-4	0.002	8260
Xylene (total)	96-47-6,	10	8260
	108-38-3,		
	106-42-3,		
	1330-20-7.		
METALS			
Antimony	7440-36-0	0.006	6010
Arsenic	7440-38-2	0.05	6010
Barium	7440-39-3	2.0	6010
Beryllium	7440-41-7	0.004	6010
Cadmium	7440-43-9	0.005	6010
Chromium	7440-47-3	0.1	6010
Cobalt	7440-48-4		6010
Copper	7440-50-8	1	6010
Lead	7439-92-1	0.015	6010
Nickel	7440-02-0	0.1	6010
Selenium	7782-49-2	0.05	6010
Silver	7440-22-4	0.1	6010
Thallium	7440-28-0	0.002	6010
Vanadium	7440-62-2		6010
Zinc	7440-66-6	5	6010

Note: Depending upon the state, additional constituents may be required.

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
VOLATILES			
Acetone	67-64-1		8260
Acetonitrile; Methyl cyanide	75-05-8		8260
Acrolein	107-02-8		8260
Acrylonitrile	107-13-1		8260
Allyl chloride	107-05-1		8260
Benzene	71-43-2	0.005	8260
Bromochloromethane; Chlorobromomethane	74-97-5		8260
Bromodichloromethane; Dichlorobromomethane	75-27-4	**	8260
Bromoform; Tribromomethane	75-25-5	**	8260
Carbon disulfide	75-15-0		8260
Carbon tetrachloride	56-23-5	0.005	8260
Chlorobenzene	108-90-7	0.1	8260
Chloroethane; Ethyl chloride	75-00-3		8260
Chloroform; Trichlorome-thane	67-66-3	**	8260
Chloroprene	126-99-8		8260
Dibromochloromethane; Chlorodibromomethane	124-48-1	**	8260
1,2-Dibromo-3-chloropro-pane; DBCP	96-12-8	0.0002	8011
1,2-Dibromoethane; Ethylene dibromide; EDB	106-93-4	0.00005	8011
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	0.6	8260
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1		8260
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	0.075	8260
trans-1,4-Dichloro-2-butene	110-57-6		8260
Dichlorodifluoromethane	75-71-8		8260
1,1-Dichloroethane; Ethylidene chloride	75-34-3		8260
1,2-Dichloroethane; Ethylene dichloride	107-06-2	0.005	8260
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	0.007	8260
cis-1,2-Dichloroethylene; cis-1,2-Dichloroethene	156-59-2	0.07	8260
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	0.1	8260
1,2-Dichloropropane; Propylene dichloride	78-87-5	0.005	8260
1,3-Dichloropropane; Trimethylene dichloride	142-28-9		8260
2,2-Dichloropropane; Isopropylidene chloride	594-20-7		8260
1,1-Dichloropropene	563-58-6		8260
cis-1,3-Dichloropropene	10061-01-5		8260
trans-1,3-Dichloropropene	10061-02-6		8260
Ethylbenzene	100-41-4	0.7	8260
Ethyl methacrylate	97-63-2	0.1	8260
2-Hexanone; Methyl butyl ketone	591-78-6		8260
Isobutyl alcohol	78-83-1		8260
Methacrylonitrile	126-98-7		8260
Methyl bromide; Bromomethane	74-83-9		8260
Methyl chloride; Chloromethane	74-87-3		8260
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3		8260
Methyl iodide; Iodomethane	74-88-4		8260
Methyl methacrylate	80-62-6		8260

### Federal Appendix II - Constituents for Assessment Monitoring (40 CFR Part 258)

### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 9 Page 2 of 5

	CAS Registry	Maximum Contaminant Level	RCRA
Appendix II Constituents	Number	(mg/L)	Method
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1		8260
Methylene bromide; Dibromomethane	74-95-3		8260
Methylene chloride; Dichloromethane	75-09-2	0.005	8260
Propionitrile; Ethyl cyanide	107-12-0		8260
Styrene	100-42-5	0.1	8260
1,1,1,2-Tetrachloroethane	630-20-6		8260
1,1,2,2-Tetrachloroethane	79-34-5		8260
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	0.005	8260
Toluene	108-88-3	1	8260
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	0.2	8260
1,1,2-Trichloroethane	79-00-5	0.005	8260
Trichloroethylene; Trichloroethene	79-01-6	0.005	8260
Trichlorofluoromethane	75-69-4		8260
1,2,3-Trichloropropane	96-18-4		8260
Vinyl acetate	108-05-4		8260
Vinyl chloride; Chloroethene	75-01-4	0.002	8260
Xylene (total)	96-47-6,	10	8260
	108-38-3,		
	106-42-3,		
	1330-20-7.		
SEMIVOLATILES			
Acenaphthene	83-32-9		8270
Acenaphthylene	208-96-8		8270
Acetophenone	98-86-2		8270
2-Acetylaminofluorene; 2-AAF	53-96-3		8270
4-Aminobiphenyl	92-67-1		8270
Anthracene	120-12-7		8270
Benzo[a]anthracene; Benzathracene	56-55-3		8270
Benzo[b]fluoranthene	205-99-2		8270
Benzo[k]fluoranthene	207-08-9		8270
Benzo[ghi]perylene	191-24-2		8270
Benzyl alcohol	100-51-6		8270
Bis(2-chloroethoxy)methane	111-91-1		8270
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4		8270
Bis(2-chloroisopropyl)ether; Bis(2-chloro-1-methylethyl) ether;	108-60-1		8270
2,2-Dichlorodiisopro-pyl ether; DCIP			
Bis(2-ethylhexyl) phthalate	117-81-7	0.006	8270
4-Bromophenyl phenyl ether	101-55-3		8270
Butyl benzyl phthalate	85-68-7		8270
p-Chloroaniline; 4-Chloro-aniline	106-47-8		8270
Chlorobenzilate	510-15-6		8270
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7		8270
2-Chloronaphthalene	91-58-7		8270
2-Chlorophenol	95-57-8		8270
4-Chlorophenyl phenyl ether	7005-72-3		8270
Chrysene	218-01-9		8270
m-Cresol; 3-Methylphenol	108-39-4		8270
o-Cresol; 2-Methylphenol	95-48-7		8270

### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 9 Page 3 of 5

	CAS Registry	Maximum Contaminant Level	RCRA
Appendix II Constituents	Number	(mg/L)	Method
p-Cresol; 4-Methylphenol	106-44-5		8270
Diallate	2303-16-4		8270
Dibenz[a,h]anthracene	53-70-3		8270
Dibenzofuran	132-64-9		8270
Di-n-butyl phthalate	84-74-2		8270
3,3'-Dichlorobenzidine	91-94-1		8270
2,4-Dichlorophenol	120-83-2		8270
2,6-Dichlorophenol	87-65-0		8270
Diethyl phthalate	84-66-2		8270
o,o-Diethyl-o-2-pyrazinyl phosphorothioate; Thionazin	297-97-2		8270
Dimethoate	60-51-5		8270
p-(Dimethylamino)azoben-zene	60-11-7		8270
7,12-Dimethylbenz[a]an-thracene	57-97-6		8270
3,3'-Dimethylbenzidine	119-93-7		8270
2,4-Dimethylphenol	105-67-9		8270
Dimethyl phthalate	131-11-3		8270
m-Dinitrobenzene	99-65-0		8270
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1		8270
2,4-Dinitrophenol	51-28-5		8270
2.4-Dinitrotoluene	121-4-2		8270
2,6-Dinitrotoluene	606-20-2		8270
Di-n-octyl phthalate	117-84-0		8270
Diphenylamine	122-39-4		8270
Disulfoton	298-04-4		8270
Ethyl methanesulfonate	62-50-0		8270
Famphur	52-85-7		8270
Fluoranthene	206-44-0		8270
Fluorene	86-73-7		8270
Hexachlorobenzene	118-74-1	0.001	8270
Hexachlorobutadiene	87-68-3	0.001	8270
Hexachlorocyclopentadiene	77-47-4	0.05	8270
Hexachloroethane	67-72-1	0.00	8270
Hexacloroproprene	1888-71-7		8270
Indeno[1,2,3-cd]pyrene	193-39-5		8270
Isodrin	465-73-6		8270
Isophorone	78-59-1		8270
Isosafrole	120-58-1		8270
Kepone	143-50-0		8270
Methapyrilene	91-80-5		8270
3-Methylcholanthrene	56-49-5		8270
Methyl methanesulfonate	66-27-3		8270
2-Methylnaphthalene	91-57-6		8270
Methyl parathion; Parathion methyl	298-00-0		8270
Naphthalene	<u>298-00-0</u> 91-20-3		8270
			8270
1,4-Naphthoquinone	130-15-4		
1-Naphthylamine	134-32-7		8270
2-Naphthylamine	91-59-8		8270
o-Nitroaniline; 2-Nitroaniline	88-74-4	<u> </u>	8270

### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 9 Page 4 of 5

	CAS Registry	Maximum Contaminant Level	RCRA
Appendix II Constituents	Number	(mg/L)	Method
m-Nitroaniline;3-Nitroaniline	99-09-2	, <b>,</b> ,	8270
p-Nitroaniline; 4-Nitroaniline	100-01-6		8270
Nitrobenzene	98-95-3		8270
o-Nitrophenol; 2-Nitrophenol	88-75-5		8270
p-Nitrophenol; 4-Nitrophenol	100-02-7		8270
N-nitrosodi-n-butylamine	924-16-3		8270
N-nitrosodiethylamine	55-18-5		8270
N-nitrosodimethylamine	62-75-9		8270
N-nitrosodiphenylamine	86-30-6		8270
N-nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-	621-64-7		8270
dipropylamine			
N-nitrosomethylethylamine	1059-95-6		8270
N-nitrosopiperidine	100-75-4		8270
N-nitrosopyrrolidine	930-55-2		8270
5-Nitro-o-toluidine	99-55-8		8270
Parathion	56-38-2		8270
Pentachlorobenzene	608-93-5		8270
Pentachloronitrobenzene	82-68-8		8270
Phenacetin	62-44-2		8270
Phenanthrene	85-01-8		8270
Phenol	108-95-2		8270
p-Phenylenediamine	106-50-2		8270
Phorate	298-02-2		8270
Pronamide	23950-58-5		8270
Pyrene	129-00-0		8270
Safrole	94-59-7		8270
1,2,4,5-Tetrachlorobenzene	95-94-3		8270
2,3,4,6-Tetrachlorophenol	58-90-2		8270
o-Toluidine	95-53-4		8270
1,2,4-Trichlorobenzene	120-82-1		8270
2,4,5-Trichlorophenol	95-95-4		8270
2,4,6-Trichlorophenol	88-06-2		8270
o,o,o-Triethyl phosphoro-thioate; Terbufos	126-68-1		8270
sym-Trinitrobenzene; 1,3,5-Trinitrobenzene	99-35-4		8270
Benzo[a]pyrene	50-32-8	0.0002	8310
ORGANOCHLORINE PESTICIDES/PCBs	50-52-0	0.0002	0310
Aldrin	309-00-2		8081
alpha-BHC; alpha-Benzene hexachloride	319-84-6		8081
beta-BHC; beta-Benzene hexachloride	309-85-7		8081
delta-BHC; delta-Benzene hexachloride	319-86-8		8081
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	0.0002	8081
alpha-Chlordane	5103-71-9	0.002	8081
	5103-74-2	0.002	8081
gamma-Chlordane		0.002	8081
4,4'-DDD	72-54-8		
4,4'-DDE	72-55-9		8081
4,4'-DDT	50-29-3		8081
Dieldrin	60-57-1		8081
Endosulfan I	959-98-8		8081

### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 9 Page 5 of 5

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
Endosulfan II	33213-65-9		8081
Endosulfan sulfate	1031-07-8		8081
Endrin	72-20-8	0.002	8081
Endrin aldehyde	7421-93-4		8081
Heptachlor	76-44-8	0.0004	8081
Heptachlor epoxide	1024-57-3	0.0002	8081
Methoxychlor	72-43-5	0.04	8081
Toxaphene	8001-35-2	0.003	8081
Polychlorinated biphenyls; PCBs; Aroclors	1336-36-3, 12674-11-2, 11104-28-2, 11141-16-5, 52460-21,0	0.0005	8082
	53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5		
HERBICIDES			
2,4-D; 2,4-Dichlorophenoxy-acetic acid	94-75-7	0.02	8151
2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid	93-76-5		8151
2,4,5-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex	93-72-1	0.05	8151
Dinoseb; DNBP; 2-sec-Butyl-4,6-dinitrophenol	88-85-7		8151
Pentachlorophenol	87-86-5	0.001	8151
METALS	=		
Antimony	7440-36-0	0.006	6010
Arsenic	7440-38-2	0.05	6010
Barium	7440-39-3	2.0	6010
Beryllium	7440-41-7	0.004	6010
Cadmium	7440-43-9	0.005	6010
Chromium	7440-47-3 7440-48-4	0.1	6010 6010
Cobalt	7440-48-4	1	6010
Copper Lead	7439-92-1	0.015	6010
Nickel	7439-92-1	0.015	6010
Selenium	7782-49-2	0.05	6010
Silver	7440-22-4	0.05	6010
Thallium	7440-22-4	0.002	6010
Tin	7440-28-0	0.002	6010
Vanadium	7440-31-3		6010
Zinc	7440-66-6	5	6010
Mercury	7439-97-6	0.002	7470
OTHER INORGANICS	1400-01-0	0.002	
Cyanide	57-12-5	0.2 as Free	9012
Sulfide	18496-25-8	0.2 001100	9034

Appendix IX Constituents	CAS Registry Number	RCRA Method
VOLATILES		
Acetone	67-64-1	8081
Acetonitrile; Methyl cyanide	75-05-8	8081
Acrolein	107-02-8	8081
Acrylonitrile	107-13-1	8081
Allyl chloride	107-05-1	8081
Benzene	71-43-2	8081
Bromodichloromethane; Dichlorobromomethane	75-27-4	8081
Bromoform; Tribromomethane	75-25-5	8081
Carbon disulfide	75-15-0	8081
Carbon tetrachloride	56-23-5	8081
Chlorobenzene	108-90-7	8081
Chloroethane; Ethyl chloride	75-00-3	8081
Chloroform; Trichloro-methane	67-66-3	8081
Chloroprene	126-99-8	8081
Dibromochloromethane; Chlorodibromomethane	124-48-1	8081
1,2-Dibromo-3-chloropro-pane; DBCP	96-12-8	8011
1,2-Dibromoethane; Ethylene dibromide; EDB	106-93-4	8011
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	8081
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1	8081
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	8081
trans-1,4-Dichloro-2-butene	110-57-6	8081
Dichlorodifluoromethane	75-71-8	8081
1,1-Dichloroethane; Ethylidene chloride	75-34-3	8081
1,2-Dichloroethane; Ethylene dichloride	107-06-2	8081
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	8081
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	8081
1,2-Dichloropropane; Propylene dichloride	78-87-5	8081
cis-1,3-Dichloropropene	10061-01-5	8081
trans-1,3-Dichloropropene	10061-02-6	8081
1,4-Dioxane	123-91-1	8081
Ethylbenzene	100-41-4	8081
Ethyl methacrylate	97-63-2	8081
Hexachlorobutadiene	87-68-3	8081
2-Hexanone; Methyl butyl ketone	591-78-6	8081
Isobutyl alcohol	78-83-1	8081
Methacrylonitrile	126-98-7	8081
Methyl bromide; Bromomethane	74-83-9	8081
Methyl chloride; Chloromethane	74-87-3	8081
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3	8081
Methyl iodide; lodomethane	74-88-4	8081
Methyl methacrylate	80-62-6	8081
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1	8081
Methylene bromide; Dibromomethane	74-95-3	8081
Methylene chloride; Dichloromethane	75-09-2	8081

### Federal Appendix IX – Groundwater Monitoring List (40 CFR Part 264)

\_\_\_\_\_

	CAS Registry	RCRA
Appendix IX Constituents	Number	Method
Propionitrile; Ethyl cyanide	107-12-0	8081
Styrene	100-42-5	8081
1,1,1,2-Tetrachloroethane	630-20-6	8081
1,1,2,2-Tetrachloroethane	79-34-5	8081
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	8081
Toluene	108-88-3	8081
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	8081
1,1,2-Trichloroethane	79-00-5	8081
Trichloroethylene; Trichloroethene	79-01-6	8081
Trichlorofluoromethane	75-69-4	8081
1,2,3-Trichloropropane	96-18-4	8081
Vinyl acetate	108-05-4	8081
Vinyl chloride; Chloroethene	75-01-4	8081
Xylenes (total)	96-47-6,	8081
	108-38-3,	0001
	106-42-3,	
	1330-20-7.	
SEMIVOLATILES	1000 20 7.	
Acenaphthene	83-32-9	8270
Acenaphthylene	208-96-8	8270
Acetophenone	98-86-2	8270
2-Acetylaminofluorene; 2-AAF	53-96-3	8270
4-Aminobiphenyl	92-67-1	8270
Aniline	62-53-3	8270
Anthracene	120-12-7	8270
Aramite	140-57-8	8270
Benzo(a)anthracene; Benzathracene	56-55-3	8270
Benzo(a)pyrene	50-32-8	8270
Benzo(b)fluoranthene	205-99-2	8270
Benzo(k)fluoranthene	207-08-9	8270
Benzo(ghi)perylene	191-24-2	8270
Benzyl alcohol	100-51-6	8270
Bis(2-chloroethoxy)methane	111-91-1	8270
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4	8270
Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiiso-propyl	108-60-1	8270
ether; DCIP		
Bis(2-ethylhexyl) phthalate	117-81-7	8270
4-Bromophenyl phenyl ether	101-55-3	8270
Butyl benzyl phthalate	85-68-7	8270
p-Chloroaniline; 4-Chloro-aniline	106-47-8	8270
Chlorobenzilate	510-15-6	8270
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7	8270
2-Chloronaphthalene	91-58-7	8270
2-Chlorophenol	95-57-8	8270
4-Chlorophenyl phenyl ether	7005-72-3	8270
Chrysene	218-01-9	8270
m-Cresol; 3-Methylphenol	108-39-4	8270
o-Cresol; 2-Methylphenol	95-48-7	8270
	90-40-7	0210

\_

	CAS Registry	RCRA
Appendix IX Constituents	Number	Method
p-Cresol; 4-Methylphenol	106-44-5	8270
Diallate	2303-16-4	8270
Dibenz(a,h)anthracene	53-70-3	8270
Dibenzofuran	132-64-9	8270
Di-n-butyl phthalate	84-74-2	8270
3.3'-Dichlorobenzidine	91-94-1	8270
2,4-Dichlorophenol	120-83-2	8270
2,6-Dichlorophenol	87-65-0	8270
Diethyl phthalate	84-66-2	8270
o,o-Diethyl-o-2-pyrazinyl phosphorothioate; Thionazine	297-97-2	8270
Dimethoate	60-51-5	8270
p-(Dimethylamino)azobenzene	60-11-7	8270
7,12-Dimethylbenz(a)anthracene	57-97-6	8270
3,3'-Dimethylbenzidine	119-93-7	8270
2,4-Dimethylphenol	105-67-9	8270
Dimethyl phthalate	131-11-3	8270
a,a-Dimethylphenethylamine	122-09-8	8270
m-Dinitrobenzene; 1,3-Dinitrobenzene	99-65-0	8270
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1	8270
2,4-Dinitrophenol	51-28-5	8270
2,4-Dinitrotoluene	121-4-2	8270
2,6-Dinitrotoluene	606-20-2	8270
Di-n-octyl phthalate	117-84-0	8270
Dinoseb; DNBP; 2-sec-Butyl-4,6-dinitrophenol	88-85-7	8270
Diphenylamine	122-39-4	8270
Disulfoton	298-04-4	8270
Ethyl methanesulfonate	62-50-0	8270
Famphur	52-85-7	8270
Fluoranthene	206-44-0	8270
Fluorene	86-73-7	8270
Hexachlorobenzene	118-74-1	8270
Hexachlorobutadiene	87-68-3	8270
Hexachlorocyclopentadiene	77-47-4	8270
Hexachloroethane	67-72-1	8270
Hexachlorophene	70-30-4	8270
Hexacloroproprene	1888-71-7	8270
Indeno(1,2,3-cd)pyrene	193-39-5	8270
Isodrin	465-73-6	8270
Isophorone	78-59-1	8270
Isosafrole	120-58-1	8270
Kepone	143-50-0	8270
Methapyrilene	91-80-5	8270
3-Methylcholanthrene	56-49-5	8270
Methyl methanesulfonate	66-27-3	8270
2-Methylnaphthalene	91-57-6	8270
Methyl parathion; Parathion methyl	298-00-0	8270
Naphthalene	91-20-3	8270
1,4-Naphthoguinone	130-15-4	8270

Appendix IX Constituents	CAS Registry Number	RCRA Method
1-Naphthylamine	134-32-7	8270
2-Naphthylamine	91-59-8	8270
o-Nitroaniline; 2-Nitroaniline	88-74-4	8270
m-Nitroaniline;3-Nitroaniline	99-09-2	8270
p-Nitroaniline; 4-Nitroaniline	100-01-6	8270
Nitrobenzene	98-95-3	8270
5-Nitro-o-toluidine	99-55-8	8270
o-Nitrophenol; 2-Nitrophenol	88-75-5	8270
p-Nitrophenol; 4-Nitrophenol	100-02-7	8270
4-Nitroquinoline 1-oxide	56-57-5	8270
N-nitrosodi-n-butylamine	924-16-3	8270
N-nitrosodiethylamine	55-18-5	8270
N-nitrosodimethylamine	62-75-9	8270
N-nitrosodiphenylamine	86-30-6	8270
N-nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-	621-64-7	8270
dipropylamine	021-04-7	8270
N-nitrosomethylethylamine	1059-95-6	8270
N-nitrosomorpholine	59-89-2	8270
N-nitrosopiperidine	100-75-4	8270
N-nitrosopyrrolidine	930-55-2	8270
Parathion	56-38-2	8270
Pentachlorobenzene	608-93-5	8270
Pentachloroethane	76-01-7	8270
Pentachloronitrobenzene	82-68-8	8270
Pentachlorophenol	87-86-5	8270
Phenacetin	62-44-2	8270
Phenanthrene	85-01-8	8270
Phenol	108-95-2	8270
p-Phenylenediamine; 1,4-Phenylenediamine	106-50-3	8270
Phorate	298-02-2	8270
2-Picoline	109-06-8	8270
Pronamide	23950-58-5	8270
Pyrene	129-00-0	8270
Pyridine	110-86-1	8270
Safrole	94-59-7	8270
1,2,4,5-Tetrachlorobenzene	95-94-3	8270
2,3,4,6-Tetrachlorophenol	58-90-2	8270
Tetraethylpyrophosphate, Sulfotep	3689-24-5	8270
o-Toluidine	95-53-4	8270
1,2,4-Trichlorobenzene	120-82-1	8270
2,4,5-Trichlorophenol	95-95-4	8270
2,4,6-Trichlorophenol	88-06-2	8270
o,o,o-Triethyl phosphoro-thioate; Terbufos	126-68-1	8270
sym-Trinitrobenzene; 1,3,5-Trinitrobenzene	99-35-4	8270
ORGANOCHLORINE PESTICIDES/PCBs		
Aldrin	309-00-2	8081
alpha-BHC; alpha-Benzene hexachloride	319-84-6	8081
beta-BHC; beta-Benzene hexachloride	309-85-7	8081

delta-BHC;delta-Benzene hexachloride;         319-86-8         8081           gamma-BHC; gamma-Benzene hexachloride;         Lindane         58-89-9         8081           gamma-Chlordane         5103-74-2         8081           gama-Chlordane         5103-74-2         8081           4,4'-DDD         72-55-9         8081           4,4'-DDT         50-29-3         8081           4,4'-DDT         50-29-3         8081           Endosulfan 1         959-98-8         8081           Endosulfan 1         33213-65-9         8081           Endosulfan 1         33213-65-9         8081           Endosulfan sulfate         1031-07-8         8081           Endrin         72-24-8         8081           Endrin aldehyde         7421-93-4         8081           Heptachlor         76-44-8         8081           Heptachlor epoxide         1024-57-3         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3,         8082           2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-7         8151           2,4-D; 2,4-Dichlorophenoxy-acetic acid         93-76-5         8151           2,4-D; 2, 4,5-Trichlor	Appendix IX Constituents	CAS Registry Number	RCRA Method
gamma-BHC:         gamma-Benzene hexachloride;         Lindane         503-71-9         8081           alpha-Chlordane         5103-71-9         8081         4.4'-DDD         72-54-8         8081           4.4'-DDD         72-55-9         8081         4.4'-DDT         50-29-3         8081           4.4'-DDT         50-29-3         8081         109-100         50-29-3         8081           Endosulfan I         959-98-8         8081         8081         109-107-8         8081           Endosulfan III         33213-65-9         8081         109-107-8         8081         109-107-8         8081           Endosulfan sulfate         1031-07-8         8081         109-107-8         8081         109-107-8         8081           Endrin aldehyde         7421-93-4         8081         109-107-8         8081         109-107-8         8081           Heptachlor epoxide         1024-57-3         8081         109-107-8         8081         109-107-8         8081           Polychlorinated biphenyls; PCBs; Aroclors         136-36-3,         8082         1267-11-2,         11104-28-2,         1109-68-1,         11096-82-5         1267-29-6,         11097-69-1,         11096-82-5         1511         2,4,5-Tr; 2,4,5-Trichlorophen-oxyacetic acid			
alpha-Chlordane         5103-71-9         8081           garma-Chlordane         5103-74-2         8081           4.4'-DDD         72-54-8         8081           4.4'-DDE         72-55-9         8081           4.4'-DDT         50-29-3         8081           4.4'-DDT         60-57-1         8081           Endosulfan I         959-98-8         8081           Endosulfan sulfate         1031-07-8         8081           Endosulfan sulfate         1031-07-8         8081           Endrin         72-20-8         8081           Endrin         72-20-8         8081           Endrin aldehyde         742-1-9.4         8081           Heptachlor         76-44-8         8081           Heptachlor         72-43-5         8081           Nethoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 12674-11-2, 11104-28-2, 11104-28-2, 11104-28-2, 111097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 11097-69-1, 12672-29-6, 11097-69-1, 12672-29-6, 11097-60-16010           Artanony         7440-38-2         6010           Arencic			
gamma-Chlordane         5103-74-2         8081           4,4'-DDD         72-54-8         8081           4,4'-DDE         72-55-9         8081           4,4'-DDT         50-29-3         8081           Dieldrin         60-57-1         8081           Endosulfan I         959-98-8         8081           Endosulfan I         3213-65-9         8081           Endosulfan sulfate         1031-07-8         8081           Endrin aldehyde         7421-93-4         8081           Endrin aldehyde         7421-93-4         8081           Heptachlor         76-44-8         8081           Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3,         8082           11004-28-2,         11104-28-2,         11104-28-2,           111097-69-1,         11096-82-5         11097-69-1,           HERBICIDES         2,4-57:         8151         2,4,5-7:           2,4-51; 2,4-5-Trichlorophen-oxyacetic acid         93-72-1         8151           2,4-51; 2,4-5-Trichlorophen-oxyacetic acid         93-72-1			
4.4-DDD         72-54-8         8081           4.4-DDE         72-55-9         8081           Jeldrin         60-57-1         8081           Dieldrin         60-57-1         8081           Endosulfan I         959-98-8         8081           Endosulfan II         33213-65-9         8081           Endosulfan sulfate         1031-07-8         8081           Endrin         72-20-8         8081           Endrin         72-20-8         8081           Heptachlor         76-44-8         8081           Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3,         8082           12672-29-6,         11097-69-1,         11096-82-5           HERBICIDES         2.4-51; 2.4-5-Trichlorophen-oxyacetic acid         93-76-5         8151           2.4,5-1; 2.4,5-Trichlorophenoxyacetic acid         93-76-5         8151           2.4,5-1; 2.4,5-Trichlorophenoxyacetic acid         93-72-1         8151           METALS         7440-38-2         6010           Arsenic         7440-38-2         6010<			
4.4-DDE         72-55-9         8081           4.4-DDT         50-29-3         8081           Endosulfan I         959-98-8         8081           Endosulfan II         33213-65-9         8081           Endosulfan II         33213-65-9         8081           Endosulfan Sulfate         1031-07-8         8081           Endrin         72-20-8         8081           Endrin         72-20-8         8081           Endrin         72-20-8         8081           Endrin         72-20-8         8081           Endrin         72-43-5         8081           Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-3-3, 8082         8082           12672-19-6, 1         11097-68-1, 1104-28-2, 11141-16-5, 53469-21-9, 12672-29-6, 11097-68-1, 11096-82-5         11097-68-1, 11096-82-5           HERBICIDES         2         4-5-77         8151           2.4-D; 2.4-D:Ichlorophenoxy-acetic acid         93-72-1         8151           2.4-5, Tichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           2.4-5, Tic			
4.4'-DDT         50-29-3         8081           Dieldrin         60-57-1         8081           Endosulfan I         959-98-8         8081           Endosulfan II         33213-65-9         8081           Endosulfan sulfate         1031-07-8         8081           Endrin         72-20-8         8081           Endrin aldehyde         7421-93-4         8081           Heptachlor         76-44-8         8081           Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3,         8082           12674-11-2,         11104-28-2,         11141-16-5,         53469-21-9,           12677-29-6,         11097-69-1,         11097-69-1,         11097-69-1,           11097-69-1,         11097-89-2,         11141-16-5,         53469-21-9,           2.4-D; 2.4-Dichlorophenoxy-acetic acid         94-75-7         8151           METALS         7440-38-0         6010           Arismic         7440-38-2         6010           Arismin         7440-38-2         6010           Copper         7			
Dieldrin         60-57-1         8081           Endosulfan I         959-98-8         8081           Endosulfan II         33213-65-9         8081           Endosulfan sulfate         1031-07-8         8081           Endrin         72-20-8         8081           Endrin aldehyde         7421-93-4         8081           Heptachlor         76-44-8         8081           Heptachlor         76-44-8         8081           Heptachlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 12674-11-2, 11104-28-2,         8082           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 12672-29-6, 11097-69-1, 11097-69-1, 11097-69-1,         8082           Actification         94-75-7         8151         8151           2,4-D; 2,4-Dichlorophen-oxyacetic acid         93-76-5         8151           2,4-S-T1; chloro-phenoxypropanoic acid; Silvex         93-76-5         8151           2,4-S-T1; chloro-phenoxypropanoic acid; Silvex         93-72-1         8151           Antimony         7440-38-0         6010           Arsenic         7440-39-3         6010           Barium         7440-43-7			
Endosulfan I         959-98-8         8081           Endosulfan sulfate         1031-07-8         8081           Endosulfan sulfate         1031-07-8         8081           Endrin         72-20-8         8081           Endrin         72-20-8         8081           Endrin aldehyde         7421-93-4         8081           Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         12672-29-6, 11097-89-1, 11096-82-5         53469-21-9, 12672-29-6, 11097-89-1, 11096-82-5           HERBICIDES         11096-82-5         12672-29-6, 11097-89-1, 11096-82-5         11097-69-1, 11096-82-5           HERBICIDES         124,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5         8151           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5         8151			
Endosulfan II         33213-65-9         8081           Endosulfan sulfate         1031-07-8         8081           Endrin         72-20-8         8081           Endrin         72-20-8         8081           Endrin aldehyde         72-20-8         8081           Heptachlor         76-44-8         8081           Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 12674-11-2, 11104-28-2, 11104-28-2,         8082           12672-29-6, 11097-69-1, 11097-69-1,         11096-82-5         11097-69-1, 1097-69-1,           HERBICIDES         11096-82-5         11096-82-5           HERBICIDES         2,4-D; 2,4-Dichlorophenoxy-acetic acid         93-76-5         8151           2,4-S-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           Antimony         7440-38-2         6010           Arsenic         7440-38-2         6010           Barium         7440-43-9         6010           Cadmium         7440-43-9         6010           Copper         7440-43-8         6010			
Endosulfan sulfate         1031-07-8         8081           Endrin         72-20-8         8081           Endrin aldehyde         7421-93-4         8081           Endrin aldehyde         7421-93-4         8081           Heptachlor         76-44-8         8081           Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 12674-11-2, 11114-16-5, 53469-21-9, 12672-29-6, 11097-69-1,         8082           Left - 1.2, 2.4-Dichlorophenoxy-acetic acid         94-75-7         8151           2.4-D; 2.4-Dichlorophen-oxyacetic acid         93-76-5         8151           2.4,5-Tr; 2.4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           METALS			
Endrin         72-20-8         8081           Endrin aldehyde         7421-93-4         8081           Heptachlor         76-44-8         8081           Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 1366-3, 13674-11-2, 11104-28-2, 11141-16-5, 53469-21-9, 12672-29-6,         8082           Yelf         11097-69-1, 11097-69-1, 11097-69-1,         11097-69-1, 11097-69-1,           2,4-D; 2,4-Dichlorophenoxy-acetic acid         93-76-5         8151           2,4,5-T; 2,4,5-Trichloro-phenoxyacetic acid         93-76-5         8151           2,4,5-T; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           2,4,5-T; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           2,4,5-T; 3,4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8010           Beryllium         7440-38-2         6010           Antimony         7440-38-2         6010           Beryllium         7440-43-9         6010           Cobalt         7440-43-9         6010           Copper         7440-48-4         6010			
Endrin aldehyde         7421-93-4         8081           Heptachlor         76-44-8         8081           Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 12674-11-2, 11104-28-2, 11104-28-2, 11104-28-2, 111097-69-1, 11097-69-1, 11097-69-1, 11097-69-1,         8082           Person Participation         94-75-7         8151           2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-5         8151           2,4-S-T; 2,4,5-Trichlorophenoxyacetic acid         93-76-5         8151           2,4,5-T; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           Antimony         7440-38-2         6010           Arsenic         7440-38-2         6010           Barium         7440-43-3         6010           Cadmium         7440-43-9         6010           Cobalt         7440-48-4         6010           Cobalt         7440-48-4         6010           Cobalt         7440-48-4         6010           Cobalt         7440-48-4         6010           Cobalt         7440-22-0         6010 <tr< td=""><td></td><td></td><td></td></tr<>			
Heptachlor         76-44-8         8081           Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 1336-36-3, 12674-11-2, 11104-28-2,         8082           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 1097-69-1, 11096-82-5         8082           HERBICIDES         2,4-5,2,4-5,7         8151           2,4-5,7; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5         8151           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5         8151           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-72-1         8151           METALS			
Heptachlor epoxide         1024-57-3         8081           Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 12674-11-2, 11104-28-2, 111104-28-2, 111104-28-2,         8082           HERBICIDES         12672-29-6, 11097-69-1, 11096-82-5         12672-29-6,           Yet, S., T., Z.4, 5-Trichlorophenoxy-acetic acid         94-75-7         8151           2,4-D; 2,4-Dichlorophenoxy-acetic acid         93-76-5         8151           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5         8151           2,4,5-TP; 2,4,5-Trichlorophenoxypropanoic acid; Silvex         93-72-1         8151           METALS			
Methoxychlor         72-43-5         8081           Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 12674-11-2, 11104-28-2, 111141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5         8082           HERBICIDES			
Toxaphene         8001-35-2         8081           Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 123674-11-2, 11104-28-2, 11104-28-2, 111141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5         8082           HERBICIDES         2         4         D; 2,4-Dichlorophenoxy-acetic acid         94-75-7         8151           2,4-5, 2,4-Dichlorophen-oxyacetic acid         93-76-5         8151         2,4,5-Tr; 2,4,5-Trichlorophen-oxyacetic acid         93-72-1         8151           2,4,5-TP; 2,4,5-Trichlorophen-oxyacetic acid; Silvex         93-72-1         8151         0           Antimony         7440-36-0         6010         6010           Arsenic         7440-39-3         6010         6010           Barium         7440-43-9         6010         6010           Chromium         7440-43-9         6010         6010           Cobalt         7440-47-3         6010         6010           Cobalt         7440-48-4         6010         6010           Copper         7440-60-8         6010         6010           Nickel         7440-20         6010         6010           Silver         7440-22-4         6010         6010           Silver         7440-62-0         6010         7440-22-2         6010			
Polychlorinated biphenyls; PCBs; Aroclors         1336-36-3, 12674-11-2, 11104-28-2, 11104-28-2, 11104-28-2, 11104-28-2, 11104-28-2, 111097-69-1, 11096-82-5         8082           HERBICIDES         I <td></td> <td></td> <td></td>			
12674-11-2, 11104-28-2, 111141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5           HERBICIDES           2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-7           2,4,5-Trichlorophen-oxyacetic acid         93-76-5           2,4,5-Trichlorophen-oxyacetic acid         93-76-5           2,4,5-Trichlorophen-oxyacetic acid         93-72-1           8151         1000000000000000000000000000000000000			
11104-28-2, 11141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5           HERBICIDES           2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-7           2,4,5-T; 2,4,5-Trichloro-phen-oxyacetic acid         93-76-5           8151         2,4,5-T; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex           93-72-1         8151           METALS	Polychionnated biphenyls, PCBS, Arociols		0002
11141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5           HERBICIDES           2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-7           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5           2,4,5-T; 2,4,5-Trichlorophenoxypropanoic acid; Silvex         93-72-1           8151         93-76-5           2,4,5-Trichlorophenoxypropanoic acid; Silvex         93-72-1           8151         93-72-1           Antimony         7440-38-2           Antimony         7440-38-2           6010         6010           Arsenic         7440-38-2           Barium         7440-39-3           6010         6010           Cadmium         7440-43-9           Cobalt         7440-43-9           Copper         7440-50-8           Copper         7440-50-8           Colol         7440-50-8           Nickel         7440-02-0           Selenium         7782-49-2           Nickel         7440-22-4           Silver         7440-22-0           Silver         7440-22-0           Silver         7440-22-0           Silver         7440-22-0           Silver         7440-62-2		,	
53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5           HERBICIDES           2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-7           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid; Silvex         93-72-1           METALS         93-72-1           Antimony         7440-36-0           Antimony         7440-38-2           Barium         7440-38-2           Barium         7440-43-3           Beryllium         7440-41-7           Codmium         7440-43-9           Cohon         7440-43-9           Cohon         7440-43-9           Cohon         7440-43-9           Cohon         7440-47-3           Cohon         7440-48-4           Cohon         7440-49-8           Cohon         7440-50-8           Cohon         7440-50-8           Cohon         7440-20-0           Silver         7440-22-4           Solto         Silver           Thin         7440-22-4           Solto         Silver           Totalium         7440-31-5           Solto         Silver           Solto         Solto <t< td=""><td></td><td>,</td><td></td></t<>		,	
12672-29-6, 11097-69-1, 11096-82-5           HERBICIDES           2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-7           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5           X,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid; Silvex         93-72-1           METALS         93-72-1           Antimony         7440-36-0           Arsenic         7440-38-2           Barium         7440-41-7           Barium         7440-43-9           Cadmium         7440-43-9           Cohon         6010           Cobalt         7440-43-9           Cobalt         7440-43-9           Cobalt         7440-23-9           Cobalt         7440-47-3           Cobalt         7440-48-4           Colon         6010           Selenium         7440-20           Silver         7440-22-0           Silver         7440-22-4           Sol10         5           Selenium         7440-28-0           Silver         7440-28-0           Theilium         7440-28-0           Sol10         7440-28-0           Selenium         7440-28-0           Silver         7440-28-0			
HERBICIDES         11097-69-1, 11096-82-5           2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-7         8151           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5         8151           2,4,5-TP; 2,4,5-Trichlorophen-oxyacetic acid; Silvex         93-72-1         8151           METALS         93-72-1         8151           Antimony         7440-36-0         6010           Arsenic         7440-38-2         6010           Barium         7440-39-3         6010           Beryllium         7440-43-9         6010           Cadmium         7440-43-9         6010           Cobalt         7440-43-9         6010           Cobalt         7440-48-4         6010           Copper         7440-50-8         6010           Lead         7440-20-0         6010           Nickel         7440-22-0         6010           Selenium         7440-22-0         6010           Silver         7440-28-0         6010           Tin         7440-28-0         6010           Silver         7440-28-0         6010           Tin         7440-28-0         6010           Tin         7440-66-6         6010			
HERBICIDES         11096-82-5           2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-7         8151           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5         8151           2,4,5-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           METALS         93-72-1         8151           Antimony         7440-36-0         6010           Arsenic         7440-38-2         6010           Barium         7440-39-3         6010           Beryllium         7440-43-9         6010           Cadmium         7440-43-9         6010           Cobalt         7440-43-9         6010           Cobalt         7440-43-9         6010           Cobalt         7440-47-3         6010           Copper         7440-48-4         6010           Copper         7440-48-4         6010           Nickel         7440-50-8         6010           Nickel         7440-20         6010           Selenium         7782-49-2         6010           Nickel         7440-28-0         6010           Silver         7440-28-0         6010           Tin         7440-28-0         6010			
HERBICIDES         1           2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-7         8151           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5         8151           2,4,5-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           METALS         93-72-1         8151           Antimony         7440-36-0         6010           Arsenic         7440-38-2         6010           Barium         7440-39-3         6010           Cadmium         7440-41-7         6010           Cadmium         7440-43-9         6010           Cobalt         7440-47-3         6010           Copper         7440-47-3         6010           Lead         7440-47-3         6010           Nickel         7440-20-0         6010           Nickel         7440-22-4         6010           Nickel         7440-22-4         6010           Selenium         7440-22-4         6010           Silver         7440-22-4         6010           Thallium         7440-22-4         6010           Tin         7440-22-2         6010           Zinc         7440-62-2         6010           Veran			
2,4-D; 2,4-Dichlorophenoxy-acetic acid         94-75-7         8151           2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5         8151           2,4,5-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           METALS         7440-36-0         6010           Arsenic         7440-38-2         6010           Barium         7440-39-3         6010           Beryllium         7440-41-7         6010           Cadmium         7440-43-9         6010           Codemium         7440-43-9         6010           Cobalt         7440-43-9         6010           Copper         7440-47-3         6010           Lead         7440-48-4         6010           Nickel         7440-50-8         6010           Selenium         7440-50-8         6010           Silver         7440-62-8         6010           Tin         7440-22-4         6010           Tin         7440-28-0         6010           Silver         7440-28-0         6010           Tin         7440-28-0         6010           Tin         7440-62-2         6010           Tin         7440-62-2         6010	HERBICIDES	11000 02 0	
2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid         93-76-5         8151           2,4,5-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           METALS         7440-36-0         6010           Arsenic         7440-38-2         6010           Barium         7440-39-3         6010           Beryllium         7440-43-9         6010           Cadmium         7440-43-9         6010           Cadmium         7440-43-9         6010           Cobalt         7440-47-3         6010           Copper         7440-48-4         6010           Copper         7440-50-8         6010           Lead         7439-92-1         6010           Nickel         7440-02-0         6010           Selenium         7440-22-4         6010           Silver         7440-22-4         6010           Tin         7440-22-0         6010           Silver         7440-22-4         6010           Tin         7440-22-4         6010           Tin         7440-22-0         6010           Tin         7440-22-2         6010           Tin         7440-22-2         6010           Tin         <		94-75-7	8151
2,4,5-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex         93-72-1         8151           METALS         7440-36-0         6010           Arimony         7440-38-2         6010           Barium         7440-39-3         6010           Barium         7440-41-7         6010           Cadmium         7440-41-7         6010           Cadmium         7440-43-9         6010           Chromium         7440-43-9         6010           Cobalt         7440-47-3         6010           Copper         7440-50-8         6010           Lead         7440-20         6010           Nickel         7440-02-0         6010           Silver         7440-22-4         6010           Silver         7440-28-0         6010           Tin         7440-28-0         6010           Tin         7440-62-2         6010           Zinc         7440-62-2         6010           Mercury         7440-66-6         6010			
METALS         7440-36-0         6010           Antimony         7440-38-2         6010           Arsenic         7440-38-2         6010           Barium         7440-39-3         6010           Beryllium         7440-41-7         6010           Cadmium         7440-41-7         6010           Cadmium         7440-43-9         6010           Chromium         7440-43-9         6010           Cobalt         7440-47-3         6010           Copper         7440-50-8         6010           Lead         7439-92-1         6010           Nickel         7440-20         6010           Selenium         7782-49-2         6010           Silver         7440-28-0         6010           Tin         7440-31-5         6010           Vanadium         7440-62-2         6010           Zinc         7440-62-2         6010           Mercury         7440-66-6         6010			
Antimony         7440-36-0         6010           Arsenic         7440-38-2         6010           Barium         7440-39-3         6010           Beryllium         7440-41-7         6010           Cadmium         7440-43-9         6010           Chromium         7440-43-9         6010           Chromium         7440-43-9         6010           Cobalt         7440-47-3         6010           Copper         7440-50-8         6010           Lead         7440-50-8         6010           Nickel         7440-02-0         6010           Selenium         7782-49-2         6010           Silver         7440-22-4         6010           Thallium         7440-22-4         6010           Tin         7440-31-5         6010           Zinc         7440-62-2         6010           Mercury         7440-66-6         6010		00-72-1	0101
Arsenic         7440-38-2         6010           Barium         7440-39-3         6010           Beryllium         7440-41-7         6010           Cadmium         7440-43-9         6010           Chromium         7440-47-3         6010           Cobalt         7440-48-4         6010           Copper         7440-50-8         6010           Lead         7440-50-8         6010           Nickel         7440-02-0         6010           Selenium         7782-49-2         6010           Silver         7440-22-4         6010           Thallium         7440-28-0         6010           Tin         7440-31-5         6010           Zinc         7440-62-2         6010           Mercury         7439-97-6         7470,7471		7440-36-0	6010
Barium7440-39-36010Beryllium7440-41-76010Cadmium7440-43-96010Chromium7440-47-36010Cobalt7440-48-46010Copper7440-50-86010Lead7439-92-16010Nickel7440-206010Selenium7782-49-26010Silver7440-22-46010Thallium7440-28-06010Tin7440-31-56010Zinc7440-66-66010Mercury7439-97-67470,7471			
Beryllium         7440-41-7         6010           Cadmium         7440-43-9         6010           Chromium         7440-47-3         6010           Cobalt         7440-48-4         6010           Copper         7440-50-8         6010           Lead         7440-20-8         6010           Nickel         7440-20         6010           Selenium         7782-49-2         6010           Silver         7440-22-4         6010           Thallium         7440-31-5         6010           Tin         7440-62-2         6010           Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Cadmium         7440-43-9         6010           Chromium         7440-47-3         6010           Cobalt         7440-48-4         6010           Copper         7440-50-8         6010           Lead         7439-92-1         6010           Nickel         7440-20         6010           Selenium         7782-49-2         6010           Silver         7440-28-0         6010           Thallium         7440-28-0         6010           Tin         7440-62-2         6010           Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Chromium         7440-47-3         6010           Cobalt         7440-48-4         6010           Copper         7440-50-8         6010           Lead         7439-92-1         6010           Nickel         7440-02-0         6010           Selenium         7782-49-2         6010           Silver         7440-22-4         6010           Thallium         7440-28-0         6010           Tin         7440-31-5         6010           Vanadium         7440-62-2         6010           Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Cobalt         7440-48-4         6010           Copper         7440-50-8         6010           Lead         7439-92-1         6010           Nickel         7440-02-0         6010           Selenium         7782-49-2         6010           Silver         7440-22-4         6010           Thallium         7440-28-0         6010           Tin         7440-31-5         6010           Vanadium         7440-62-2         6010           Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Copper7440-50-86010Lead7439-92-16010Nickel7440-02-06010Selenium7782-49-26010Silver7440-22-46010Thallium7440-28-06010Tin7440-31-56010Vanadium7440-62-26010Zinc7440-66-66010Mercury7439-97-67470,7471			
Lead         7439-92-1         6010           Nickel         7440-02-0         6010           Selenium         7782-49-2         6010           Silver         7440-22-4         6010           Thallium         7440-28-0         6010           Tin         7440-31-5         6010           Vanadium         7440-62-2         6010           Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Nickel         7440-02-0         6010           Selenium         7782-49-2         6010           Silver         7440-22-4         6010           Thallium         7440-28-0         6010           Tin         7440-31-5         6010           Vanadium         7440-62-2         6010           Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Selenium         7782-49-2         6010           Silver         7440-22-4         6010           Thallium         7440-28-0         6010           Tin         7440-31-5         6010           Vanadium         7440-62-2         6010           Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Silver         7440-22-4         6010           Thallium         7440-28-0         6010           Tin         7440-31-5         6010           Vanadium         7440-62-2         6010           Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Thallium7440-28-06010Tin7440-31-56010Vanadium7440-62-26010Zinc7440-66-66010Mercury7439-97-67470,7471			
Tin         7440-31-5         6010           Vanadium         7440-62-2         6010           Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Vanadium         7440-62-2         6010           Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Zinc         7440-66-6         6010           Mercury         7439-97-6         7470,7471			
Mercury 7439-97-6 7470,7471			
	Mercury OTHER INORGANICS	1439-97-6	1410,1411

Appendix IX Constituents	CAS Registry Number	RCRA Method
Cyanide	57-12-5	9012
Sulfide	18496-25-8	9034
DIOXINS		
2,3,7,8-Tetrachlorodibenzo-o-dioxin	1746-01-6	8280 / 8290
Polychlorinated dibenzo-furans; PCDFs	NA	8280 / 8290
Polychlorinated dibenzo-p-dioxins; PCDDs	NA	8280 / 8290

TCL/TAL Constituents	CAS Registry Number	RCRA Method
VOLATILES	07.04.4	0000
Acetone	67-64-1	8260
Benzene	71-43-2	8260
Bromochloromethane	74-97-5	8260
Bromodichloromethane; Dichlorobromomethane	75-27-4	8260
Bromoform; Tribromomethane	75-25-5	8260
Carbon disulfide	75-15-0	8260
Carbon tetrachloride	56-23-5	8260
Chlorobenzene	108-90-7	8260
Chloroethane; Ethyl chloride	75-00-3	8260
Chloroform; Trichlorome-thane	67-66-3	8260
Cyclohexane	110-82-7	
Dibromochloromethane; Chlorodibromomethane	124-48-1	8260
1,2-Dibromomethane	106-93-4	8260
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	8260
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1	8260
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	8260
Dichlorodifluoromethane	75-71-8	8260
1,1-Dichloroethane; Ethylidene chloride	75-34-3	8260
1,2-Dichloroethane; Ethylene dichloride	107-06-2	8260
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	8260
cis-1,2-Dichloroethylene; cis-1,2-Dichloroethene	156-59-2	8260
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	8260
1,2-Dichloropropane; Propylene dichloride	78-87-5	8260
cis-1,3-Dichloropropene	10061-01-5	8260
trans-1,3-Dichloropropene	10061-02-6	8260
1,4-Dioxane	123-91-1	8260
Ethylbenzene	100-41-4	8260
2-Hexanone; Methyl butyl ketone	591-78-6	8260
Isopropylbenzene	98-82-8	8260
Methyl acetate	79-20-9	8260
Methyl bromide; Bromomethane	74-83-9	8260
Methyl chloride; Chloromethane	74-87-3	8260
Methylcyclohexane	108-87-2	8260
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3	8260
Methyl tert-butyl ether	1634-04-4	8260
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1	8260
Methylene chloride; Dichloromethane	75-09-2	8260
Styrene	100-42-5	8260
1,1,2,2-Tetrachloroethane	79-34-5	8260
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	8260
Toluene	108-88-3	8260
1,2,3-Trichlorobenzene	87-61-6	8260
1,2,4-Trichlorobenzene	120-82-1	8260
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	8260
1,1,2-Trichloroethane	79-00-5	8260
Trichloroethylene; Trichloroethene	79-01-6	8260

## Federal Target Compound and Analyte List

	CAS	
	Registry	RCRA
TCL/TAL Constituents	Number	Method
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	8260
Vinyl chloride; Chloroethene	75-01-4	8260
Xylene (total)	96-47-6,	8260
	108-38-3,	
	106-42-3,	
	1330-20-7.	
SEMIVOLATILES		
Acenaphthene	83-32-9	8270
Acenaphthylene	208-96-8	8270
Acetophenone	98-86-2	8270
Anthracene	120-12-7	8270
Atrazine	1912-94-9	8270
Benzaldehyde	100-52-7	8270
Benzo(a)anthracene; Benzathracene	56-55-3	8270
Benzo(a)pyrene	50-32-8	8270
Benzo(b)fluoranthene	205-99-2	8270
Benzo(k)fluoranthene	207-08-9	8270
Benzo(ghi)perylene	191-24-2	8270
1,1'-Biphenyl	92-52-4	8270
Bis(2-chloroethoxy)methane	111-91-1	8270
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4	8270
Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiisopropyl	108-60-1	8270
ether; DCIP; 2,2'-Oxybis(1-chloropropane)		
Bis(2-ethylhexyl) phthalate	117-81-7	8270
4-Bromophenyl phenyl ether	101-55-3	8270
Butyl benzyl phthalate	85-68-7	8270
Caprolactam	105-60-2	8270
Carbazole	86-74-8	8270
4-Chloroaniline	106-47-8	8270
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7	8270
2-Chloronaphthalene	91-58-7	8270
2-Chlorophenol	95-57-8	8270
4-Chlorophenyl phenyl ether	7005-72-3	8270
Chrysene	218-01-9	8270
m-Cresol; 3-Methylphenol	108-39-4	8270
o-Cresol; 2-Methylphenol	95-48-7	8270
p-Cresol; 4-Methylphenol	106-44-5	8270
Dibenz(a,h)anthracene	53-70-3	8270
Dibenzofuran	132-64-9	8270
Di-n-butyl phthalate	84-74-2	8270
3,3'-Dichlorobenzidine	91-94-1	8270
2,4-Dichlorophenol	120-83-2	8270
Diethyl phthalate	84-66-2	8270
2,4-Dimethylphenol	105-67-9	8270
Dimethyl phthalate	131-11-3	8270
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1	8270
2,4-Dinitrophenol	51-28-5	8270
2,4-Dinitrotoluene	121-4-2	8270
2,6-Dinitrotoluene	606-20-2	8270

### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 11 Page 3 of 4

	CAS	
	Registry	RCRA
TCL/TAL Constituents	Number	Method
Di-n-octyl phthalate	117-84-0	8270
Fluoranthene	206-44-0	8270
Fluorene	86-73-7	8270
Hexachlorobenzene	118-74-1	8270
Hexachlorobutadiene	87-68-3	8270
Hexachlorocyclopentadiene	77-47-4	8270
Hexachloroethane	67-72-1	8270
Indeno(1,2,3-cd)pyrene	193-39-5	8270
Isophorone	78-59-1	8270
2-Methylnaphthalene	91-57-6	8270
Naphthalene	91-20-3	8270
o-Nitroaniline; 2-Nitroaniline	88-74-4	8270
m-Nitroaniline;3-Nitroaniline	99-09-2	8270
p-Nitroaniline; 4-Nitroaniline	100-01-6	8270
Nitrobenzene	98-95-3	8270
o-Nitrophenol; 2-Nitrophenol	88-75-5	8270
p-Nitrophenol; 4-Nitrophenol	100-02-7	8270
n-Nitrosodiphenylamine	86-30-6	8270
n-Nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-	621-64-7	8270
dipropylamine		
Pentachlorophenol	87-86-5	8270
Phenanthrene	85-01-8	8270
Phenol	108-95-2	8270
Pyrene	129-00-0	8270
1,2,4,5-Tetrachlorobenzene	95-94-3	8270
2,3,4,6-Tetrachlorophenol	58-90-2	8270
2,4,5-Trichlorophenol	95-95-4	8270
2,4,6-Trichlorophenol	88-06-2	8270
ORGANOCHLORINE PESTICIDES/PCBs		
Aldrin	309-00-2	8081
alpha-BHC; alpha-Benzene hexachloride	319-84-6	8081
beta-BHC; beta-Benzene hexachloride	309-85-7	8081
delta-BHC;delta-Benzene hexachloride	319-86-8	8081
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	8081
alpha-Chlordane	5103-71-9	8081
gamma-Chlordane	5103-74-2	8081
4.4'-DDD	72-54-8	8081
4,4'-DDE	72-55-9	8081
4,4'-DDT	50-29-3	8081
Dieldrin	60-57-1	8081
Endosulfan I	959-98-8	8081
Endosulfan II	33213-65-9	8081
Endosulfan sulfate	1031-07-8	8081
Endrin	72-20-8	8081
Endrin aldehyde	7421-93-4	8081
Endrin ketone	53494-70-5	8081
Heptachlor	76-44-8	8081
Heptachlor epoxide	1024-57-3	8081
Methoxychlor	72-43-5	8081

TCL/TAL Constituents	CAS Registry Number	RCRA Method
Toxaphene	8001-35-2	8081
Polychlorinated biphenyls; PCBs; Aroclor 1016, 1221, 1232,	12674-11-2,	8082
1242, 1248, 1254, 1260, 1262, 1268	11104-28-2,	
	11141-16-5,	
	53469-21-9,	
	12672-29-6,	
	11097-69-1,	
	11096-82-5,	
	37324-23-5,	
	11100-14-4	
METALS		
Aluminum	7429-90-5	6010
Antimony	7440-36-0	6010
Arsenic	7440-38-2	6010
Barium	7440-39-3	6010
Beryllium	7440-41-7	6010
Cadmium	7440-43-9	6010
Calcium	7440-70-2	6010
Chromium	7440-47-3	6010
Cobalt	7440-48-4	6010
Copper	7440-50-8	6010
Iron	7439-89-6	6010
Lead	7439-92-1	6010
Magnesium	7439-95-4	6010
Manganese	7436-96-5	6010
Nickel	7440-02-0	6010
Potassium	7440-09-7	6010
Selenium	7782-49-2	6010
Silver	7440-22-4	6010
Sodium	7440-23-5	6010
Thallium	7440-28-0	6010
Vanadium	7440-62-2	6010
Zinc	7440-66-6	6010
Mercury	7439-97-6	7470/7471
MISCELLANEOUS		
Cyanide	57-12-5	9012

## Federal Priority Pollutant List

PPL Constituents	CAS Registry Number	CWA Method
VOLATILES	Number	method
Acrolein	107-02-8	624
Acrylonitrile	107-13-1	624
Benzene	71-43-2	624
Bromodichloromethane; Dichlorobromomethane	75-27-4	624
Bromoform; Tribromomethane	75-25-5	624
Carbon tetrachloride	56-23-5	624
Chlorobenzene	108-90-7	624
Chloroethane; Ethyl chloride	75-00-3	624
2-Chloroethyl vinyl ether	110-75-8	624
Chloroform; Trichloromethane	67-66-3	624
Dibromochloromethane; Chlorodibromomethane	124-48-1	624
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	624
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1	624
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	624
1,1-Dichloroethane; Ethylidene chloride	75-34-3	624
1,2-Dichloroethane; Ethylene dichloride	107-06-2	624
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	624
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	624
1,2-Dichloropropane; Propylene dichloride	78-87-5	624
cis-1,3-Dichloropropene	10061-01-5	624
trans-1,3-Dichloropropene	10061-02-6	624
Ethylbenzene	100-41-4	624
Methyl bromide; Bromomethane	74-83-9	624
Methyl chloride; Chloromethane	74-87-3	624
Methylene chloride; Dichloromethane	75-09-2	624
1,1,2,2-Tetrachloroethane	79-34-5	624
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	624
Toluene	108-88-3	624
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	624
1,1,2-Trichloroethane	79-00-5	624
Trichloroethylene; Trichloroethene	79-01-6	624
Vinyl chloride; Chloroethene	75-01-4	624
SEMIVOLATILES		
Acenaphthene	83-32-9	625
Acenaphthylene	208-96-8	625
Anthracene	120-12-7	625
Benzidine	92-87-5	625
Benzo(a)anthracene; Benzathracene	56-55-3	625
Benzo(a)pyrene	50-32-8	625
Benzo(b)fluoranthene	205-99-2	625
Benzo(k)fluoranthene	207-08-9	625
Benzo(ghi)perylene	191-24-2	625
Bis(2-chloroethoxy)methane	111-91-1	625
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4	625
Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiisopropyl ether; DCIP; 2,2'-Oxybis(1-chloropropane)	108-60-1	625

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 12 Page 2 of 3

	CAS	
	Registry	CWA
PPL Constituents	Number	Method
Bis(2-ethylhexyl) phthalate	117-81-7	625
4-Bromophenyl phenyl ether	101-55-3	625
Butyl benzyl phthalate	85-68-7	625
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7	625
2-Chloronaphthalene	91-58-7	625
2-Chlorophenol	95-57-8	625
4-Chlorophenyl phenyl ether	7005-72-3	625
Chrysene	218-01-9	625
Dibenz(a,h)anthracene	53-70-3	625
Di-n-butyl phthalate	84-74-2	625
3,3'-Dichlorobenzidine	91-94-1	625
2,4-Dichlorophenol	120-83-2	625
Diethyl phthalate	84-66-2	625
2,4-Dimethylphenol	105-67-9	625
Dimethyl phthalate	131-11-3	625
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1	625
2,4-Dinitrophenol	51-28-5	625
2,4-Dinitrotoluene	121-4-2	625
2.6-Dinitrotoluene	606-20-2	625
Di-n-octyl phthalate	117-84-0	625
1,2-Diphenylhydrazine	122-66-7	625
Fluoranthene	206-44-0	625
Fluorene	86-73-7	625
Hexachlorobenzene	118-74-1	625
Hexachlorobutadiene	87-68-3	625
Hexachlorocyclopentadiene	77-47-4	625
Hexachloroethane	67-72-1	625
Indeno(1,2,3-cd)pyrene	193-39-5	625
Isophorone	78-59-1	625
Naphthalene	91-20-3	625
Nitrobenzene	98-95-3	625
o-Nitrophenol; 2-Nitrophenol	88-75-5	625
p-Nitrophenol; 4-Nitrophenol	100-02-7	625
N-nitrosodimethylamine	62-75-9	625
N-nitrosodiphenylamine	86-30-6	625
N-nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-	621-64-7	625
dipropylamine	021-04-7	025
Pentachlorophenol	87-86-5	625
Phenanthrene	85-01-8	625
Phenol	108-95-2	625
Pyrene	129-00-0	625
1,2,4-Trichlorobenzene	129-00-0	625
	88-06-2	625
2,4,6-Trichlorophenol ORGANOCHLORINE PESTICIDES/PCBs	00-00-2	020
Aldrin	200.00.2	609
	309-00-2	608
alpha-BHC; alpha-Benzene hexachloride	319-84-6	608
beta-BHC; beta-Benzene hexachloride	309-85-7	608
delta-BHC;delta-Benzene hexachloride	319-86-8	608
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	608

### Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 12 Page 3 of 3

PPL Constituents	CAS Registry Number	CWA Method
alpha-Chlordane	5103-71-9	608
gamma-Chlordane	5103-74-2	608
4,4'-DDD	72-54-8	608
4,4'-DDE	72-54-8	608
4,4-DDE 4,4'-DDT	50-29-3	608
Dieldrin	60-57-1	608
Endosulfan I	959-98-8	608
Endosulfan II	33213-65-9	608
Endosulfan sulfate	1031-07-8	608
Endrin	72-20-8	608
Endrin aldehyde	7421-93-4	608
Endrin ketone	53494-70-5	608
Heptachlor	76-44-8	608
Heptachlor epoxide	1024-57-3	608
Methoxychlor	72-43-5	608
Toxaphene	8001-35-2	608
Polychlorinated biphenyls; PCBs; Aroclor 1016, 1221, 1232,	12674-11-2,	608
1242, 1248, 1254, 1260	11104-28-2.	000
1242, 1240, 1204, 1200	11141-16-5,	
	53469-21-9,	
	12672-29-6,	
	11097-69-1,	
	11096-82-5	
METALS		
Antimony	7440-36-0	200.7
Arsenic	7440-38-2	200.7
Beryllium	7440-41-7	200.7
Cadmium	7440-43-9	200.7
Chromium	7440-47-3	200.7
Copper	7440-50-8	200.7
Lead	7439-92-1	200.7
Nickel	7440-02-0	200.7
Selenium	7782-49-2	200.7
Silver	7440-22-4	200.7
Thallium	7440-28-0	200.7
Zinc	7440-66-6	200.7
Mercury	7439-97-6	245.1
DIOXIN		
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	8280/ 8290
MISCELLANEOUS		
Asbestos		
Cyanide	57-12-5	SM4500- CN C, E
Phenols	NA	420.1& 420.4

#### Industrial Hygiene Specific Information

**A13.1** TestAmerica Nashville holds accreditation with the American Industrial Hygiene Association for metals and particulates analyses. The QA/QC systems and objectives described in this QA Manual are the same as those followed for analysis of industrial hygiene samples. The methods used are those currently approved by the U.S. OSHA and NIOSH, in particular from the <u>NIOSH Manual of Analytical Methods</u>.

#### A13.2 QUALITY ASSURANCE OBJECTIVES

	LCS	Reporting Limit
ANALYTE	ACCURACY %	µg/FILTER or TUBE
Metals by NIOSH 730		
Aluminum	80-120	1.25
Arsenic	80-120	1.25
Beryllium	80-120	1.25
Cadmium	80-120	1.25
Calcium	80-120	1.25
Chromium	80-120	2.50
Cobalt	80-120	1.25
Copper	80-120	1.25
Iron	80-120	1.25
Lead	80-120	1.25
Lithium	80-120	1.25
Magnesium	80-120	1.25
Manganese	80-120	1.25
Molybdenum	80-120	1.25
Nickel	80-120	1.25
Selenium	80-120	1.25
Silver	80-120	1.25
Sodium	80-120	1.25
Strontium	80-120	1.25
Thallium	80-120	1.25
Titanium	80-120	1.25
Vanadium	80-120	1.25
Zinc	80-120	12.5
Particulates by NIOSH 0500	NA	NA

NA: Not Applicable

Parameter	Maximum Holding Time	Cool to 4°C	Sampling Media	Flow Rate, L/min	Maximum Volume, Liters
Metals	Indefinitely	No	MCE Filter Casssette	1-4	200-2000
Particulates	Indefinitely	No	PVC Filter Cassette with Preweighed Filter (Do not use matched-weight filters for paint sampling; use filters pre- weighed by the lab.)	1-2	7-133

### A13.3 SAMPLE HOLDING TIMES AND MEDIA REQUIREMENTS

### A13.4 PROFICIENCY TESTING

**A13.4.1** Proficiency testing is performed for each accredited AIHA Field of Testing (FoT). The laboratory participates in and follows all proficiency testing requirements, including frequency of testing, as outlined in current effective AIHA Policy Modules 3, 6A, and 6B.

**A13.4.2** Where an AIHA PT program is available for a certified FoT, the laboratory participates in that program. For FoTs not covered by available AIHA supplied PT samples, the laboratory demonstrates competency annually for that FoT through participation in a round robin program between a minimum of 3 AIHA accredited laboratories within the TestAmerica network. The round robin program includes at least two rounds a year, with each study completed within a six month time frame. The TestAmerica round robin program instructions for particulates are outlined in work instruction CF-IH-WI-02.

### A13.5 AMENDED REPORTS

**A13.5.1** Amended AIHA reports contain the following statement in **bold print** in the comments section on the cover page of the amended report, in addition to the notation requirements in Section 26.9:

"Supplement to Test Report" along with the WorkOrder number of the original report, the date of the original report, and the time the original report was generated.

#### A13.6 SUBCONTRACTING

**A13.6.1** Unless directed otherwise by a client or regulatory agency, subcontracted work for Fields of Testing covered by the scope of accreditation of TestAmerica Nashville is performed by an AIHA accredited laboratory.

### A13.7 TEST REPORTS

**A13.7.1** For client projects containing only AIHA-certified parameters (NIOSH methods), "AIHA" is entered into the "Location" field in the Element project set-up screen. If the client's project includes other non-NIOSH methods, the state of origin is entered into the "Location" field to ensure proper reporting per AIHA requirements.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 Appendix 13 Page 3 of 3

**A13.8 Control Limit generation for Duplicate QC data** The Taylor\* approach is used to generate control limits for Duplicate QC data for NIOSH methods. This is a root-mean-square approach, where the root-mean-square sum of the RPD value is derived and that value is then used as a standard deviation. 2x and 3x that value are plotted as warning and control limits.

\*Taylor, John K., Quality Assurance of Chemical Measurement, pp. 22-23, Lewis Publishers, Chelsea, MI 1987.

Document No. 0001 Section Revision No.: 21 Section Effective Date: 8/1/2011 End Page 1 of 1

# End of Document

